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BURST TESTING FOR PAPERBOARD ASEPTIC PACKAGES WITH
FUSION SEALS

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Major professor

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**BURST TESTING FOR PAPERBOARD ASEPTIC PACKAGES WITH
FUSION SEALS**

By

George William Arndt, Jr.

A THESIS

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

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1990**

ABSTRACT**BURST TESTING FOR PAPERBOARD ASEPTIC PACKAGES WITH
FUSION SEALS****By****George William Arndt, Jr.**

Burst testing may be used to determine the strength of fusion seals for 250 ml brik paks. Packages with strong fusion seals are more likely to maintain hermetic integrity in distribution. Separation of packages with weak seals from packages with strong seals is more easily accomplished using a restraining device during burst testing. Statistical methods are employed to compare sample lots having unknown seal integrity to standards of "good" and "bad". Data derived from dynamic burst testing is used to establish the requirements for a static burst test similar to that required for retortable pouches. Visual inspection requirements for metal cans, retortable pouches, plastic cans with double seamed metal ends, plastic trays with peelable and fusion sealed flexible ends, and composite paperboard packages are summarized. Sixteen approved methods for determining hermetic integrity in shelf-stable, low-acid food packages are reviewed in 168 pages, 19 illustrations, and 50 tables.

DEDICATION

To Theron Downes, Ph.D., my major professor and to Roger Griffin of the School of Packaging for direction, insight and enthusiasm; and to my wife Jacqueline for patience, support and encouragement.

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INTRODUCTION

Packaging is evolving very rapidly. There are many new food packages in distribution today that did not exist ten years ago. These include thermostabilized or aseptic plastic cups and trays with fusion or peelable flexible lids, plastic cans with double seams, retortable pouches with and without aluminum foil, and paperboard composite packages which are aseptically filled prior to sealing. The U.S. Department of Agriculture (USDA) has expressed a desire to see test methods employed by food packers, which would give indication of the ability of these packages to survive the rigors of distribution. This is especially true where commercially sterile low-acid foods are packaged in these new containers. Low-acid foods having a pH greater than 4.6 will support the growth of pathogenic microorganisms (AOAC, 1984). If the hermetic barrier of a commercially sterile package were breached, these ubiquitous organisms would multiply in the food. If consumed, the product could cause illness or death. The consequences to the food processor are severe. Product recall, loss of market, and bankruptcy often follow in the wake of public health incidents involving commercial food products.

Double seamed metal cans have had over 150 years of development in the laboratory. The integrity of these new flexible and semirigid containers is monitored using many of the guidelines developed for metal cans. The retort pouch was the first flexible thermostabilized package approved by the U.S. Food and Drug Administration (FDA) and USDA for low-acid foods.

They have been used in the United States principally for military food rations. The guidelines for MRE (Meals, Ready to Eat) pouches have formed the basis upon which many of these new packages are tested during packing by food processors.

Military and commercial specifications for the retort pouch require a tensile test and a burst test to monitor the strength of fusion seals prior to distribution. The requirements for burst testing are described as preretort (prior to thermal processing) 30 psig for 30 seconds at 3/4 inch restraint, and post retort 20 psig for 30 seconds at 3/4 inch restraint (MIL-P-44073 B, 1986). Pouches are emptied prior to testing and inflated at a rate of 1 psig/second. The burst test requirement has been in effect since U.S. commercial production began in 1978 and has not been changed. The principle advantage of the burst test is that it stresses the entire package and exerts an even pressure upon the entire seal area. The assumption that seals which resist burst pressure are more likely to survive the rigors of distribution than seals which fail under burst pressure is an important point.

Fusion sealing is used to create a hermetic barrier in plastic cups, bowls, trays, and aseptic composite paperboard packages. The physical appearance of the new packages differ considerably from the retort pouch. However, there is little difference in the mechanism of closure using plastic fusion. Heat is employed to melt the plastic sealant material, and pressure is used to force mixing and extrusion. Time is required to attain sufficient heating and cooling before seals are released from the clamping mechanism. The commonalty of sealing methods should permit the use of accepted testing methods for those packages having fusion seals. Thus a burst test may be employed to ascertain whether or not these new packages

might survive distribution without a loss of hermetic barrier. The information which follows describes how the burst test that was developed for the retort pouch, may be used to test aseptic composite packages.

Presently, there are approximately 160 aseptic sealing machines operating in the United States. Many produce aseptic composite packages consisting of paper, aluminum foil, and various layers of plastic materials. The most popular aseptic composite package is the 250 ml rectangular shaped paper box. These packages and packaging equipment are produced and marketed by three companies. These are Combibloc, International Paper Company, and Brik Pak. Brik Pak, formerly known as Tetra Pak is currently the U.S. market leader. All three packages are approved for use with low-acid, still liquid foods. The packages are sterilized using hydrogen peroxide (H_2O_2) prior to filling the heat sterilized product and sealing under sterile conditions. Still liquids are essential when the package is sealed below the surface of the liquid. If food particles are present in the product they may contaminate seals causing seal defects and contribute to a loss of hermetic barrier when packages are later stressed during distribution. Both International Paper Company and Brik Pak use vertical form, fill, and seal, below the level of the liquid. Combibloc employs individual blanks which are sealed on the side and bottom prior to depositing product and sealing the top of the finished package. This method of filling permits solids and fibers to be present in the product without contaminating the sealant surface. Once thermal processing of low-acid foods containing solids is approved by the FDA and USDA, there will be renewed activity in the U.S. to manufacture and distribute aseptic foods with particulates. The significant benefit to processors is elimination

of the labor intensive batch retorting process typical in today's food plants. For the processor, this not only affords a higher quality product with the potential for selling at a greater cost to consumers, but also the opportunity to reduce the cost of manufacturing and the stressing of package seals in the retort. However, regulatory agencies advise caution.

Recent advances in thermal processing enable food processors to aseptically process and package low-acid foods of high viscosity containing particulates as large as 3/4 inch in cross section (Hersom and Shore, 1981). A number of aseptic filling machines are capable of packaging these products at speeds in excess of 100 units per minute. However, the desire to aseptically package low-acid foods containing vegetables and meats has raised concern from regulatory agencies (Densford, 1983). Fred Phillips, Special Assistant for Low Acid Foods at the Bureau of Foods, stated:

"We're not in the business to make money. We're in the business to protect public health. This puts us in a sensitive position regarding aseptic packaging. On the one hand, we don't want to hinder the development of a new process or equipment. But on the other, we must make certain these do not create potential health problems. This is especially true of low acid foods." (Densford, 1984).

Packaging integrity of aseptic systems is a critical FDA concern. Poor heat seals have caused a number of spoilage problems. Phillips (Densford, 1984) stated that "either not enough testing is being done on seals or that tests are incomplete." This fact was recognized ten years ago by Natick researchers developing the retort pouch for Meals, Ready to Eat (MRE) rations.

"Although seal performance has long been recognized as vital, especially with rigid, thermoprocessed food containers, flexible package seals have not had to perform in accordance with strict and defined criteria as they do now that flexible packages are being used for thermoprocessed foods. Primarily because flexible package seals have not received attention from a critical performance aspect and because seal failures have occurred within somewhat high but tolerable percentages for foods not requiring a hermetic package, the definition and requirements for a good seal have been subjective and somewhat nebulous." (Lampi, et.al., 1976).

FDA and USDA are concerned about the integrity of flexible and semirigid packaging, both retorted and aseptically packaged. There is a need to know when food packages are safe and when they are a risk to consumers. The current authority governing hermetic integrity is contained in the Congressional Federal Register, 21CFR part 113. This document describes "what shall be measured and how it shall be measured." However, this document covers only metal double seams. Part 113 does not contain closure requirements for plastic cans with double seams, aseptic composite packages, or fusion and peelable septum lids on aseptic and retortable containers. The criteria for determining the integrity of seals on these new packages is currently not part of 21CFR.

The food industry of the United States has a responsibility for total control of container integrity. In the event of a problem, the regulatory agencies will address the packer as the cause. The Good Manufacturing Practices (GMP's) apply to the packer because the final quality control inspection point prior to distribution is under the packer's control. When the Can Manufacturer's Institute drafted proposed guidelines for metal cans with double seams five years ago, this information was quickly

formalized by the regulatory agencies. These regulations caused adverse economic impact and consternation within the industry in the years that followed. A photo chart illustrating metal can defects was published by Standards Canada and later by the Association of Analytical Chemists (A.O.A.C.) in the U.S. This photo chart, along with the National Food Processor's Association (NFPA) Guidelines for Evaluation and Disposition of Damaged Canned Food Containers, Bulletin 38-L (NFPA, 1979), form the existing basis for the determination of metal can inspection criteria.

Regulations, inspection criteria, and test methods for flexible packages having fusion seals developed from MIL-SPEC 32-74A Retortable Pouches (U.S. Superintendent of Documents, 1974) were the only guidelines available for many years. These were restated in the USDA's Guidelines for Aseptic Processing and Packaging Systems in Meat and Poultry Plants published in June of 1984. The package Testing required by this document is listed below.

Package Testing - The establishment must define the procedures to be used to assure that product is properly filled and sealed in the packaging unit. The establishment must propose appropriate procedures and descriptions to ensure that each individual container is free from the following defects before shipment:

1. Improper closure or seal
2. Delamination of package
3. Overfill
4. Leaker
5. Swollen or blown
6. Severely damaged
7. Soiled or stained
8. Other (specify)

The vagueness of these guidelines is intentional. Neither the USDA nor the FDA desire to restrict development of technology. When situations involving unsafe food packages develop, these regulatory agencies have the authority to impose methods for sampling and testing. Test methods developed for aseptic packages with fusion seals by committees of the American Society for Testing Materials (ASTM) D-10 and F-2 are firmly based upon the retort pouch. The lidding materials for retortable semirigid containers currently available are "extensions of the retort pouch technology" (Lopez, 1987).

A recent survey of food processors indicated that food processors do not believe that a major contamination crisis would result in severely restrictive legislation by regulatory agencies (Duprey, 1984). An example of this is the observation that contamination occasionally occurs in cans and bottles. Yet following a recall, these packages are still acceptable. However, regulations in the U.S. are often a response to topics of national attention. Following the Listeria epidemic resulting from a cross-connection in the clean-in-place (C.I.P.) processing system at Jewel Dairy in Chicago, there were many packaging concerns expressed along with a perceived need to educate dairy operators. The FDA issued guidelines following the Listeria incident stating that "the industry will clean itself up or get cleaned up by the FDA." Information was distributed by the FDA in every state and many seminars were held on the topic. The Pasteurized Milk Ordinance (U.S. Government, 1978) has not evolved with advancing technology. Consequently, during the Listeria incident this contributed to a situation where many felt a need to quickly update. A number of incidents followed which perpetuated the opinion that there needs to be a lock on every package. A prime example is the Clostridium botulinum incident

involving canned salmon, which occurred in Europe during 1981. Investigators determined the cause was post process contamination resulting from a container defect. The National Food Processors Association (NFPA) committee for Microbiology in the Food Industry determined that for metal cans, the incidence for post process contamination due to container defects is less than or equal to the incidence of problems associated with underprocessing.

A second example involved a recall of MRE rations in 1985 and a Medical Hold issued by the U.S. Surgeon General. Subsequent investigation revealed inadequate quality control and worn out retort racks at some establishments (Densford, 1987). Extensive testing of samples from many processors revealed the incidence of holes in these containers was widespread. Fluorescent dye (zyglo) was used to identify holes in the surfaces and seal areas of retort pouches. Research by American National Can revealed that zyglo fluorescent dye pigment permeates the polypropylene sealant when left in contact for more than two hours. The current test allows exposure for 30 to 60 minutes (Genske, 1986). Despite these findings the military continues using zyglo dye testing.

A third event grew out of the canned salmon problem previously mentioned. The British have developed a two-class defect system for inspecting imported canned foods: critical and minor. Defects which are not classified as minor are immediately classified as critical. Canada has recently converted to the British classification system and CODEX ALIMENTARIOUS has entertained proposals to use the two-class defect system world-wide. The U.S. historically has employed a three class defect system: critical, major, and minor. The NFPA is concerned, and has pointed out to their member companies that acceptable US canned product

could be placed "on hold" by inspectors for minor defects. The NFPA conducted a test survey involving over 5 million containers covering six different metal can types. They concluded four to ten times more rejections of 12,000 can lots would occur using the two class British system than using the three class US system for metal can defects (Denny, 1987). Some members of the canning industry would like to see a four class system: critical, major, minor, and cosmetic in place. However, when considered, NFPA Flexible Packaging Integrity Committee (FPIC) members recommended a four class system be avoided because of the likelihood of regulatory agencies grouping critical and major defects to form a very oppressive system with three defects: critical, minor, and cosmetic.

In November 1984, the NFPA organized the Flexible Packaging Integrity Group (FPIG) to deal with this issue and to perform three tasks for each of four emerging packaging groups which are not covered by 21CFR, Part 113. (This is shown in Table 1)

TABLE 1
FPIG Package Groups (Aseptic or Retortable)

1. Flexible Pouches
2. Plastic Cans with Double Seamed Metal End
3. Plastic Packages with Heat Sealed Lids
4. Paperboard Packages

Three tasks for Each Group

1. Define visible package defects observed at the retail level and illustrate each with a photograph of the defects on the package.
2. Draft Good Manufacturing Practices for the manufacture of flexible and semirigid packages and rollstock.
3. Draft quality control guidelines and propose test methods for package integrity verification during packing.

The objectives set down by the FPIG are aimed at providing technical information to the FDA and USDA to fulfill two purposes. These purposes are (1) to document that these new packages are safe and reliable, and (2) to provide information that may be used to draft guidelines and regulations should regulatory agencies elect to add this information to 21CFR, part 113.

A. History Of Aseptic Packaging

Aseptic packaging had its origins in the U.S. in the late 1920's when Dole and Martin developed a process for sterilizing metal cans in an atmosphere of super heated steam (Buchner, 1984). In the early 1950's Loelinger and Reges developed hydrogen peroxide (H_2O_2) sterilization for paper/polyethylene tetrahedral shaped cartons.

Ruben Rausing developed a folded pouch - paperboard carton he called "Brik Pak" in 1952. By 1961 Rausing had devised an aseptic processing system that used H_2O_2 to sterilize both the package and the product (Westerman, 1982). Verbands Molkerie in Bern, Switzerland was the first commercial dairy using H_2O_2 to market aseptically packaged milk in paperboard cartons (Johnson, 1966). The liquid milk was thermally processed in heat exchangers in a process which was termed "ultra high temperature."¹ Paperboard packages were sterilized by immersion in 35 percent H_2O_2 (in water) and dried with heated air. Loelinger and Reges

¹ Ultra high temperature processing or UHT describes a thermal process for pumpable liquids. The food product is forced through a heat exchanger at a carefully controlled rate. It is heated in small diameter tubes, between closely held plates, or forced through an atmosphere of steam. Once heated, it is held for a short period of time to assure destruction of viable microorganism and spores of Cl. botulinum. The final step is a cooling process using a second heat exchanger. The process temperature generally exceeds 250°F and the time is shorter than required for the same product inside most containers sterilized in a retort.

continued development of the thermal process using milk sterilized by direct steam injection within a closed vessel (Johnson, 1966). The sterile product was then piped to filling machines, where paperboard packages sterilized by H_2O_2 were filled under aseptic conditions. This technology was further refined by the Swedish firm that came to be known as Tetra Pak. The advantages of aseptic packing were quickly realized. With most nations lacking refrigeration capabilities for fresh milk and suffering bacterial contamination of water sources, the inexpensive paperboard aseptic packages quickly found a world market. Tetra Pak machines and aseptic processing systems are now found in 80 countries. The success of the Tetra Pak and brik shaped packages led to competition for Tetra Pak as International Paper Company (IP) and Combibloc (PKL) developed and marketed their own versions of the aseptic paperboard package. In a push to expand into the lucrative U.S. market, Tetra Pak petitioned and received approval from the FDA on January 9, 1981 for H_2O_2 sterilization of polyethylene food contact surfaces (Westerman, 1982). Harold Thorkilsen, President and C.E.O. for Ocean Spray Cranberries, Incorporated, had followed the development of the brik pak in Europe for many years. He viewed this package as an opportunity to expand Ocean Spray's share of the mature U.S. bottled fruit juice market (Pehanich, 1983). In the summer of 1983, Ocean Spray became the first U.S. processor to market aseptically packaged drinks in the U.S. The great success for Ocean Spray occurred without a loss of market share of their glass packaged beverages (Pehanich, 1983). Ocean Spray had created a new market for convenience refreshments; school lunch boxes and vending machine sales. Hawaiian Punch by DelMonte, the first brand name product to be marketed nationally was introduced in 1983 in 250 ml brik paks (Buehler, 1983).

The rapid expansion in aseptic packaging of fruit drinks soon outpaced the industry's capability to manufacture machinery. International Paper and Combibloc entered the U.S. market in competition with Tetra Pak. Petitions for approval of sterilization techniques, machines, and new aseptic packaging materials flooded into FDA. Sterilization methods included ultra violet light, gamma irradiation, hot air, and heat from plastic formation. To expand the potential for H₂O₂ sterilization and to permit alternate packaging materials to be used on approved packaging systems, DuPont made application in 1983 for approval of E.V.A., polyester, acid copolymers and a number of ionomers to be sterilized using hydrogen peroxide. On February 2, 1985 the FDA announced their decision to amend 21CFR Part 178.1005 and grant approval of DuPont's request. This followed an announcement by FDA issued on March 13, 1984 to approve an NFPA petition allowing H₂O₂ sterilization of olefin food contact surfaces to be used for aseptic packaging (CFR, 1984). A current summary of food contact materials for sterilization using hydrogen peroxide is shown in Table 2.

TABLE 2
NFPA INFORMATION APPROVALS OF PACKAGING MATERIALS
WHERE HYDROGEN PEROXIDE STERILIZATION IS PERMITTED*

<u>NAME</u>	<u>COMPANY</u>	<u>APPROVAL DATE</u>
Vinylidene chloride/methyl acrylate copolymers	Dow Chemical Co.	Nov. 22, 1988
Poly-1-butene resins and butene/ethylene copolymers	Shell Oil Company	March 21, 1988**
Vinylidene chloride-vinyl chloride copolymers	Dow Chemical Co.	June 4, 1987**
Ethylene-acrylic acid copolymer	Dow Chemical Co.	Dec. 23, 1986
Ethylene-carbon monoxide copolymers (heat seal layer)	Dow Chemical Co.	Sept. 24, 1986**
Polystyrene and rubber - modified polystyrene resins	Dow Chemical Co.	Dec. 20, 1985
Polycarbonate resins	General Electric Co.	Nov. 15, 1985**
Ethylene - methyl acrylate copolymer	Gulf Oil Co.	April 5, 1985
Ethylene - Vinyl Acetate copolymer	DuPont	Sept. 26, 1984
Polyethylene phythalate polymers	DuPont	Sept. 26, 1984
Polyethylene terephythalate polymers	DuPont	Sept. 26, 1984
Ionomeric resins (Surlyn Ionomer resin, Elvax resin, Nucler Acid copolymer resin, Mylar polyester film, Surlyn Ionomer dispersion)	DuPont	Sept. 26, 1984
Olefin polymers	NFPA	Sept. 26, 1984
Polyethylene	Tetra Pak	Jan. 9, 1981

*See Federal Register, Vol. 53, No. 225, Tuesday, November 22, 1988, page 47184 for details.

**Files with FDA under 21CFR 178.1005 but not yet finalized. (Meaning - you can't use if for interstate shipment of foods.)

Industry experts estimate that there were 110 aseptic filling machines operating in the U.S. in 1983 (Tillotson, 1984). By 1984 this number had reached 150 (Bertrand, 1984), the most common aseptic package in the U.S. being the 250 ml Brik Pak. By 1986 the number of new installations were reduced. The list of systems with FDA approved low-acid thermal process is shown in Table 3.

TABLE 3

Aseptic Packaging Systems in the U.S. in 1987 with FDA approved thermal processes for low-acid products (FDA speaker NFPA National Convention Aseptic Session, Chicago 26 Jan, 1987.)

Benco
Bosch
Connofast
Combibloc
Dole
International Paper
Metal Box
Tetra Pak

In theory, any food product that can be pumped can be aseptically processed and packaged. Eliminating the retort process poses significant economic advantages to food processors. In 1987 aseptic packaging in the U.S. was mainly limited to liquids. It may be several years before low-acid foods with large particulates are commercially available. High-acid foods for reprocessing and institutional feeding have been available for a number of years. Lyons-Magnus in Fresno, California for example, aseptically packages fruit concentrates and toppings. Many fruit drink products use imported pulp concentrates aseptically packaged in pallet sized bag-in-box containers.

The integrity of high-acid aseptic packages is of less consequence than that of low-acid packages. Spoilage organisms living in high-acid environments do not possess the pathogenic capabilities associated with spoilage organisms in low-acid foods. The line of demarcation being pH 4.6, and a water activity (A_w) of .85, (ASTM F2.3, 1985). Consequently, regulatory agencies, industry, and the public at large place greater emphasis upon the hermetic integrity of low-acid food packages. As the industry moves from replacement of traditional retorted low-acid foods in metal cans with double seams, toward convenience oriented aseptically filled containers with fusion and peelable lids, many questions must be addressed. Neither the regulatory agencies nor industry have all the answers to questions concerning these package defects. Some of the methods currently proposed for determining hermetic (barrier to microorganisms) integrity in low-acid shelf stable food products will be outlined later. First package defects and their significance will be reviewed.

B. Classification Of Container Defects For Low-Acid Foods

Defects may be classified by degree of significance in relation to the health risks associated with human consumption. There can be three levels: critical, major, or minor. They are defined by the A.O.A.C. in Classification of Visible Can Defects. (1984)

CRITICAL DEFECT

Definition: "Defects which provide evidence that the container has lost its hermetic seal (e.g. holes,

fractures, punctures, product leakage, etc.) or evidence that there is, or has been, microbial growth in the can contents." (A.O.A.C. 1984).

MAJOR DEFECT

Definition: "Defects that result in cans which do not show visible signs of having lost their hermetic seal, but are of such magnitude that they may have lost their hermetic seal." (A.O.A.C. 1984).

MINOR DEFECT

Definition: "Defects which have no adverse effects on the hermetic seal." (A.O.A.C. 1984).

The identification of any defect in a food package requires a decision on the part of the processor, regulatory inspector, or customer. The actions required by the food processor or inspector are shown below for each decision involving food package inspections.

<u>DEFECT</u>	<u>REQUIRED ACTION</u>
Critical	"When one critical defect is found the lot must be set aside and thoroughly inspected and sorted to ensure that no containers that have lost their hermetic seal are distributed." (A.O.A.C., 1984).
Major	"When a major defect is found the lot is set aside and each defective package must be examined to determine if the defect might result in a loss of hermetic integrity. If testing verifies that the package may lose its hermetic integrity in distribution the lot must be thoroughly inspected and sorted to ensure that

no containers having this defect are distributed. If testing determines that no containers having this defect are likely to fail in distribution the lot may be released." (A.O.A.C., 1984).

Minor "Minor defects have no affect upon the hermetic integrity of food packages." (A.O.A.C., 1984). These may be released for distribution.

Release authorization usually follows sampling and comparison of the number of defects in a lot of specified size with acceptable quality levels (AQL) which a processor has established. Aside from regulatory and public health concerns the AQL for package defects is based upon the market's tolerance of these defects. A defect of minor significance from a public health standpoint would be important from a processor's standpoint if it affects commercial sales. Dented cans are one example of a minor defect which processors seek to remove from distribution. The hermetic integrity may be acceptable but customers resist purchasing these cans.

The risk to human health derives from contamination by pathogenic micro-organisms. This has been clearly defined by the ASTM subcommittee on Aseptic Packaging Terminology F2.5 in publications to members following a meeting on 17 April 1985.

"Contamination is the entry of viable microorganisms into a finished package due to a loss of container integrity (NFPA). Container integrity is defined as the physical condition of a finished package, including, but not limited to, the security of package seals, which ensures the maintenance of the package contents in a commercially sterile condition (NFPA). Commercial sterility as applied to aseptic

packaging refers to the condition achieved by application of heat, chemical sterilants, or other appropriate treatment that renders the equipment and containers free of viable microorganisms having public health significance, as well as microorganisms of non-health significance, capable of reproducing in the food under normal non-refrigerated conditions of storage and distribution.

Classification of package defects by a processor conducting on-line and predistribution inspections is required if the processor is to comply with 21CFR part 113.

"Regular observation shall be maintained during production runs for gross closure defects. Any such defect shall be recorded and corrective action shall be taken and recorded. At intervals of sufficient frequency to ensure proper closure, the operator, closure supervisor, or other qualified closure inspection personnel shall visually examine the closure of any (package) being used."

This document also states the definition of a hermetic closure.

"Hermetically sealed container means a container which is designed and intended to be secure against the entry of micro-organisms and to maintain the commercial sterility of its contents." (21CFR part 113).

Package defects may be the result of mechanical damage incurred in manufacturing, due to inadequate maintenance of machinery or occur as a consequence of some problem inherent to the packaging material. Defects often go undetected because of their low incidence or because methods for detection are inadequate. Failure may occur during transportation, warehousing, or at the retail store. The great concern for public health officials and regulatory agencies involves the consumption of foods from

defective containers when the defect was not detected prior to the product being eaten. When detected, defects must be identified according to their significance. Defect classification is a team effort involving Q.C. lab personnel, line workers, maintenance, machine operators, foremen and plant management. The tools are specifications, tests, and records which permit a reconstruction of the history of occurrences of package defects. Given sufficient information, the cause and effect relationships contributing to defects may be revealed. Test methods which objectively measure physical variables that are indicative of package defects are needed. These are reviewed in the section on Test Methods for Packages with Fusion Seals contained in Appendix B.

The purpose of the NFPA - FPIC guidelines, tables, and classification of defects is to give regulatory agencies confidence that the industry is taking actions that will preclude problems. A summary of visible package defects involving these packages at the retail level is shown in Table 4. Tables 5,6,7,8 and 9 show container defects classified in terms of their risk to public health. Information for metal cans with double seams is from A.O.A.C. (1984) and information on new containers not covered by 21CFR 113, is from the NFPA-FPIG (unpublished April, 1986 Draft).

TABLE 4
NEPA - EPIG LIST OF VISIBLE PACKAGE DEFECTS

Package Description

- 1.Flexible Pouches
- 2.Plastic Can with Double Seamed Metal End
- 3.Plastic Package with Heat Sealed Lid
- 4.Paperboard Package

Term: X = definition exists
blank = definition does not exist

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Abrasion*	X	X	X	X
Blister	X			
Burnt Seal			X	
Channel Leak(er)	X		X	
Clouded Seal	X			
Compressed Seal	X			
Contaminated Seal	X		X	
Convolution	X			
Corner Dent				X
Corner Leaker				X
Crooked Seal	X			
Crushed		X	X	X
Cut*	X	X	X	X
Defective Seal			X	
Deformed				X
Deformed Seal				X
Delamination*	X	X	X	X
Embossing	X			
Flexcracks	X	X	X	
Foreign Matter (Inclusion)		X	X	
Fracture	X	X	X	
Gels		X	X	
Hotfold	X			
Incomplete Seal			X	
Label Foldover			X	
Leaker	X			
Loose Flaps				X
Malformed		X	X	
Misaligned Seal	X			X
Nonbonding	X			
Notch Leaker	X			
Puncture*	X	X	X	X

* = Common Term

TABLE 4 (continued)
NEPA - FPIG LIST OF VISIBLE PACKAGE DEFECTS

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Seal Creep	X			
Seal Leaker				X
Seal Width Variation			X	
Shrinkage Wrinkle			X	
Stringy Seal	X			
Swell (Swollen Package)*	X	X	X	X
Uneven Impression			X	
Uneven Seal Junction	X			
Waffling	X			
Weak Seal				X
Wrinkle	X		X	

* = Common Term

TABLE 5
CLASSIFICATION OF VISIBLE DEFECTS FOR METAL CANS WITH
DOUBLE SEAMS
(A.O.A.C. - 1984)

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
food cooked on lid showing rust			X
rust confined to double seam, superficial pitting only			X
rust nearly perforating		X	
rust superficial pitting only			X
affecting appearance, but not integrity			X
moderate, double seam distorted but not affected materially			X
severe body dent, affecting double seam		X	
severe body dent with fractured plate	X		
moderate dent that does not significantly affect the side seam or double seam			X
if side seam or double seams are significantly affected		X	
if plate fractured or opening below double seam	X		
body dent below double seam with no opening visible		X	
obviously open below the double seam	X		
if plate is fractured in double seam or body dent in double seam not fractured	X	X	
double seam dent not creased or sharp			X
double seam dent creased or sharp		X	
double seam dent fractured	X		
double seam not severely fractured			X
mislocked side seam if leaking or loss hermetic seal	X		
mislocked side seam if potential leaker		X	
defective welded side seam blowout crack or hole	X		
defective welded side seam burn through crack or hole	X		
defective welded side seam unbonded	X		
portion of side seam not welded	X		
body wall punctured	X		
pinhole in plate	X		

TABLE 5 (continued)
CLASSIFICATION OF VISIBLE DEFECTS FOR METAL CANS WITH
DOUBLE SEAMS
(A.O.A.C. - 1984)

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
gas formation in can: flipper, soft swell, hard swell, blown can	X		
lid buckle not involving double seam			X
lid buckle barely extending into double seam			X
lid buckle extending further into double seam		X	
cable burn through double seam	X		
cable burn not through double seam		X	
cable-slight abrasion on double seam			X
closure double seam not completed	X		
closure double seam if loss of vacuum or hermetic seal	X		
closure double seam if no loss of vacuum or hermetic seal		X	
cutover or fractured seam, plate fractured or loss of hermetic seal	X		
cutover or fractured seam, if sharp seam and not fractured	X		
torn body flange caused by can reformer	X		
torn body flange caused without obvious hole	X		
droop causing a reduction in cover hook length, with loss of hermetic seal	X		
droop causing a reduction, if droop is more than 1/3 double seam height		X	
droop causing a reduction, if droop is not more than 1/3 double seam height			X
end curl knocked down with loss of hermetic seal	X		
end curl knocked down and a potential leaker		X	
multiple vees with end curl knocked down and loss of hermetic seal	X		
multiple vees with end curl knocked down and potential leaker		X	
knocked down flange with loss of of hermetic seal	X		

TABLE 5 (continued)
CLASSIFICATION OF VISIBLE DEFECTS FOR METAL CANS WITH
DOUBLE SEAMS
(A.O.A.C. - 1984)

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
knocked down flange		X	
false seam, knocked down flange	X		
fractured curl with loss of hermetic seal	X		
fractured curl		X	
cut seam fractured or leaking	X		
score line fracture leaking	X		
fractured lid	X		
torn lid, knocked down end	X		

TABLE 6
CLASSIFICATION OF VISIBLE DEFECTS FOR FLEXIBLE POUCHES
(NFPA - FPIG 21 APRIL 1986, UNPUBLISHED)

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
Abrasion - if halfway through - if less than halfway through		X	X
Blister - if significant - if slight			X X
Channel leaker	X		
Clouded seal			
Compressed seal - if significant - if slight		X	X
Contaminated seal - if significant - if slight		X	X X
Convolution			X
Crooked seal			
Cut			X
Delamination - if significant - if slight - if barely noticeable		X	X X
Flexcracks			X
Fracture	X		
Hot fold			X
Leaker	X		
Misaligned seal - if significant - if slight		X	X
Nonbounding	X		
Notch leaker	X		
Puncture	X		
Seal creep - if significant - if slight		X	X X
Stringy seal			X
Swollen package	X		
Uneven seal juncture - if slight			X
Waffling			
Wrinkle - if greater than halfway through the seal - if less than halfway through the seal		X	X

TABLE 7
CLASSIFICATION OF VISIBLE DEFECTS FOR PLASTIC CAN WITH
DOUBLE SEAMED METAL END
(NFPA - FPIG 21 APRIL 1986, UNPUBLISHED)

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
Abrasion			
- if more than halfway through the plastic		X	
- if less than halfway through the plastic			X
Crushed			
- if double seam is significantly affected		X	
- if double seam has not been materially affected			X
Cut	X		
Delamination			X
Fracture	X		
Gels			X
Malformed			
- improper plastic end reform buckle, if rocker			X
- panel body affecting appearance but not integrity			X
- surface irregularity due to manufacturing having no bearing on integrity			X
- improper plastic end reform buckle, if not a rocker			X
Puncture	X		
Swollen package	X		

TABLE 8
CLASSIFICATION OF VISIBLE DEFECTS FOR PLASTIC PACKAGE
WITH HEAT SEALED LID
(NFPA - FPIG 23 APRIL 1986)

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
Abrasion - if halfway through		X	
- if less than halfway through			X
Burnt seal			X
Channel leak	X		
Contaminated seal		X	
Crushed - if affecting the seal area		X	
- if not affecting the seal area			X
Cut	X		
Delamination			X
Flexcracks - if on body			X
- if on lid			X
Foreign matter inclusion			X
Fracture	X		
Gels			X
Incomplete seal	X		
Label fold over			X
Malformed		X	
Puncture	X		
Seal width variation		X	
Swollen package	X		
Uneven impression		X	
Wrinkle			X

TABLE 9

**CLASSIFICATION OF VISIBLE DEFECTS FOR ASEPTIC PAPERBOARD
PACKAGES (NFPA - FPIG 22 APRIL 1986)**

	<u>CRITICAL</u>	<u>MAJOR</u>	<u>MINOR</u>
Abrasion - if significant		X	
- if slight			X
Channel leaker	X		
Corner leaker			X
Cut	X		
Crushed - if significant		X	
- if slight			X
Loose flap (or ear)			X
Misaligned seal - if significant		X	
- if slight			X
Perforation leaker	X		
Pulltab leaker	X		
Puncture	X		
Seal leaker	X		
Swollen package	X		

C. Methods For Creating Fusion Seams

This section will focus on hermetic fusion seams and exclude consideration of double seams for metal and plastic cans. Package types include flexible pouches, semirigid cups, and tubs which are retortable or aseptically filled and paperboard aseptic cartons.

Methods for creating heat seals were described by Young (1984) and may be summarized by the list shown in TABLE 10.

TABLE 10
METHODS OF SEALING (YOUNG, 1984)

Bar	Hot Melt
Band	Pneumatic
Impulse	Solvent
Hot Wire or Knife	Electronic
Ultrasonic	Magnetic
Friction	Induction
Hot Gas	Radiant
Contact	

The method of sealing is based upon economics and the physical properties of the packaging material. Bar sealing is the first choice because it is the simplest and cheapest method. Band sealing is used for pouches where speed is important and seal wrinkles are not a concern. Impulse sealing is used for pouches and has the advantage of permitting cooling in place before sealing jaws release. Unsupported films such as shrinkwrap may be sealed with a hot wire or knife. The resulting bead seal has an increased mass that often results in a seal stronger than the packaging material alone. Where paper separates a laminate structure, induction sealing has the advantage of creating heat within the aluminum foil adjacent to the sealant material. External heating will often burn paper

before the sealant melts and is not practical for aseptic paperboard cartons. If packages possess sufficient structural rigidity, friction sealing or spin welding may be used - providing the process does not damage oxygen barrier layers. Radiant sealing may be used for materials that would otherwise melt onto hot contact surfaces. Solvent sealing may work well for applications where solvents evaporate completely or react fully. They are not used in food packaging as toxic solvents may accidentally contaminate the product. Hot melt coatings are used extensively for peelable seals. These are applied to lidstock using extrusion coating or by adhesive mounting. No known applications of hermetic hot metal closures exist where material is injected into the seal area prior to closing the package. The last hole-in-cap metal can closure was used for evaporated milk. Iron containing compounds blended into polyolefins may be heated by magnetic fields to melt and form fusion seals. Tyvec may be sealed using radiation to induce heating of polyester, polypropylene, nylon, and polyolefins, (Young 1984). Hot gas whether direct or to heat a contacting roller may be used to fuse plastic films. This method is used to apply the longitudinal back strip to aseptic paperboard packages.

Fusion seals require specific conditions of time, temperature, and pressure. This is true whether the material being sealed is supported or unsupported. The consistency of hermetic seals depends upon the shape of the sealing bars, correct conditions of time, temperature and pressure, and the absence of contamination in the seal area. Once molten, the sealant material flows. The interface that previously existed disappears in fusion. Pressure is essential for the molten sealant to redistribute itself evenly. The process of redistribution may be enhanced when curved sealing surfaces are used. By compressing the seal area, the molten sealant flows outward

transporting contaminates and filling voids. Once cooled, the extruded sealant forms a bead or dike which is greater in cross section than the thickness of the fused seal. This is shown in FIGURE 1.

A slight deformation of seals is also employed to reduce wrinkling if the package is not supported or held in tension by grippers. To obtain the pressures required to seal through citrus pulp, vegetable fibers, or meat particles, some machine designers have found that a single ridge centered on the sealing dies can be useful. The cetrelli bars used on Brik Pak's AB-9 sealing jaws and the visual inspection ring used on semirigid cups with flexible laminate lidstock material are examples.

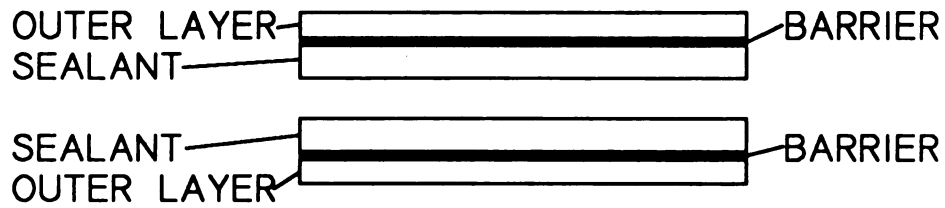
D. Defects Occurring In Fusion Seals And Their Causes

Packaging defects stem from four primary sources:

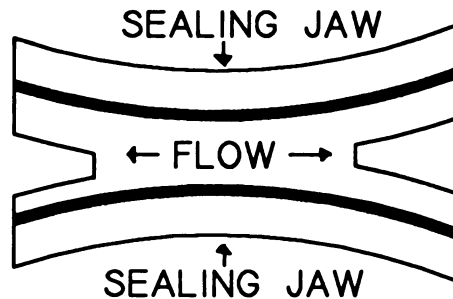
1. Product
2. Packaging Material
3. Sealing Machine
4. Human Error

Product in the seal area creates many problems contributing to defective seals. Water droplets deposited in the seal area of packages with fusion seals may be the result of product splattering under the force of the steam evacuation or be the result of condensation. When trapped at the interface in molten sealant, these tiny droplets expand to their equivalent volume of steam at the temperature of the hot plastic. When pressed flat by the sealing process, the surface area covered by the expanded droplet becomes significant. In some cases expanding steam forces the fluid sealant beyond the normal edge of the seal leaving a void within the seal

TWO OPPOSING SURFACES OF MULTILAMINATE MATERIALS.



EXTRUSION PROCESS CAUSED BY PRESSURE DURING THE SEALING PROCESS.



RESULTING SEAL WITH SEALANT DIKES.

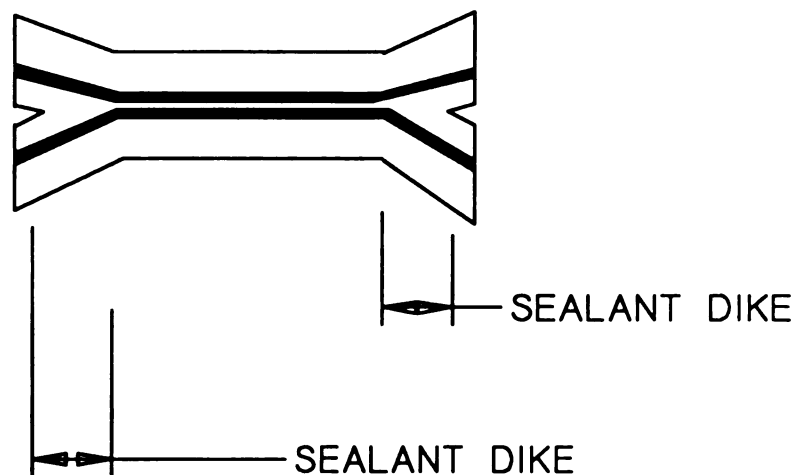


FIGURE 1 - FUSION SEALS

area. Often tiny moisture droplets expand uniformly forming circular patterns randomly distributed within the seal. After the seals cool and the steam condenses, these voids collapse making the depression visible from both sides of the seal. Cups with fused lids display similar defects. Aseptically filled paperboard packages overcome this problem by using extreme force to displace water film from the sealing surface prior to energizing with radio frequency energy and melting the polyethylene sealant. Grease droplets expand in a manner similar to water droplets when heated. In the case of grease, the degree of expansion is not as great as moisture. After cooling, grease blisters collapse and are often visible from both sides of a seal.

Grease contamination may sometimes be visually distinguished from moisture droplets by the shape of the blister left in the seal area. Water droplets generally leave blisters which are round and have smooth edges. Grease contamination creates irregular shaped blisters with rougher edges.

Food fibers and particles present in the interface of a seal commonly result in an open channel leading to the product. The product may dry to become a plug or remain moist forming a wick for bacterial penetration. Fiber, pulp and small flecks of product create major and critical defects if they occlude more than half of the width of the seal. For retortable pouches formed by a hot bar sealer, the requirement for a hermetic barrier is a 1/8 inch wide tortuous path along the top seal which extends fully from one side seal to the opposite side seal. For retortable pouches sealed with an impulse ribbon this requirement is 1/16 inch. (MIL-STD 32-74 Retort Pouches.) The distinction is purely historical. When the determination was issued by the U.S. Army Natick Laboratories, most resistance sealing

bars were 1/4 inch and most impulse sealing bands were 1/8 inch. No minimum seal width standards for paperboard packages or cups with flexible lids have been set by USDA or FDA. Guidelines proposed by the NFPA-FPIC recommend the following definition.

Thermoformed Containers With Heat Sealed Lid
Contaminated Seal - foreign matter in the seal area such as, but not limited to, water, grease, food, where the effective closure seal is reduced to less than 1/16 inch. The effective closure seal is defined as any uncontaminated, fusion bonded continuous path, and minimum of 1/16 inch wide from inner to outer edge, that produces a hermetic seal (Genske, 1986).

E. Testing Of Flexible Packages

The current lack of guidelines published in 21CFR is being addressed both by industry and by the regulatory agencies. The NFPA-FPIC guidelines for visually detectable package defects are a major contribution in this regard. Guidelines for fusion seal width are being developed by the USDA. A summary of testing devices and methods to verify package integrity will illustrate the current state of the art of testing hermetically sealed packages containing low acid foods. (TABLE 11) Classifications are debatable and the summary shown in TABLE 11 are the author's interpretations based on the state of the art and personal experience. Future innovation will result in reclassifications and new categories of test methods.

Non-destructive test methods have a significant economic advantage. Package testing is expensive and time consuming. Sampling provides only statistical inference and leaves the concern for unforeseen risks as a burden for the food processor. Food processors desire low cost and low risk

packaging. Inference by non-destructive testing has severe limitations. The true test is hermetic integrity and the ability of a package to withstand distribution requirements. Detecting holes as small as 0.2 microns and stressing packages to measure strength at line speeds is not possible today. However, techniques which remotely sense important indicators or uniformly stress some packages to non-destructively measure a threshold value are available. The reliability of these devices during manufacturing is difficult to ascertain when package defects include a broad range of characteristics. A simple testing device senses only one variable. When the response is positive or negative with no indecision the method is objective. Objective methods are nonparametric in the statistical sense. Results are black or white, good or bad, accept or reject. There is not opportunity for indecision. The limited number of viable indicators for package integrity is reflected by the small number of tests that may be automated. A summary of these methods, including commercially available devices is contained in Appendix B. There are four possible methods identified in TABLE 11 which may be developed into non-destructive on-line leak detection devices for aseptic composite packages. These include compression testing, gas leak detection, sensing metal by a proximity detector and vacuum measured over a period of time. All four methods are employed to evaluate filled and sealed packages on the production line.

1. Compression stresses a package by placing an external force in such a manner as would cause the package to change its shape or rupture. The disadvantage is in causing damage to packages which may result in their failure during distribution.

TABLE 11
CLASSIFICATION FOR TEST METHODS

	<u>Non- Destructive</u>	<u>Simple</u>	<u>Objective</u>	<u>Automated</u>
<u>TEST METHODS</u>				
1. Air leak testing	yes	yes	yes	no
2. Biotesting	no	no	no	no
3. Burst testing	no	no	yes	no
4. Chemical etching	no	no	yes	no
5. Compression testing	yes	yes	yes	yes
6. Distribution (abuse) test	no	no	yes	no
7. Dye penetration	no	no	yes	no
8. Electester	yes	yes	no	no
9. Electroconductivity	no	yes	yes	no
10. Gas leak detection	yes	yes	yes	yes
11. Light and lasers	yes	yes	no	yes
12. Machine vision	yes	yes	no	yes
13. Proximity tester	yes	yes	yes	yes
14. Sound	yes	yes	no	yes
15. Tensile testing	no	no	yes	no
16. Vacuum testing	yes	yes	yes	yes
17. Visual inspection	yes	no	no	no
18. Incubation	yes	no	yes	no

2. Gas leak detection is a passive method that relies on the partial pressure of gas at a higher concentration within a package to pass through holes in the package and into an atmosphere at a lower concentration of that gas. Sensors may detect helium, argon, xenon, ethylmercaptan, or carbon dioxide. The disadvantages include a method of trapping the gas within the package, reaction with food products, toxicity, regulatory restrictions, blocked holes, and a lagging response time.

3. Sensing the location of foil or a metal lid is possible using a metallic or frequency specific proximity detector. When pressure is exerted external to a fusion seal and the seal fails, the package experiences a slight increase in volume. The change in the position of a flexible portion of the package may be detected. Movement of the package indicates a loss of hermetic integrity.

4. Vacuum may be used to distort a flexible lid. This may enhance the ability of other methods to identify variation among packages which do not otherwise fall outside of the normal range of responses. A leaking package will behave differently from a properly sealed package when tested under a pressure differential sufficient to create a deflection in one or more parts of the package. This is also true when a gas or liquid may be forced through a small orifice and cause distortion of one or more parts of a package.

There are a number of commercially available testing devices which enable a food processor to test packages nondestructively on-line. Currently, none of these devices or test methods fulfill the requirements of regulatory agencies in a manner that precludes sampling and destructive testing. According to Bandes (1988) it can safely be said that no one

technology will test all types of leakage. While development continues, both the regulatory agencies and food processors seek methods which permit confidence in the hermetic integrity of those low-acid food packages being produced today. There are a number of test methods which permit commercial activities to continue within what experience has determined to be an acceptable range. For aseptic paperboard packages, the current test methods are: manually peeling fusion seals, electroconductivity, and dye testing. Incubation of all or a portion of production is used by most aseptic food processors to verify package integrity. Tensile testing and burst testing are not used to test these packages during the form/fill/seal operation. This document will focus upon the mechanical methods of determining fusion seal strength. Other methods are described in Appendix B. It is assumed that a mechanically strong seal is less likely to lose hermetic integrity in distribution than a seal that displays minimal strength. Because the burst test stresses all points within a closed package equally, it is well received by regulatory agencies as a desirable test method. A short review of the origins of test methods for retortable pouches will serve to illustrate why the burst test and the tensile test are favored as techniques for measuring seal strength.

The first proposed application for a flexible package containing shelf-stable, low-acid food was made by Schultz (1973) following experiments by Duxbury (1970). Package testing was one portion of a scale-up, simulating form, fill, and seal manufacturing of Meals Ready to Eat (MRE) Rations at Swift and Company in Chicago under contract with the U.S. Army Natick Laboratories. According to Lampi et.al. (1976):

"The internal burst test for seal integrity is a good overall measure of the ability of a package to withstand transportation and handling. The prime advantage of this test is its ability to detect the weakest part of the seal."

Lampi et.al. (1976) proposed the following criteria for retort pouch seals,
TABLE 12.

TABLE 12
SEAL CRITERIA FOR THE RETORT POUCH

Fusion must exist
Burst test 20 psig for 20 seconds with a
maximum seal separation of 1/16 inch
Restrained thickness for burst test 1/2 inch
Tensile strength 12 pounds per 1 inch sample
with a crosshead speed of 20 inches per minute
and a sample width of 1/2 inch
No visible abrasions exist

In specifications for burst test strengths used by retort pouch manufacturers and packers, the military required 30 psig for 30 seconds prior to retorting and 20 psig for 30 seconds after retorting. A maximum separation of 1/16 inch was given in MIL- STD 32-74. This requirement reflects a weakening of fusion seals during thermal processing.

Polvino (1986) evaluated various methods for burst testing retortable semirigid plastic containers with peelable and/or fused lids used for low-acid foods. He proposed the following variables to be defined when burst testing. See TABLE 13.

TABLE 13
BURST TESTING VARIABLES TO BE DEFINED
SEMRIGID RETORTABLE FOOD CONTAINERS

Fused or peelable lidstock
Retorted or not retorted
Sealing conditions (time, temperature, pressure)
Type and degree of confinement
Rate of pressure increase
Ratio of surface area to container volume
Dynamic or static burst test method

During his experiments Polvino determined that the maximum allowable expansion for flexible lids should not exceed 1/4 inch. When reporting test results the sample size, average, and standard deviation should be reported. Polvino used a dynamic test method. Dynamic in this sense means that packages were subjected to an ever increasing internal pressure until they ruptured. No mention is made of an allowable seal separation under pressure as is permitted with retortable pouches under MIL-STD 32-74. However, weakening of the fusion seal by thermal processing was noted with retortable cups just as with retortable pouches. This is shown in TABLE 14.

TABLE 14
DIFFERENCES IN BURSTING STRENGTH OBSERVED
IN RETORTABLE SEMIRIGID CONTAINERS BEFORE
AND AFTER THERMAL PROCESSING (POLVINO, 1986)

	<u>Before Thermal Process (lb/in)</u>		<u>After Thermal Process (lb/in)</u>	
	<u>Mean</u>	<u>s.d.</u>	<u>Mean</u>	<u>s.d.</u>
Lidstock Sealed at 335°F				
8 fusion	12.75	1.89	10.75	0.5
8 peelable	10.	no data	9.5	0.58
Lidstock Sealed at 375°F				
8 fusion	20	no data	19.	1.85
8 peelable	13	2.62	11.5	2.0

There are a number of commercially available burst testing devices. The features that are common to these devices are shown in TABLE 15.

TABLE 15
FEATURES FOR BURST TESTING DEVICES

Injection port or adaptor for air or water entry into
the package Gross pressure regulator
Fine pressure regulator
Solenoid with timer
Pressure gauge
Septum seal at injection port to prevent air leaking
during the test
Restraining device (optional)

Lampi, et.al. (1976) described the method for preparing and testing retort pouches using a burst testing device which tests three seals only.

"an empty or emptied pouch is placed over an air source, the jaws are clamped to seal the pouch around the air source, and the internal pressure is increased (at a constant rate) to a predetermined level. Either the pressure to burst, time to burst at a constant pressure, or withstanding a preset pressure time cycle is recorded."

For plastic cans, Narish Swaroop, Ph.D. of Central States Can Company (1986) recommends burst testing with a pressure greater than the retort operating pressure. Polvino (1986) used lower burst values for retortable cups. Lampi, et.al. (1976) recognized that restraining the thickness of the retort pouch was necessary for burst testing. During thermal processing, pouches in retort racks are restrained to a vertical dimension of 3/4 inches. The Reycon burst tester restrains pouches to 1/2 inch. Damage by insufficient overriding air pressure during thermal processing may be avoided by restraining flexible and semirigid packages. Although the requirement for pouch restraint during thermal processing was understood as early as 1970 by Duxbury (1970), described by Lampi, et.al. (1976), and made a requirement for M.R.E. processors in 1979 by MIL-STD 32-74, it remains a problem today. Florren Long of Ludlow Corporation reported that 60 percent of retort pouch failures seen in a recent tour of MRE packer plants for the Research and Development Associates for Military Foods and Packaging Systems (R.&D.A.) could be attributed to internal pressure or abuse. Leaks in the corners of pouches, excess headspace gas, and insufficient overriding air pressure during thermal

processing, along with cuts and punctures were responsible for the loss of the hermetic barrier. (Long, 1987).

Lampi, et.al. (1976) described a reduction in bursting strength for retort pouches at various stages of manufacturing which are shown in TABLE 16. When food contamination, moisture or foreign materials occur in the fused seal area, the burst strength of retort pouch seal strength diminishes during storage, as shown in TABLE 17.

TABLE 16
BURSTING STRENGTH FOR RETORT POUCH SEALS
(Lampi, et.al. 1976)

Pouches restrained to 1/2 inch for test

Material: polyester/aluminum foil/modified polyolefin

Immediately after sealing	35 psig,	30 seconds
24 hours after sealing	30 psig,	30 seconds
After thermal processing	20 psig,	30 seconds
Indefinite storage after thermal processing	20 psig,	30 seconds

TABLE 17
BURSTING STRENGTH FOR CLEAN AND CONTAMINATED RETORT
POUCH SEALS AFTER STORAGE
(Lampi, et.al. 1976)

Measurement Time	<u>0.32 CM WIDE SEAL</u>		<u>0.64 CM WIDE SEAL</u>	
	Without Particles psig	With Particles psig	Without Particles psig	With Particles psig
Before retort	40	39.5	49.5	45.0
After retort	28.3	29.8	25.6	29.8
6 months	23.4	12.8	25.6	22.0
12 months	21.0	13.0	26.0	21.0

The method for restraining retort pouches during testing was described as a heavy metal plate which restricts the thickness of a retortable pouch to 1/2 inch. Lampi, et.al. (1976). Restraint during burst testing has advantages which will be discussed later.

There are some disadvantages involved with burst testing food packages. Burst testing is a destructive test requiring off-line testing devices and personnel trained to conduct testing. Empty retort pouches may be tested on three seals using the Reycon, Continental Can and Aro burst testing devices. They may be tested on four seals using the FMC or Aro burst testers and packages must be emptied and washed prior to testing. Product creates problems for burst testers. Product plugs small holes in test packages and fills air lines obstructing pressure gauges. Catastrophic failure is often required for a burst package to register on these testing devices. Air bursts at 35 psi creates a loud noise disruptive to line workers, even in noisy packaging areas. If packages are opened and rinsed prior to testing a sink is required. Both the sink and the testing device require cleaning. When testing is conducted adjacent to the packaging line, unretorted food may be deposited back into the filling machine. Few processors and many inspectors for regulatory agencies prefer to see the product discarded. The expense of discarding good product is undesirable for food processors. The requirements for a burst test for aseptic composite packages should include the following points contained in TABLE 18.

TABLE 18
REQUIREMENTS FOR A BURST TEST FOR PAPERBOARD ASEPTIC
PACKAGES

Failure in a normal manner.
Failure at the weakest point
Distinguish between normal and
defective packages
Reproducible results on a reliable basis

Handling and sample preparation can influence test results. Conveying samples without regard to edge damage will contribute defects not normally attributed to packaging machinery or bad packaging materials. Food particles remaining in packages during burst testing will affect sensitivity as previously stated. Correct design of test apparatus is essential if package failure is to be indicative of defects.

Rampart Packaging, a manufacture of plastic retortable and aseptically fillable cups and cans, uses burst testing on-line and states that burst testing is a good test. (Marcy, 1985). According to Polvino (NFPA):

"The burst test is good for retortables. For non-retortables (aseptic packages) the electroconductivity test is preferred. The burst test will not identify microleaks. In general a burst test indicates seal strength. Strong seals are more likely to pass distribution than weak seals." (Polvino, 1985).

Burst testing has been judged not to be reliable in predicting the performance of aseptic paper box containers in distribution according to

DeGeronomo, (1986) of International Paper Company. Tetra Pak, the best known supplier of aseptic paperboard boxes does not want to see a requirement for a burst test.

"A burst test delaminates the Tetra package and does not break at the seam." (Sizer, 1985)

The sections which follow, outline the procedures and results for burst Testing 250 ml brik paks. This information will show that a burst test may be applied to paperboard composite aseptic food packages. The specific points which will be elaborated in the Discussion section illustrate the following points (TABLE 19).

TABLE 19
BURST TESTING

1. Burst testing measures seal strength.
2. Burst testing results in failure at the package seals.
3. Burst tests conducted during manufacturing may be used to identify when the sealing device is producing strong or weak seals.
4. Lots on hold may be separated into groups of "good" and "bad" using a burst test to measure seal strength.
5. Accepted statistical methods may be employed to provide confidence in accepting or rejecting lots.

F. Application Of A Burst Test To Aseptic Composite Packages

A burst test stresses a package uniformly in all directions. Stress applied to the weakest point usually results in package failure. Therefore, the burst test may be used to identify the location of the weakest point and the pressure at which it fails. If the force causing failure is known and the

pressure is reliably controlled under the right conditions it may be possible to create a pass/fail test for packages. Those which pass should retain their hermetic integrity. Those which fail may be classified according to the mode of failure. By studying the packaging equipment, material structure, and conditions, a test should provide insight into the nature of the defect which precedes a package failure. When cause and effect are understood, machine operators may be able to correct the cause and eliminate many package defects. The burst test, because it stresses all points of a package, offers the widest spectrum for identifying defects.

MATERIALS AND METHODS

Samples of 250 ml aseptically formed, filled, and sealed Tetra Pak "brik paks" were obtained from the production line and warehouse of Squirt Pak International located in Holland, Michigan. The structure of the Tetra Pak brik pak is shown in FIGURE 2. Samples were produced on a Tetra Pak model AB-3 vertical form, fill, seal machine. The condition of samples were first determined by electroconductivity and dye testing as described in Appendix B. Visual inspection consisted of measuring packages to certify that they meet specified design parameters and manual separation of the seal to detect areas lacking the required fusion. Burst testing was conducted both in the packaging room at Squirt Pak International under ambient conditions and later at the School of Packaging at Michigan State University in a controlled environment at $72 \pm 2^\circ\text{F}$, $50 \pm 5\%$ R.H.

Before burst testing, samples were emptied using the vacuum trap apparatus shown in FIGURE 3. Emptying brik paks prior to burst testing is necessary to insure that liquid food products do not enter the burst testing device and alter the response of the controllers and pressure gauges. A

POLYETHYLENE SURFACE COATING
PRINTING
CLAY COATING ON PAPER
BLEACHED PAPER
POLYETHYLENE
ALUMINUM FOIL
SURLYN OR POLYETHYLENE
POLYETHYLENE SEALANT

FIGURE 2 - STRUCTURE OF 250ml BRIK PAK

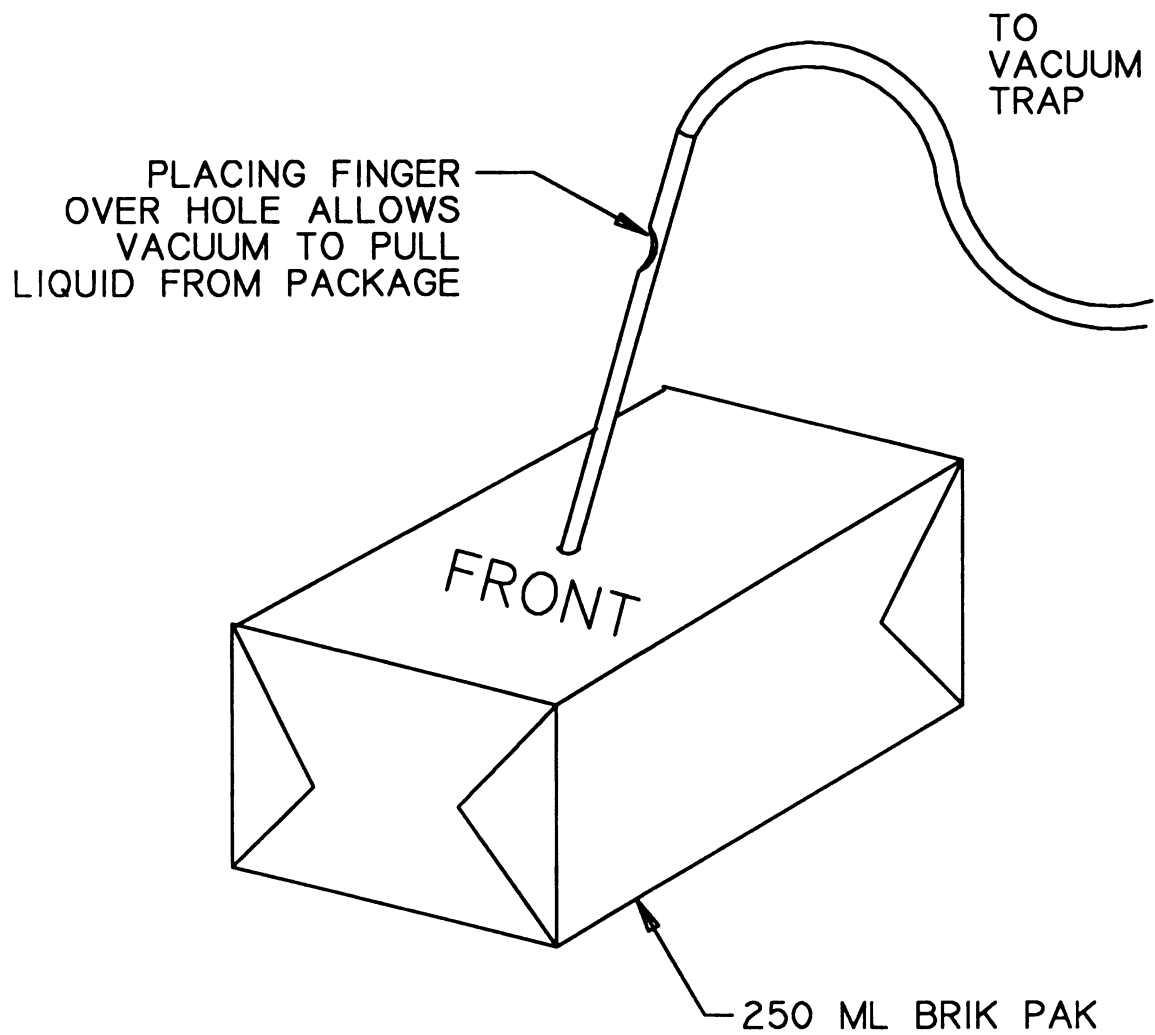
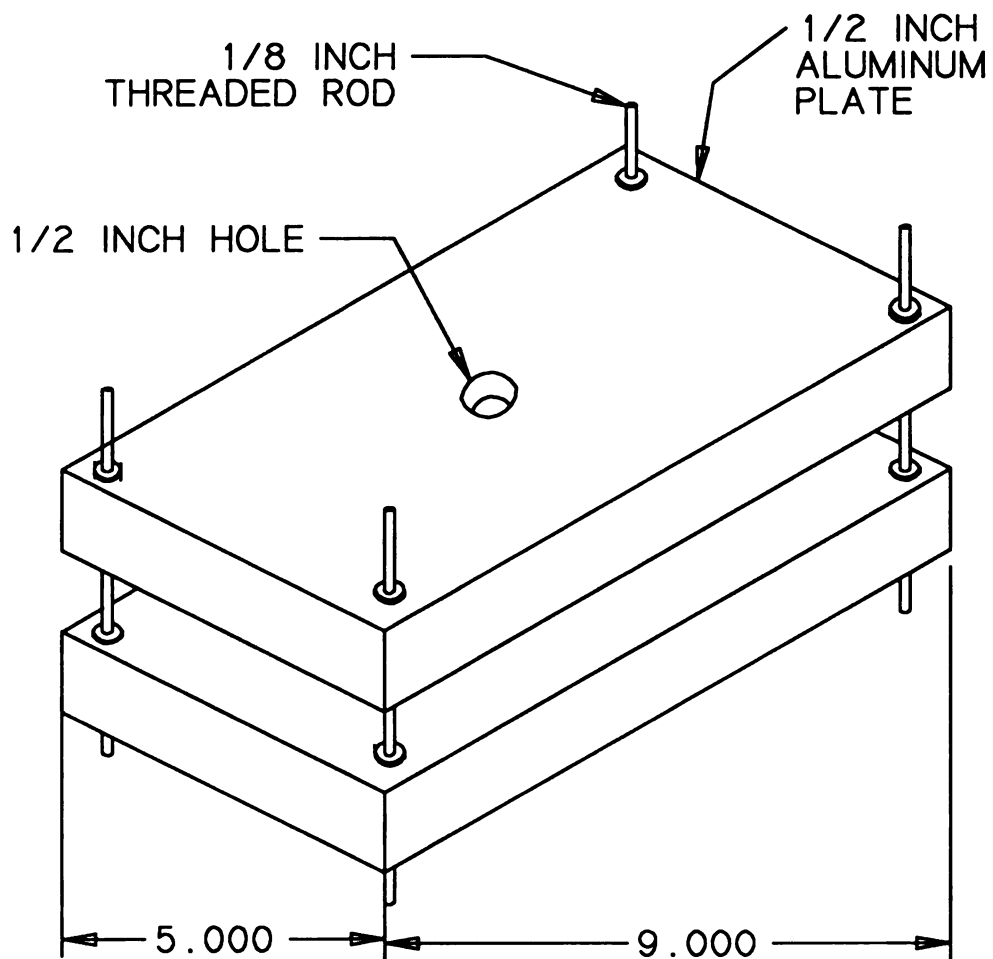


FIGURE 3 - REMOVING PRODUCT FROM BRIK PAK

restraining device, shown in FIGURE 4 was used to prevent expansion of samples during burst testing. A hole in one of the flat, parallel aluminum plates permits inserting of the Aro Model F100-1320 double needle fixture. Pressure (0-100 psig) is created by an Aro reversible pump. The airflow is controlled by an Aro Model F100-1380-3 console which regulates the inflation rate of 1 psig/second. The pressure gauge reads from 0 to 50 psig. Two moveable indicators display the pressure on the gauge. The first is equipped with a small post which pushes the second. The second indicator is designed to stop when the first reaches maximum pressure. This allows the user to observe the maximum internal pressure following the burst failure of a sample, or observe the maximum pressure within the sample if the internal pressure is maintained for an extended period of time.

A clear plexiglass twenty gallon aquarium, 24 x 12 x 12 inches was used to control the dispersion of particles ejected by brik paks which exploded during the burst test. Because the explosion is loud and disruptive to personnel working in the area a sheet of clear plexiglass was placed over the top of the aquarium. The explosion muffle and other test apparatus were contained on a three tier stainless steel cart. Grounded extension cords supplied electrical power from wall sockets. Because the plant production area was wet, the connections between extension cords were taped with waterproof electrical tape as a precaution against shock.

Samples were collected from the production line and warehouse. During the manufacturing operation, machine operators and quality control inspectors visually examine brik paks every 15 minutes. In addition, every 30 minutes one package from each set of sealing jaws is manually measured and undergoes electroconductivity and dye testing to assure the hermetic integrity of the brik pak. The Tetra Pak AB-3 possesses



PLATES ARE SPACED USING
NUTS (NOT SHOWN).

FIGURE 4 - RESTAINING DEVICE FOR BURST
TESTING 250ml Brik Paks

two sets of sealing jaws which form transverse seals on the 250 ml brik paks. Seals are manually separated while the operator carefully observes the separation of the inner polyethylene plies. This is done to determine that fusion in the seal areas are complete. Quality control inspectors perform these same tests every 30 minutes alternating with the machine operator. This provides a verification of the critical package parameters by the establishment every 15 minutes during continuous operation. More frequent tests are conducted when the operator makes adjustment to the AB-3 during operation. Normally, adjustments are made without stopping the vertical form, fill, and seal machine. Stopping increases the potential for producing nonsterile packages so monitoring of the critical control parameters serves to maintain package integrity while the production flow is maintained in a steady state. Samples for burst testing were removed every 15 minutes from the production line.

Samples collected from the warehouse are generally identified following Military Standard 105-D or the square root of the number of packages in a production lot. A random sampling method is required if statistical validity is desired. However, samples used in burst testing were obtained by Squirt Pak warehouse personnel, and may not conform to either method of identifying samples. Samples collected represented an opportunity to conduct burst testing on samples of known and unknown hermetic integrity following a period of "incubation" during which a portion of a production lot containing defective packages was placed "on-hold" by Quality Control inspectors. During the manufacturing process, a number of 250 ml brik paks with defective seals were discovered. Two cases of product (27 brik paks) designated "known bad" were obtained. Following adjustment to the AB-3 machine two cases of product designated

"Unknown #1" were obtained. After a short period of time the operator determined that a second adjustment to the AB-3 was required. Following the second adjustment, two cases designated "unknown #2" were obtained. As production continued, Quality Control personnel monitored package integrity. When tests conducted by Squirt Pak's Quality Control personnel revealed consistently good hermetic integrity, the transition points preceding and following the time when defective packages occurred were identified. Pallet loads were relocated segregating normal product from questionable product. Quality Control "HOLD" signs were applied to the product in question before pallet loads were transferred to the warehouse for subsequent observation.

Student's t-test was used to compare the burst test results of both of the samples identified as "unknown" with samples identified as "known good" and "known bad". Because the samples represented only two cases each (27 brik paks) ANOVA or Duncan's multiple range test was not employed in evaluating results. If a larger sample population were available, these latter statistical tests would be beneficial. Testing conducted at the School of Packaging was undertaken to determine whether internal or external pressure application would influence burst testing. Both static and dynamic testing was employed with the objective of determining whether or not the static burst test criteria developed for the retort pouch would be applicable to 250 ml brik paks. External pressure was created by screw driven compression platens on a Baldwin Impac model SR-4 platen press. Sealed 250 ml brik paks were placed on end, on their sides, and laid flat on their largest surface while being compressed by force applied external to the package. One package at a time was compressed in this manner. Because each sample contained liquid food

product (with no headspace) all samples were contained in large sealed plastic bags to retain squirting liquids. Static and dynamic testing modes were used to determine how the 250 ml brik paks might respond. A static burst test inflates the package at 1 psig/second until a predetermined pressure is attained. The pressure is then held constant for a specified period of time. If a loss of pressure is observed during the test, the sample has failed. Fusion seals sometime separate during the test without an observed loss in pressure as displayed by the gauge. For retortable pouches restrained at 3/4 inch and inflated at 1 psig/second, the preretort static burst test requirement is 30 psig for 30 seconds. Following thermal processing, this requirement is 20 psig for 30 seconds. A seal separation in excess of 1/16 inch at the edge of fusion is unacceptable even though the package has withstood the internal pressure. A 1/16 inch separation of the sealant at the inner edge of fusion renders the sample a failure under these test conditions.

Dynamic Testing describes the inflation of a package at a constant rate until failure is observed. Usually failure of the package obliterates the point of failure so separation less than 1/16 inch in a fusion seal is not generally detectable. For dynamic testing of 250 ml brik paks, the inflation rate is 1 psig/second until failure. The sweep hand of the pressure gauge displays the maximum internal pressure prior to failure.

Both air pressure and liquid pressure may be used for burst testing. The use of liquids in hydrostatic and hydrodynamic testing has the advantage that failure of the closed container will not be accompanied by a loud explosion. In addition, there is less likelihood that liquid will spray from the failed package unless it is through a restricted opening.

The procedure for burst testing using the Aro equipment is shown in TABLE 20. A list of materials is shown in TABLE 21.

TABLE 20
PROCEDURE FOR BURST TESTING

1. Obtain sample.
2. Detach corner flaps and visually inspect for external defects.
3. Puncture geometric center of front panel with siphon tube.
4. Place finger tip over hole on side of siphon tube to permit vacuum to form.
5. Siphon liquid contents into vacuum trap.
6. When the brik pak is empty and becomes flat break the vacuum by removing your finger tip from the vent hole on the siphon tube.
7. Remove the siphon tube from the flatened brik pak.
8. Place the brik pak in the restraining device and insert the needles into the hole in face of brik pak. As air enters the sample it inflates sealing itself against the needle apparatus.
9. Slide the cover over the plexiglass aquarium.
10. Press the "test" button on the control console and observe the sample as it inflates at 1 psi/sec. Observation will sometimes reveal seal separation as it occurs. Note the location of any seal failure.
11. After the package seals rupture, examine the point of failure.
12. Record observations and peak pressure at failure.
13. Reset the sweep needle on the control console and place a fresh sample in the restraining device.
14. Burst 10 samples at one restraint height before changing the distance of the aluminum plates.

TABLE 21
MATERIALS USED FOR BURST TESTING

250 ml Brik Paks, formed, filled with liquid product sealed.
Aro Model F100-1380 Burst Testing Device
Aro Model F100-1320 Package Holding Device
Plexiglass 20 gallon aquarium (24 X 12 X 12 inches), 1/2" thick plate
Plexiglass plate 24 X 12 X .125 inches
Aero Model F100-1320 Double Needle Restraining device
Tigon plastic tubing

DATA AND RESULTS

Emperical testing using a platen press was first used to identify factors which influence the method of seal failure for brik shaped, aseptically filled paper/poly/foil packages. The effects of external pressure on container failure was observed and compared to observations of the effects of internal pressure on container failure.

A. External Pressure

Ten filled brik paks were crushed between moving platens and the method of failure observed. Filled cartons were placed within a plastic bag to retain the liquid contents which sprayed from the point of failure. Results are shown in TABLE 22. Orientation of packages is shown in FIGURE 5.

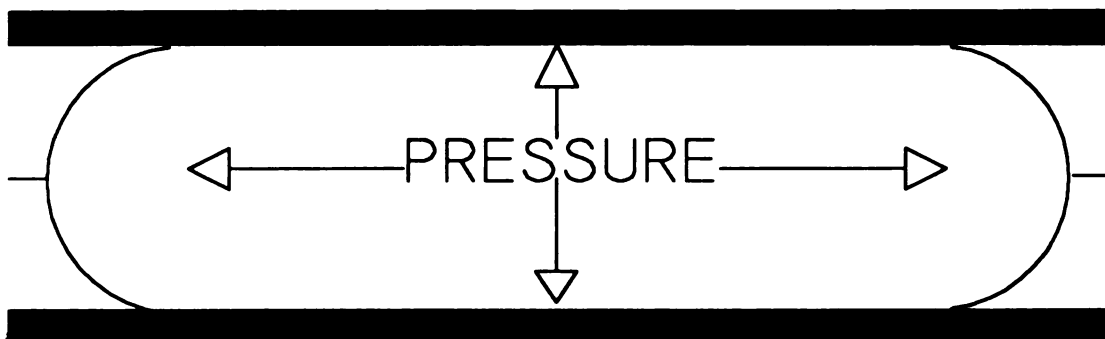


FIGURE 5 - 250ml BRIK PAK WITH INTERNAL
PRESSURE RESTRAINED BETWEEN
METAL PLATES

TABLE 22
COMPRESSION FAILURE OBSERVATIONS

<u>SAMPLE NUMBER</u>	<u>CONFIGURATION</u>	<u>LOCATION OF FAILURE</u>
1	Standing on top	Leak through
2	Standing on bottom	longitudinal seal
3	Lying on left side	
4	Lying on right side	Corner longitudinal
5	Lying on front	seal
6	Lying on back	Bottom transverse
7	Lying on back	seal
8	Lying on back	
9	Lying on back	Top transverse seal
10	Lying on back	Bottom transverse seal

When the brik paks are stood on the smallest end and crushed by a moving platen, the circumference of the package increases until the package ruptures at the longitudinal seal. When brik paks are laid on their side, failure occurs at a corner or in the longitudinal seal. When the brik paks were placed on their front or back and crushed, the folded ends unfolded as the platens were lowered. The internal pressure was relieved by the changing configurations of the package. Resistance to the crushing pressure did not begin until the brik pak had assumed a pouch configuration. The maximum resistance to crushing pressure occurred just prior to rupture of the container.

If the container was manually converted from the brik to a pouch configuration prior to crushing the container between the platens, the force required to rupture the package was greater. If the container was placed

between the platens in the brik configuration and crushed until the end flaps unfolded, the container was likely to fail at one of the corners before assuming the pouch configuration.

B. Internal Pressure

Liquid food products must be removed from brik paks before being burst tested to avoid forcing fluids into moving parts and pressure gauges. Ten empty brik paks were placed between stationary platens adjusted to contact both faces of the brik-shaped package as it is laid on its back panel. Air pressure is fed through the top platen and into the package through a needle. A second needle is used to permit the internal package pressure to be sensed. Upon inflation, the brik shape expands and the end flaps unfold as the container assumes a pouch conformation. All of the containers failed on the bottom transverse seal. Results are shown in TABLE 23.

TABLE 23
INFLATION FAILURE OBSERVATIONS - UNRESTRAINED

<u>SAMPLE NUMBER</u>	<u>PRESSURE AT FAILURE (PSIG)</u>	<u>LOCATION OF FAILURE</u>
1	16.0	Bottom transverse seal
2	14.5	Bottom transverse seal
3	12.0	Bottom transverse seal
4	14.5	Bottom transverse seal
5	10.5	Bottom transverse seal
6	15.0	Bottom transverse seal
7	11.0	Bottom transverse seal
8	14.8	Bottom transverse seal
9	10.5	Bottom transverse seal
10	13.4	Bottom transverse seal

The method of testing influences the point at which containers fail, the location of the failure, and the pressure required to induce failure. When the ten brik paks shown in TABLE 23 unfolded under the force of inflation, the bottom seal possibly received more stress than the top seal. The folded top seal tabs which are heat sealed to the side of the 250 ml brik pak detach before those which are heat sealed to the bottom of the transverse seals. This trend was not evident in brik paks which were manually converted to the pouch configuration prior to external or internal pressure testing. With external compression, the damage caused by moving platens is different if the containers are in the brik configuration, pouch configuration or in the process of unfolding. External pressure from moving plates focuses forces on the corners of the package as they change from the brik to the pouch configuration.

Similar forces exist with an internal or an external pressure test. But these forces are in opposite directions. The pressure within the container is the same using either method. However, the forces affecting the package are more uniform when platens are stationary and the package changes conformation by inflation. A test using inflation to achieve internal pressure will not be subject to the effects of secondary forces resulting from crushing. The internal burst test method is therefore preferred.

C. Comparison Of Pouch vs Brik Shape Upon Bursting Strength

The natural response of the containers to change from a brik shape to a pouch shape when pressure is applied was observed. To examine the effect of changing the configuration of the containers on burst pressure, a group of sealed packages were tested using differing profile thicknesses.

One hundred freshly sealed brik paks were removed from the production line and placed within the restraining device. The separation between the platens was 1.55 inches. This distance was selected because this is the front to back dimension of the 250 ml brik pak container when it is in the brik configuration. TABLE 24

TABLE 24
COMPARISON OF PACKAGE CONFIGURATION
TO BURSTING STRENGTH

<u>250 ml Brik Pak Configuration</u>	<u>Sample</u>	<u>$\bar{x}$ Burst Pressure (psig)</u>	<u>s</u>
Brik Pouch	50	11.00	1.53
	50	11.03	2.49

Forty percent of the containers tested in the brik configuration failed in the top transverse seal and 60 percent failed in the bottom transverse seal. All of the containers tested in the pouch configuration failed on the bottom transverse seal. Observation was made that by manually unfolding the brik pak containers, a test condition was created which produced greater consistency in the location of burst failures. When containers unfold by internal pressure, they are subject to secondary forces which contribute to premature failure due to unidentified causes. The test must be sensitive to the broadest range of response to the test stimulus. The range for burst failures of the pouch-shaped samples was greater than for the brik-shaped containers. The increased standard deviation in this situation indicates that there was a greater separation between the average bursting force of "good" and "bad" samples when in the pouch

configuration. When these packages are burst tested without first detaching the folded corners, they resist pressure up to the point where the tab seals release and the packages assume the pouch configuration. This movement is rapid, and may be accompanied by a rapid redistribution of forces contributing to hydraulic shock and a loss of resistance to pressure at the seals.

D. The Effect Of Restraining Samples During Burst Testing

Three tests were performed to determine the effect of restraining test samples. All samples were selected from the same production lot, represented normal production, and possessed acceptable seals. Product was removed from the containers at the time the samples were collected. Burst testing was performed at the production facility during the time of production and in the laboratory on the fourth and tenth day following production. Results are shown in TABLE 25. FIGURE 6 shows the relationship between the average bursting pressure and the distance between restraining plates. Failures occur when the internal pressure exceeds the strength of fusion or laminate adhesion along the container seal. As the plates are moved closer together the average bursting pressure increases. The standard deviation also increases. This reflects the observation that strong seals burst at greater pressures and weak seals, continue to burst at lower pressures. This is shown in FIGURE 7. The increasing range over which containers burst when restrained is significant. The separation between strong and weak seals becomes more apparent when packages are restrained during burst testing. The increase in the standard deviations reflects outlying values. These are lower burst pressures associated with packages with weak seals. Subtle defects in seal

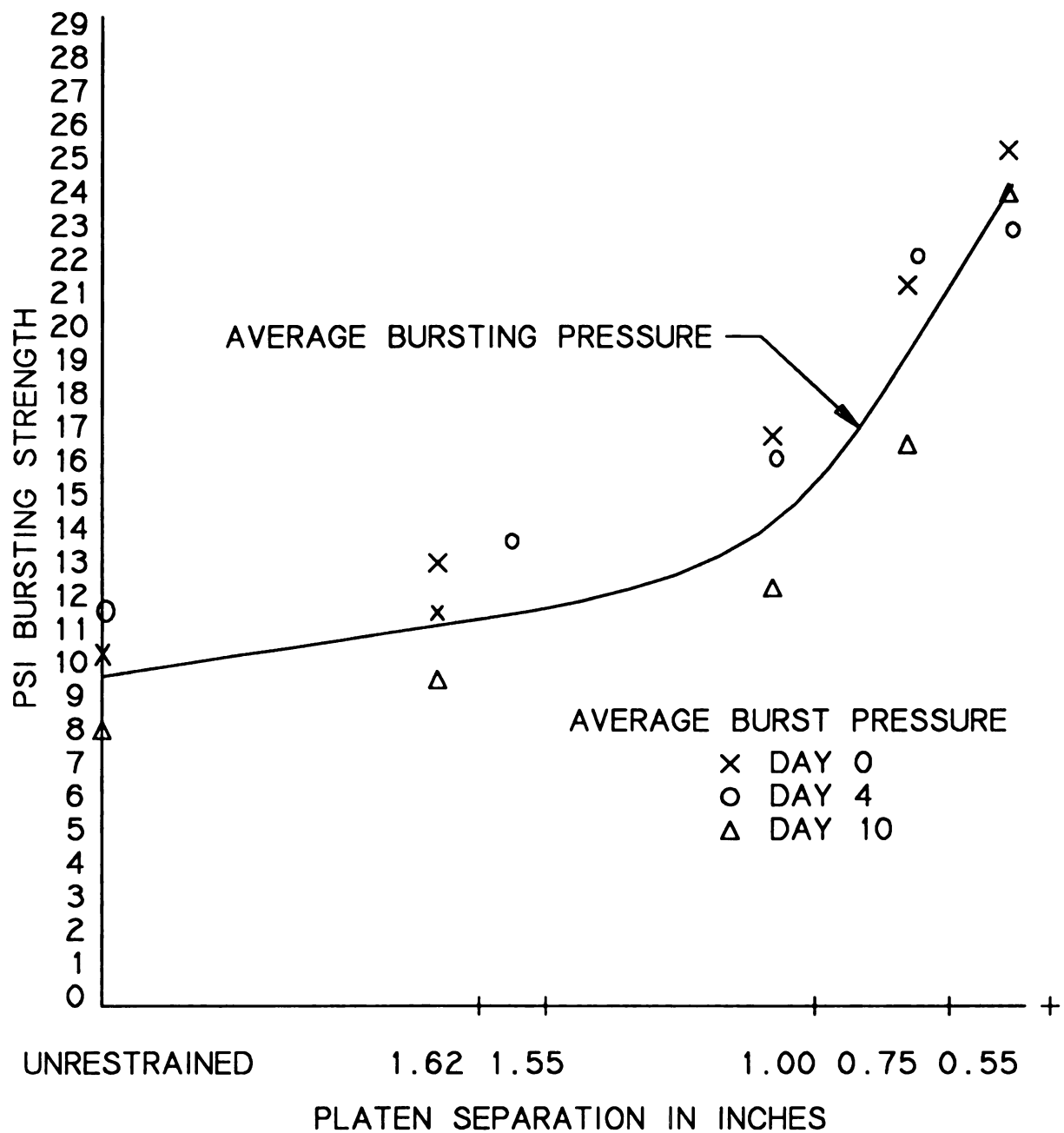


FIGURE 6 - RELATIONSHIP BETWEEN BURST PRESSURE AND OBSERVATION OF FIBER TEAR

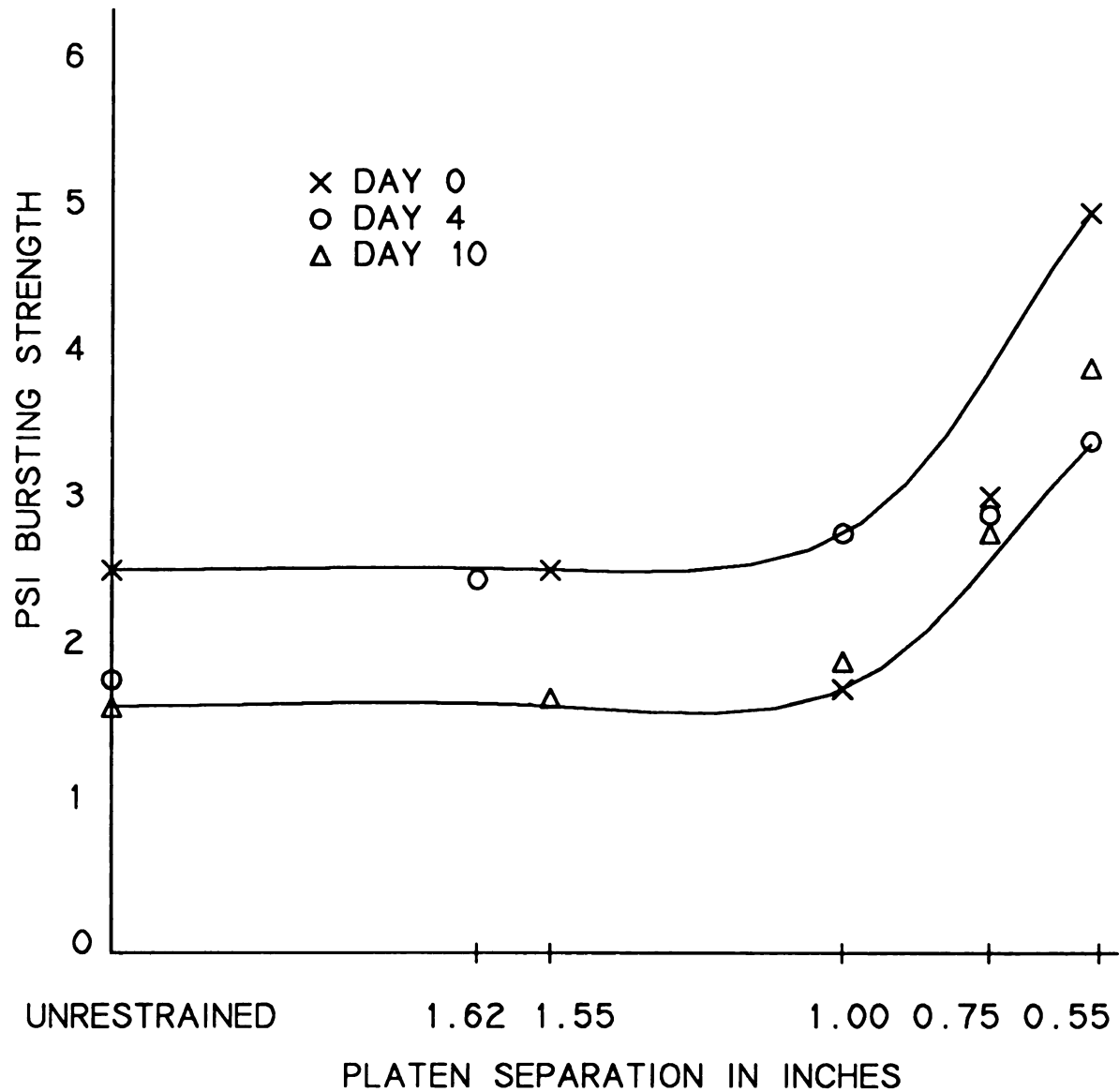


FIGURE 7 - EFFECT OF PLATEN SEPARATION ON STANDARD DEVIATION OF BURSTING PRESSURE

TABLE 25
EFFECT OF RESTRAINING SAMPLES DURING
INTERNAL BURST TEST

	n	<u>$\bar{x}$</u> (PSIG)	<u>s</u>
Day 0 on Production Line 75°F, 60%RH			
Unrestrained	10	11.03	2.49
Restrained at 1.55 in	10	13.23	2.06
Restrained at 1.00 in	10	15.54	1.65
Restrained at 0.75 in	10	21.91	3.29
Restrained at 0.55 in	10	23.42	4.74
Day 4 Laboratory Test 75°F 50%RH			
Unrestrained	20	10.55	1.81
Restrained at 1.62 in	10	10.35	2.10
Restrained at 1.00 in	10	16.30	2.72
Restrained at 0.75 in	10	20.60	2.97
Restrained at 0.55 in	10	24.75	3.24
Day 10 Laboratory Test 75°F, 50%RH			
Unrestrained	20	9.87	1.77
Restrained at 1.55 in	10	11.02	1.43
Restrained at 1.00 in	10	15.77	1.89
Restrained at 0.75 in	10	19.5	2.73
Restrained at 0.55 in	10	23.9	3.58

integrity are more easily distinguished when the difference in bursting pressure between the means for good and bad seal populations is at a maximum. When good and bad populations are distinctly separated, greater significance with statistical tests may be attained.

The containers which were restrained at .55 inches do not explode. Instead, minute channels open through which air or residual product is emitted. Normally when containers fail the entire seal area is destroyed. Interpretation of the remnant is difficult and subjective. When the failed seal is left mostly intact, the defect can be examined by locating it on the seal, applying dye, dissecting it and it may be possible to determine its cause. Thus, a major advantage of restraining containers during burst testing is to obtain information on the nature of the failure by examining the sample following testing.

E. Paper Tear as a Visual Indication of Seal Fusion

Many containers having lower than average burst pressures exhibit an absence of paper tear in the area where the seal fails. This phenomenon appears when separation occurs within the fused area. Separation sometimes occurs between the layers which would normally be made inseparable by the sealing process. This sealing defect is termed "non-fusion", "cold seals", or "seal blocking". The lack of fusion represents a major sealing defect which cannot be detected by visual inspection, unless the seal is opened by package failure or destructive testing. These defective packages could be released by the food packer into distribution to the consuming public.

Sixty containers were burst tested at various platen separations and the seals examined for paper tear. Samples were collected at the production facility, emptied and transported to the laboratory where they were held at $75 \pm 2^\circ\text{F}$, 50% $\pm 5\%$ RH for four days prior to burst testing. Results are shown in TABLE 26.

TABLE 26
PAPER TEAR AS AN INDICATOR OF SEAL FUSION

	<u>n</u>	<u>$\bar{x}$</u> (psig)	<u>s</u> (psig)
Unrestrained			
Total samples	20	10.55	1.81
With paper tear	15	11.27	1.40
Without paper tear	4	8.00	0.41
Leaking		1	
Restrained at 1.62 inches			
Total samples	10	11.35	2.10
With paper tear	5	13.20	0.76
Without paper tear	5	9.50	0.94
Restrained at 1.00 inch			
Total samples	10	16.30	2.72
With paper tear	8	17.25	2.17
Without paper tear	2	12.5	0.71
Restrained at 0.75 inch			
Total samples	10	20.60	2.97
With paper tear	9	21.06	2.76
Without paper tear	1	16.50	0.00
Restrained at 0.55 inch			
Total samples	10	24.75	3.24
With paper tear	6	25.42	3.56
Without paper tear	4	23.75	2.87

The difference in bursting strength of seals displaying paper tear and the absence of paper tear are displayed in FIGURE 8. To determine the effect of restraining containers on the amount of observable fiber tear along the fused seal area, containers were burst tested and the results displayed in TABLE 27 and FIGURE 9. An evaluation of "known good" and "known bad" briq pak for paper tear indicates two important factors. First, the bursting strengths of bad seals are on the average weaker, and secondly there is a great deal more variation in the amount and distribution of exposed paper in the "bad seals". Paper tear in "good seals" is more uniform, being usually a single unfragmented tear. Results are shown in TABLE 28 and FIGURE 10.

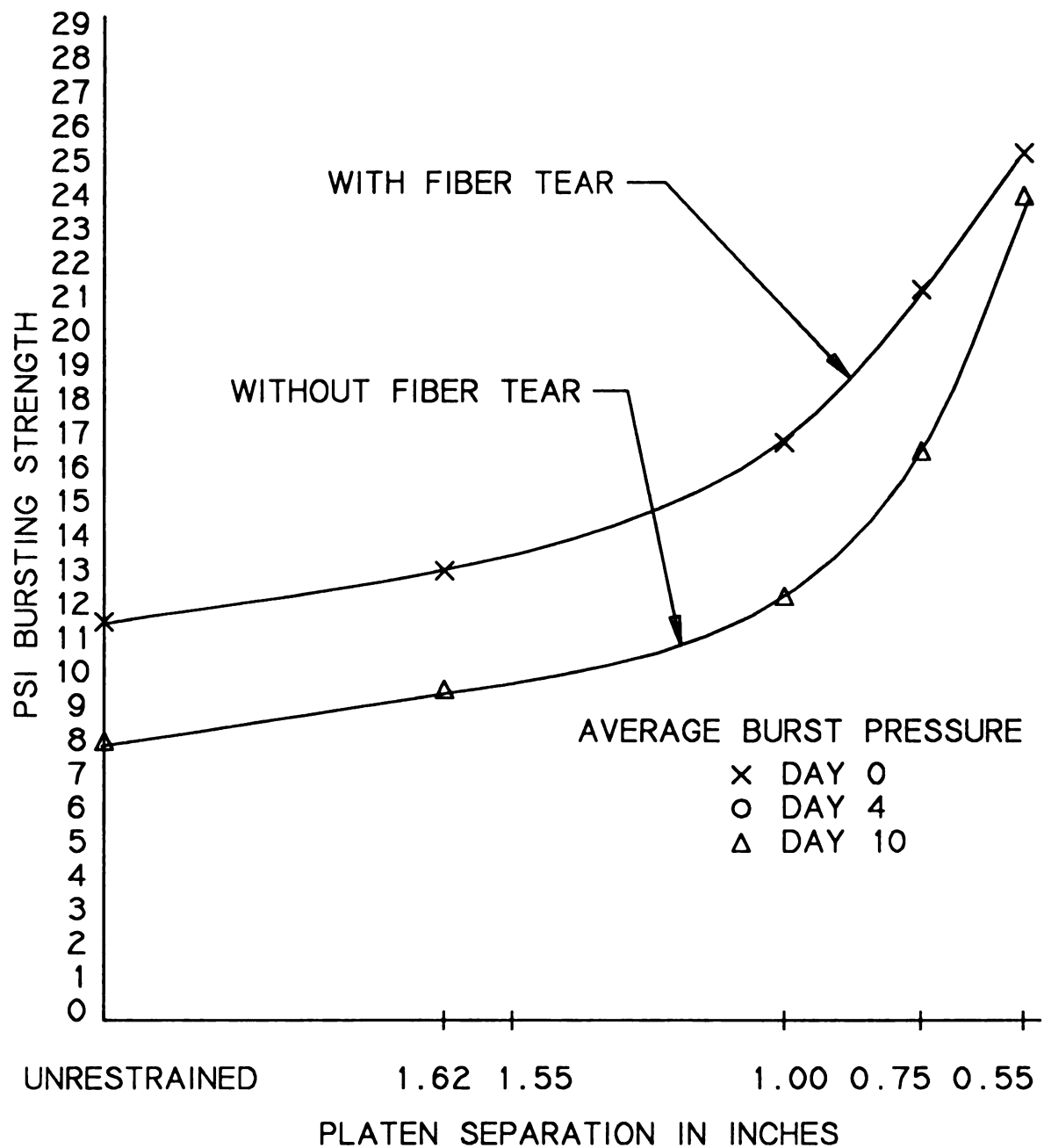


FIGURE 8 - RELATIONSHIP BETWEEN BURST PRESSURE AND OBSERVATION OF FIBER TEAR

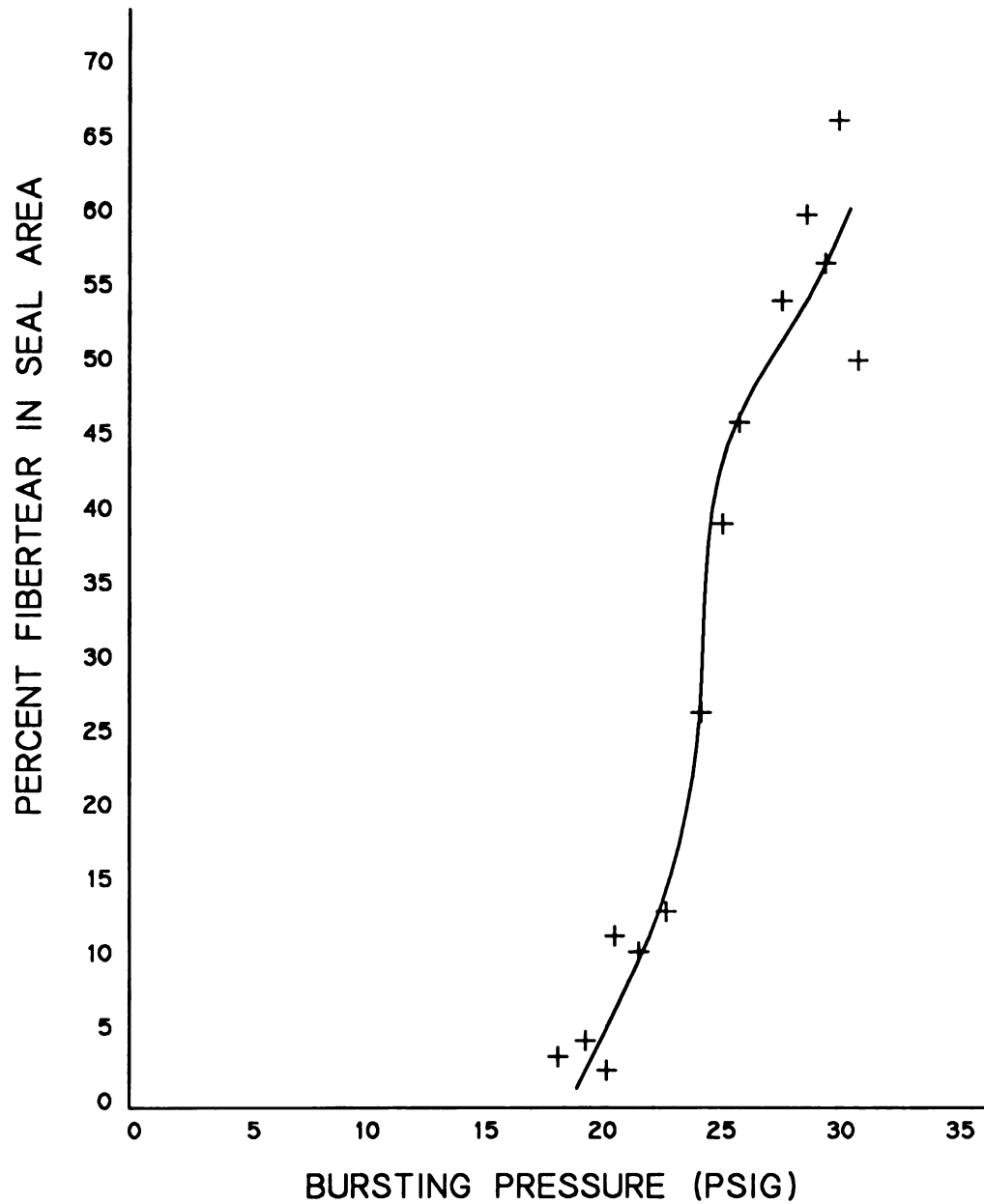


FIGURE 9 - PERCENT OF SEAL AREA SHOWING FIBER TEAR AS A FUNCTION OF BURST PRESSURE

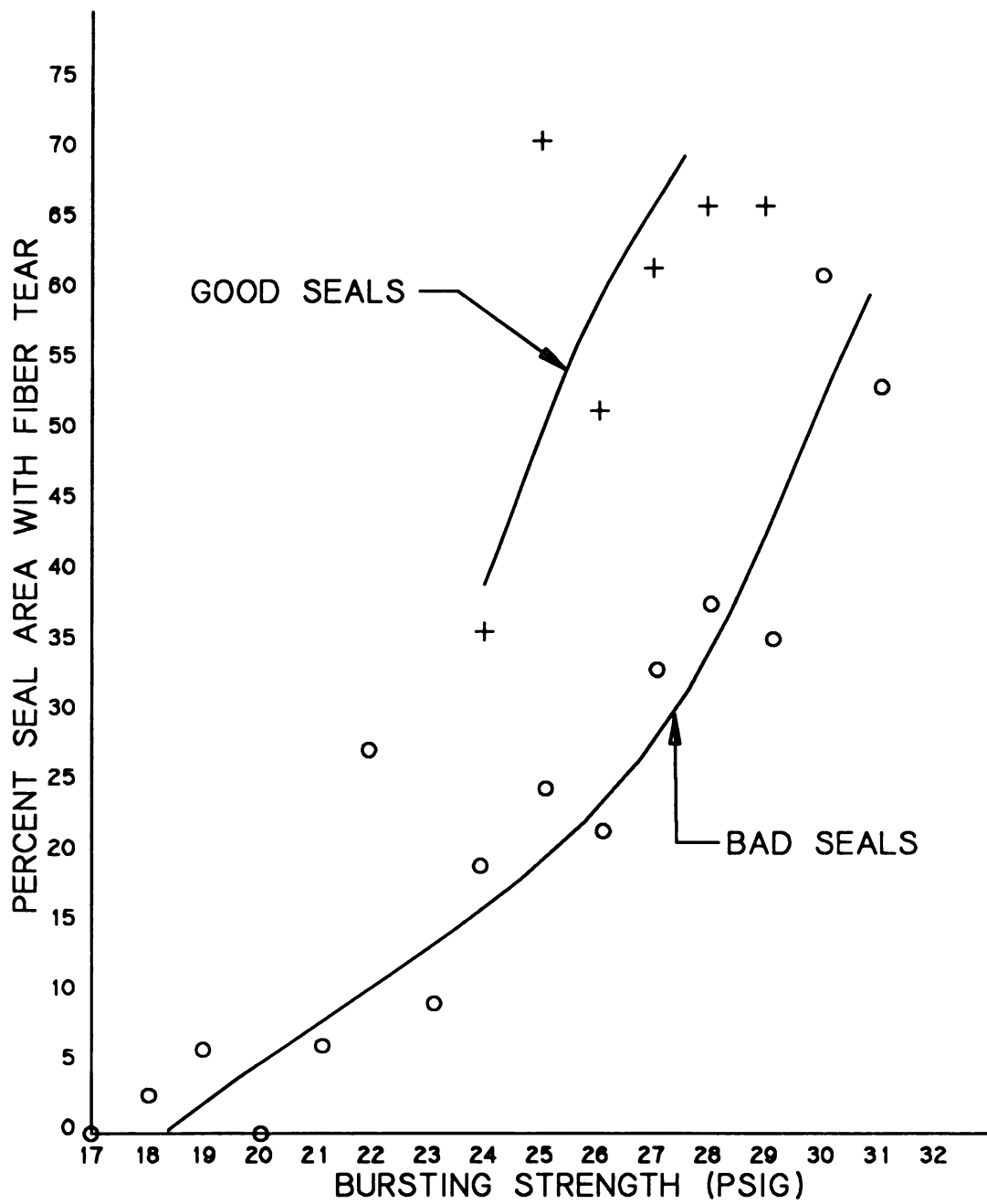


FIGURE 10 - FIBER TEAR IN GOOD AND BAD BRIK PAK SEALS

TABLE 27
PERCENT OF SEAL AREA SHOWING FIBER TEAR AS A FUNCTION OF
BURST PRESSURE - Platen Separation 0.625 inches

Burst Pressure <u>psi</u>	Number of samples	Average area of paper tear	Standard deviation of area of paper tear
7	1	0	0
16	1	0	0
17	1	0	0
18	13	2.9	3.9
19	16	4.7	9.5
20	8	2.7	2.6
21	8	11.9	5.9
22	20	10.6	10.4
23	31	13.5	11.9
24	16	27.0	13.9
25	12	39.6	23.6
26	27	46.8	17.7
27	21	54.8	17.3
28	19	60.5	12.9
29	18	57.1	15.5
30	4	67.2	6.0
31	2	52.1	4.0

TABLE 28
PAPER TEAR IN "GOOD" AND "BAD" BRIK PAK SEALS

<u>GOOD SEALS</u>		<u>BAD SEALS</u>	
Bursting Strength <u>psi</u>	% Area of Seal as <u>Exposed Paper</u>	Bursting Strength <u>psi</u>	% Area of Seal as <u>Exposed Paper</u>
		17	0
		18	3.125
		19	6.6
		20	0
		21	6.25
		22	28.2
		23	9.8
24	36.5	24	18.8
25	70.8	25	25.0
26	51.6	26	23.8
27	65.3	27	33.1
28	66.3	28	37.5
29	66.1	29	35.4
		30	62.5
		31	53.1

The key to seal integrity is the formation of an uninterrupted barrier which extends throughout the the seal area. If this is present, it is inferred that a hermetic seal exists. Paper tear is evidence that the site of seal failure is not between the layers which are fused together to produce a homogenous plastic seal. The concern is to objectively determine whether or not a hermetic seal existed by mechanically determining resistance to internal pressure. There are two methods by which this may be tested: static and dynamic burst testing.

F. Static Test Method

The static test method consists of inflating test containers to a predetermined pressure and holding that pressure for a specific period of time. If the test conditions are correctly established, all containers with defective seals will fail and all containers having hermetic seals of sufficient strength will pass.

Four groups of samples were obtained from the warehouse of Squirt Pak International in Holland, Michigan. The aseptically filled 250 ml brik pak containers were produced during a period when initial production was destroyed by quality control representatives. The samples range from "known bad" to "known good". Management selected two "unknown" sample sets for comparison with the "known bad" and "known good" samples. Two cases (27 containers per case) were tested for each sample under the conditions shown in TABLE 29.

TABLE 29
STATIC BURST TESTING OF 250 ml BRIK PAK CONTAINERS

<u>Sample</u>	<u>Pass</u>	<u>Fail</u>
Known Bad	36	18
Unknown #1	32	22
Unknown #2	45	9
Known Good	51	3

Conditions: Inflation rate 1 psi/second, 30 seconds hold at 20 psig, plate separation 0.55 inches.

The test conditions used are similar to those used for the 5 ounce MRE retort pouch, except that the platen separation for 250 ml brik paks is

.55 inch and is less than the 3/4 inch platen separation used for retort pouch burst testing. Samples of brik paks were collected then tested following the sequence of production. The number of passing containers increased in a gradual transition from the "bad" to the "good" similar to the manner in which the beverage producer had reduced the sealing problem during production. The processor held all questionable production within his warehouse pending the results of incubation. The observation that some "good" packages failed indicates that the test conditions for retort pouches may be too severe for a static burst test involving brik paks.

The static burst test adds a severe stress factor to the test which is not present in the dynamic burst test described below. A constant static pressure will cause the sealant to slowly distort and flow in a manner described as "seal creep". This stress will accelerate laminate separation and force partition of partially fused areas where seals may be contaminated with water, grease, or product. In practice, the static burst test represents a pass/fail requirement indicating go/no-go conditions for packaging operations.

G. Dynamic Test Method

The dynamic test method involved raising the internal pressure of containers restrained at .55 inches at a rate of 1 psig/second until failure was observed. Known good, known bad and two unknown samples were evaluated using this method. The two-tailed t-test was utilized to determine if there was a significant difference between the groups, or if the unknowns could be described as good or bad by statistical comparison with the means of these groups at a 95% confidence level. The results of dynamic testing are shown in TABLE 30. Data is contained in Appendix A.

TABLE 30
DYNAMIC BURST TESTING OF 250 ml BRIK PAK CONTAINERS
Samples restrained at .55 inches rate inflation 1 psi/sec inflate to failure

	<u>Mean psi</u>	<u>Standard deviation (psi)</u>	<u>Sample size</u>
Known Bad	22.8	4.064	54
Unknown #1	22.9	4.056	54
Unknown #2	23.8	2.923	54
Known Good	27.7	1.469	54

Student's two-tailed t-test was employed to determine whether or not differences between the known standards could be detected at the 95% confidence level. The known "good" sample was first compared with the known "bad" sample with the expected result that they are not equal ($\neq$). The two-tailed t-test established that a significant difference exists between known "good" and known "bad". The two-tailed t-test also shows that when the order of the data is reversed by comparing known "bad" to known "good" the answer remains the same ($\neq$). Next the sample unknowns #1 and #2 were compared with the known standards "good" and "bad". The statistical test revealed that unknown #1 is significantly different from the known "good" sample. The test could not detect a significant difference between unknown #1 and the known "bad" sample. By inference we conclude that unknown #1 does not confirm to the criteria for "good" and is therefore "bad". Unknown #2 similarly was compared to the standards for "good" and "bad". The test indicated a significant difference existed between the known "bad" sample. It did not detect a significant difference between unknown #2 and the known "good" sample. By inference we

conclude that unknown #2 is therefore "good" The results of this statistical evaluation are illustrated by Table 31.

TABLE 31
RESULTS OF TWO-TAILED T-TESTING
95% CONFIDENCE LEVEL

	<u>Known "Good"</u>	<u>Known "Bad"</u>
Known "Good"	good	not equal
Unknown #1	not equal	bad
Unknown #2	good	not equal
Known "Bad"	not equal	bad

This statistical analysis indicates that as the average burst test increased gradually the package seals being produced changed from "bad" to "good". Similarly as the observed burst pressure for samples increased their standard deviation became smaller analagons to process coming under control.

H. Determining Trends During Production

Control charts are commonly used to collect production data which enable the the food packer to identify trends. A good seal, having a hermetic seal will rupture at a higher internal pressure than one which is partially fused or contaminated by food, grease or water. Furthermore, failure of a good seal will not occur between the sealant layers in the fused area but between other laminates or within the paper layer itself.

To establish a preliminary control chart, the average burst pressure of known good containers was chosen as the midpoint. The upper and lower control limits are set at three standard deviations from the mean ($\bar{x} \pm 3s$). During production, packages may be removed from the production

line, burst tested and the values plotted on the control chart as a function of sampling time as shown in FIGURE 11. A trend toward increasing burst pressure indicates greater seal strength and less seal contamination. A trend toward decreasing seal strength indicates weaker seals and a greater likelihood of seal defects.

VI. DISCUSSION

The objectives for testing of food packages and the requirements for any test method are straightforward. The purpose of testing is to establish that package contents are safe for human consumption and are likely to survive the rigors of distribution without loss of hermetic integrity. The ideal test would employ sensing techniques that do not adversely affect the package, require subjective human inspection, or create economic loss by testing. After sealing, food packages would flow past the testing station in an uninterrupted flow into their shipping cases. The test method would be precise and reproducible. All packages approved by the tester would be documented as safe. All samples rejected by the tester could be indicative of a process drifting toward a control limit. The operator would make adjustments to the process as necessary to eliminate the cause of package defects. Testing on the production line would provide all of the necessary feedback to assure the processor, regulatory agency, and consumer that a defective package cannot escape detection.

Very few packaging test methods are nondestructive. Those which are provided varying degrees of confidence. Nondestructive test methods include machine vision, sound, gas, reflected light, magnetic or proximity detection, and visual inspection. Destructive test methods include biotesting, dye penetration, electroconductivity, tensile or burst testing, and

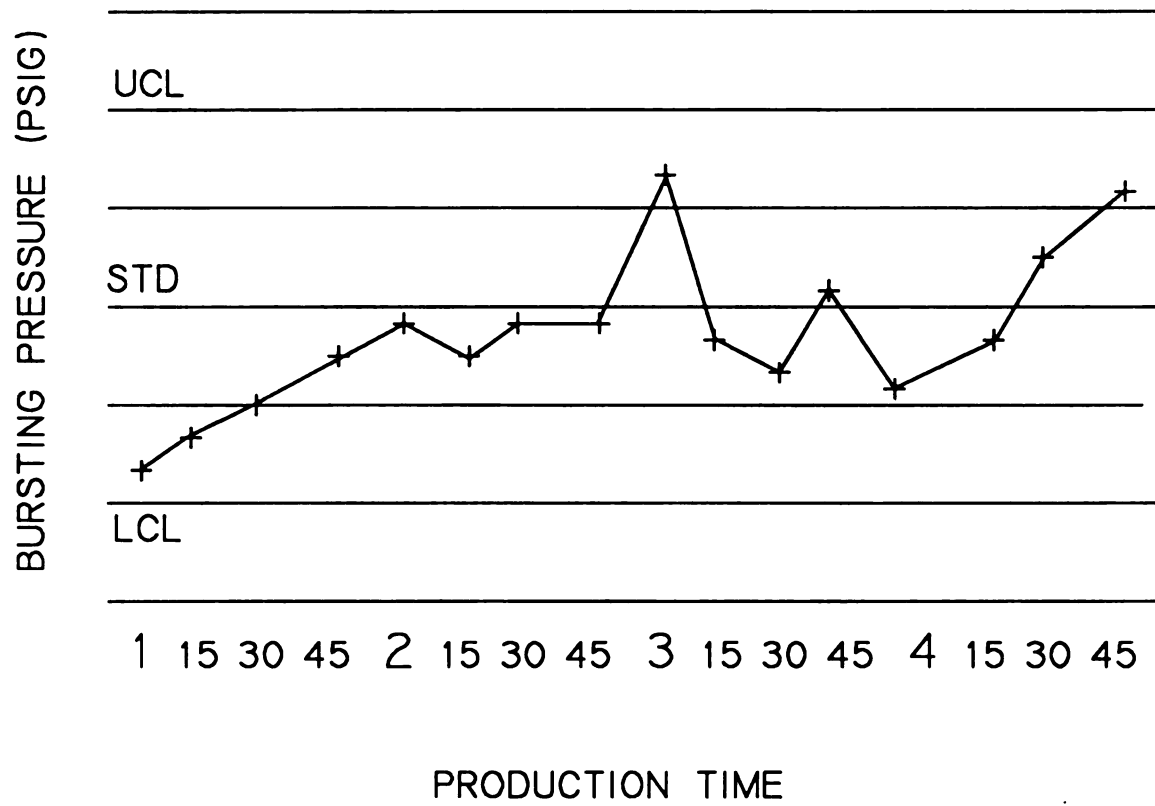


FIGURE 11 - CONTROL CHART

physical teardown of the package. At the present time there are no tests which are considered reliable enough to eliminate the need for destructive testing for low-acid shelf-stable foods. Statistical sampling from the production line (moving lot) and the warehouse (stationary lot) is required to assure the greatest confidence with the least number of samples. Samples, labor, test equipment and time are all economically significant. The packager must balance quality control costs against liability in the marketplace.

The burst test has been cited by representations of the NFPA, FDA, and USDA as the best overall package test for low-acid shelf-stable foods in flexible packages because it stresses a package uniformly. Burst Testing of 250 ml brik paks formed, filled, and sealed on a Tetra Pak model AB-3 machine at Squirt Pak, International at Holland, Michigan provide insight into this observation.

During initial testing of the 250 ml brik pak packages by injecting air, failure occurred consistently at the transverse seals. When first inflated, the packages unfold and the corner tabs become detached. The package first attains the shape of a pouch then inflates like a small rectangular football. Failure of the back seal seldom occurs under these conditions because the package does not expand when in contact with restraining plates. Presumably the lap of material and internal longitudinal strip (LS) creates a seal that has more resistance to internal pressure than the transverse seals possess. The relationships between internal pressure, angle of the transverse seal and the tension forces relating to burst failure in 250 ml brik paks are proposed herein.

FIGURE 12 shows a 250 ml brik pak with internal pressure creating stress on the package walls.

FIGURE 13 shows the same brik pak restrained by parallel metal plates. The distance between these plates may be adjusted.

As 250 ml brik paks are inflated, they expand to a point of maximum internal volume. Additional air injected into the package causes a corresponding increase in pressure. Tension develops in the packaging material. Since the material is homogenous, the tension in the material is uniform up to the point where expansion creates stress. The restraining plates resist the pressure. Packaging material against a plate is pulled taut by the expanding two sides and ends of the package. FIGURE 14 shows the relationship between vectors of tension (σ) and pressure (P) in a cross section of a fused transverse seal. The package wall under even pressure is curved with a constant radius (r). TABLE 32 summarizes terms used in the equations which follow.

TABLE 32
DEFINITION OF TERMS USED IN EQUATIONS 1 THROUGH 6

r	=	radius at one half (D) the distance between the restraining plates in inches
b	=	thickness of the paper in the composite structure in inches
D	=	distance between the restraining plates in inches
P	=	pressure within the brik pak during testing as observed on the pressure gauge in units of pounds per square inch gauge (psig)
R	=	resistance to pressure in units of pounds per square inch gauge (psig)
σ TS	=	sigma tensile strength in units of pounds per square inch, because this value exceeds the tensile strength of the composite structure and the tensile strength of the seal other (k) factors may be present
t	=	the thickness of the composite material in inches

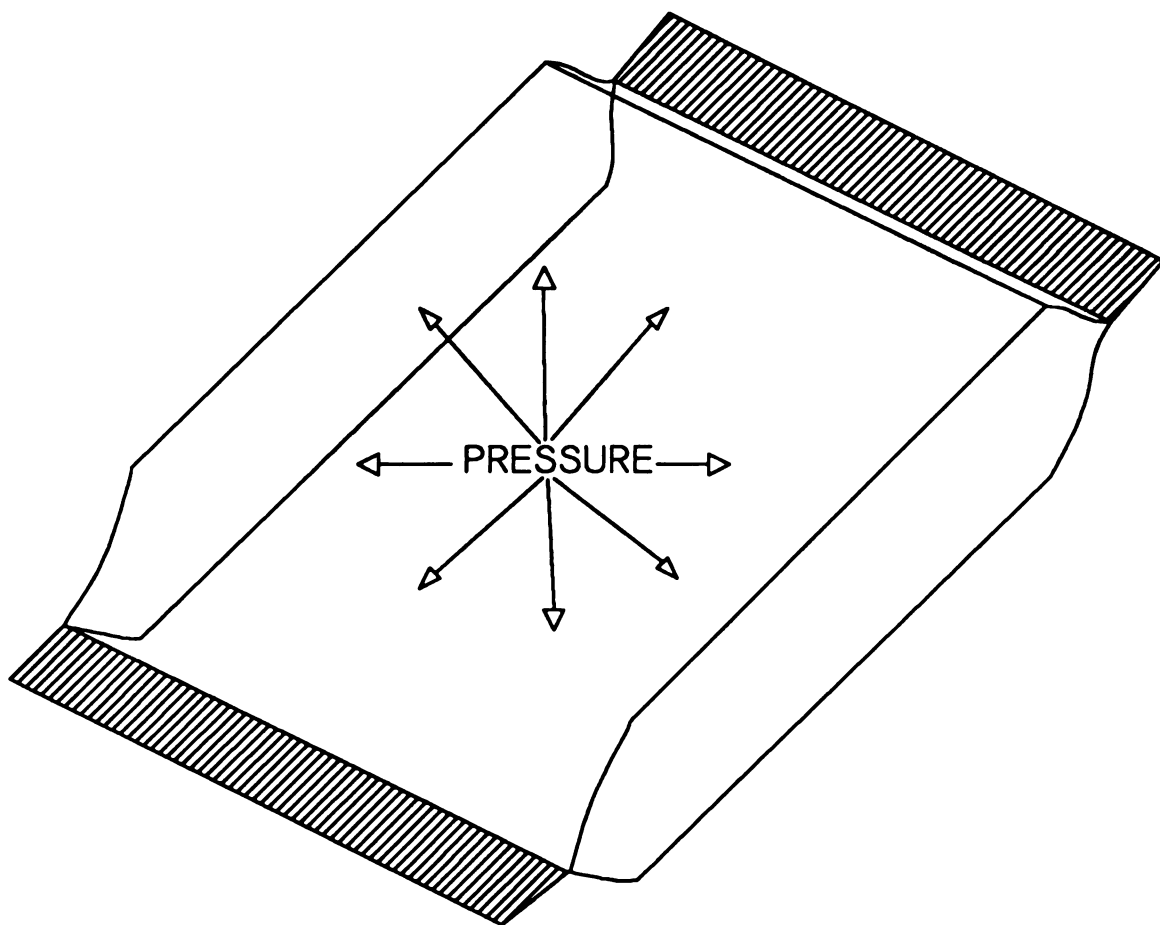


FIGURE 12 - 250ml BRIK PAK WITH
INTERNAL PRESSURE EQUAL
AT ALL POINTS

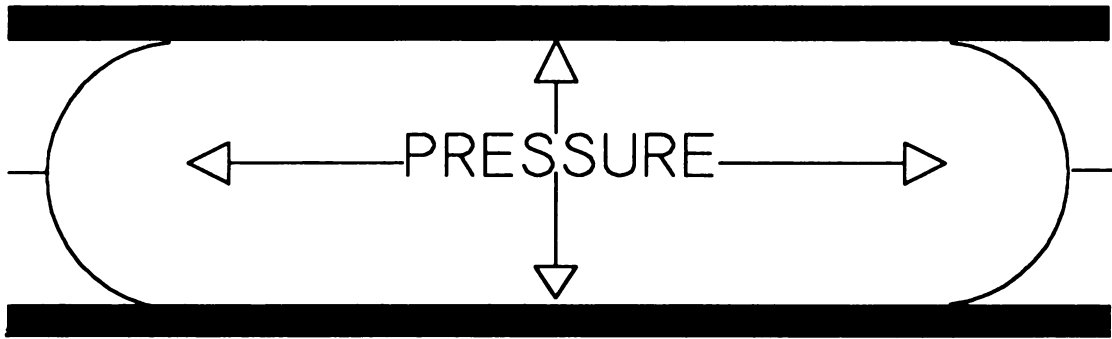


FIGURE 13 - 250ml BRIK PAK WITH INTERNAL
PRESSURE RESTRAINED BETWEEN
METAL PLATES

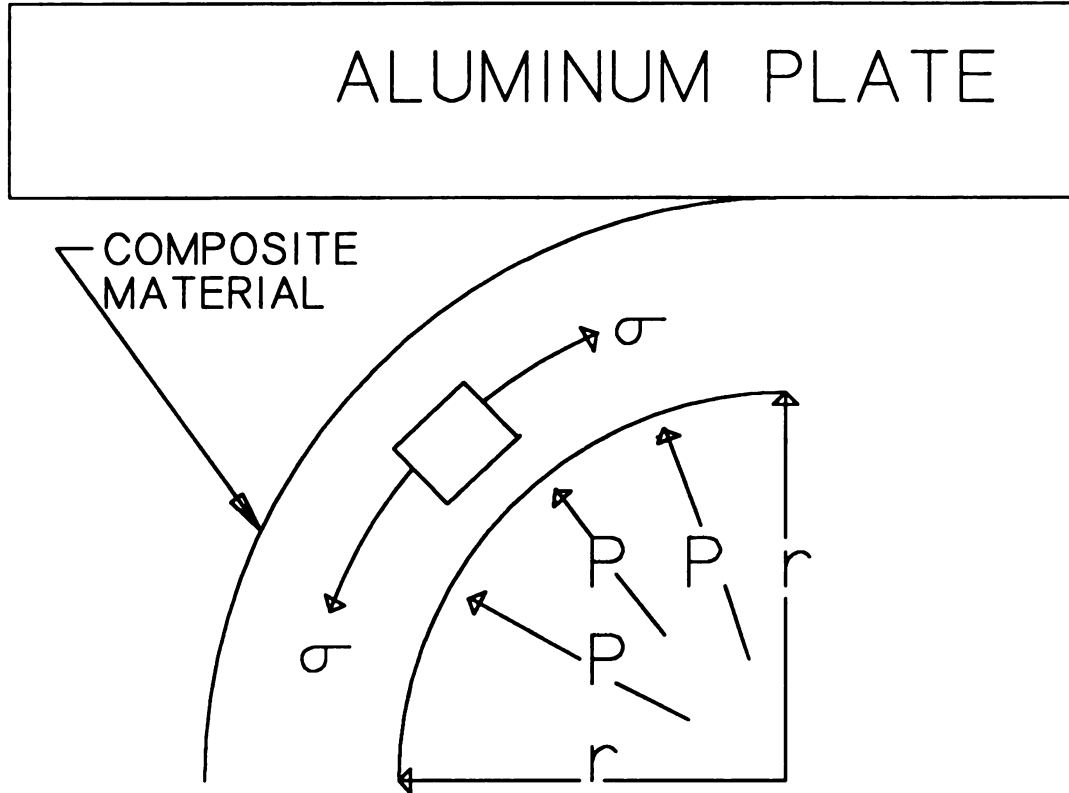


FIGURE 14 - VECTORS DEPICTING TENSION
IN TRANSVERSE SEAL OF
250ml BRIK PAK

TYPICAL VALUES

$$\begin{aligned}
 D &= .50 \text{ to } 1.625 \text{ inch} \\
 P &= 0 \text{ to } 50 \text{ pounds/square inch} \\
 \text{TS} &= 602.9182 \text{ pounds/square inch on the average} \\
 t &= .013 \text{ to } .0135 \text{ inch}
 \end{aligned}$$

The thickness (t) of the package wall is constant and the composite material behaves like a membrane. The curvature of the end of the 250 ml brick pak at the transverse seal is a function of the material, the internal pressure and the distance (D) between the platens in the restraining device. This is shown in FIGURE 15.

When a membrane under stress responding to pressure lies in the shape of a 90° curve with a constant radius, the stress may be described by the relationship shown in EQUATION 1.

EQUATION 1

$$\sigma = P \frac{R}{t}$$

There are two elements to the composite structure which are of interest. First, the sealant material which forms the hermetic barrier and second, the paper which provides the tensile strength. The boundary (b) between the paper and sealant lies at an intermediate point in the Composite structure (bt). When all forces are at equilibrium EQUATION 2 applies.

EQUATION 2

$$2 \sigma (bt) - P (bD) = 0$$

This relation may be reduced to EQUATION 3.

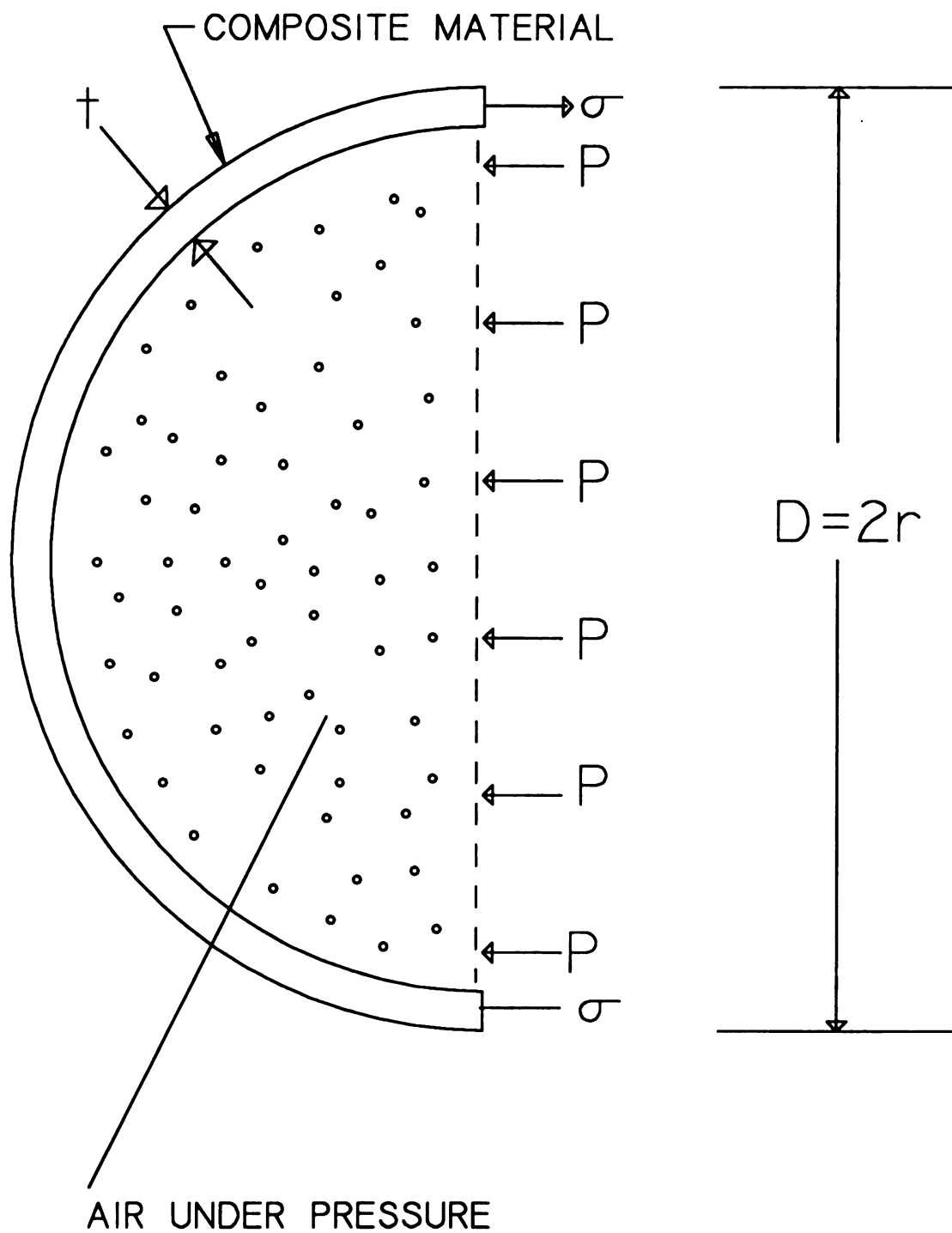


FIGURE 15 - AIR UNDER PRESSURE
IN 250ml BRIK PAK

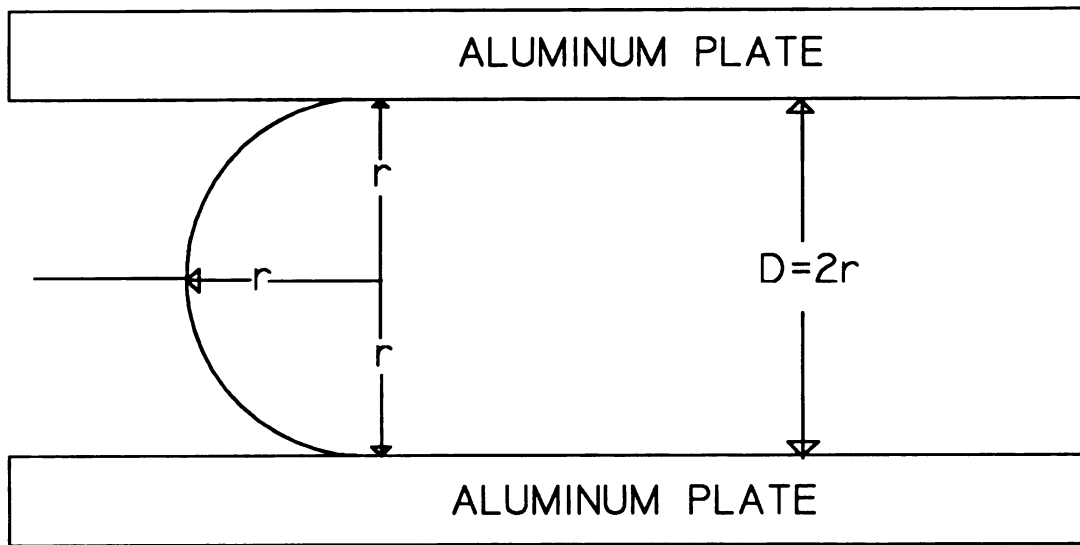


FIGURE 16 - 250ml BRIK PAK SEAL
UNDER PRESSURE BETWEEN
ALUMINUM PLATES

EQUATION 3

$$2 \sigma_t = PD$$

The relationships of EQUATION 3 are illustrated in FIGURE 16. EQUATION 3 may be rearranged to solve for tension as shown in EQUATION 4.

EQUATION 4

$$\sigma_t = \frac{P(D/2)}{t}$$

Pressure is easily measured using a gauge (psig). EQUATION 5 shows the relationship of the variable to pressure.

EQUATION 5

$$P = \frac{2\sigma_t t}{D}$$

When a 250 ml brik pak bursts while being restrained it is observed that the tensile strength (σ_t) of the material is less than the stress applied to it. In EQUATION 6 tensile strength is substituted for tension.

EQUATION 6

$$PD = 2\sigma_{TS} t$$

EQUATION 6 states that for a composite material consisting of two segments in a fusion seal, the tensile strength at equilibrium is a function of the distance between restraining plates and the internal pressure. This relationship exists only for an instant before the seal bursts. As the paper fibers separate under stress, the sealant either fails by adhesion or cohesion. If the seal is fused the separation will occur between the plastic and the paper and delamination will result. This is evident by the amount of fiber tear observed in the seals. EQUATION 6 also describes a continuous function. The data indicates that transverse seals burst at

greater pressures as the restraining plates are brought closer together. TABLE 33 contains estimated pressure values for various separation distances for the platens during burst testing. These are compared with experimental evidence. Both are shown in FIGURE 17.

TABLE 33
ESTIMATED AND ACTUAL BURST PRESSURES AS PLATES ARE
BROUGHT CLOSER TOGETHER

<u>D</u> <u>observed</u>	<u>P</u> <u>estimated</u>	<u>P</u> <u>average observed</u>
1.625	9.23	10.35
1.55	9.68	12.125
1.00	15.00	15.87
0.75	20.00	20.67
0.55	27.27	24.00

The values of P estimated in Table 33 use the burst test values for the 5 and 7 ounce retortable pouch: $P = 20.0$ psig and $r = .75$ inch. The thickness for the composite wall structure of a brik pak $t = .135$ inch. The value for ϕ is estimated using EQUATION 4 and P is estimated using EQUATION 5 for the various platen separation distances D used for the experiments.

Burst Test versus Tensile Test

The burst test stresses the entire package uniformly. The tensile test, ASTM D 882 tests only a segment of the seal. The same seal tested by both methods should give identical results for seal strength. However, this only holds true when the angle of the seal in both test samples is identical. The tensile test pulls a clamped sample only at an angle of 180° . This is the

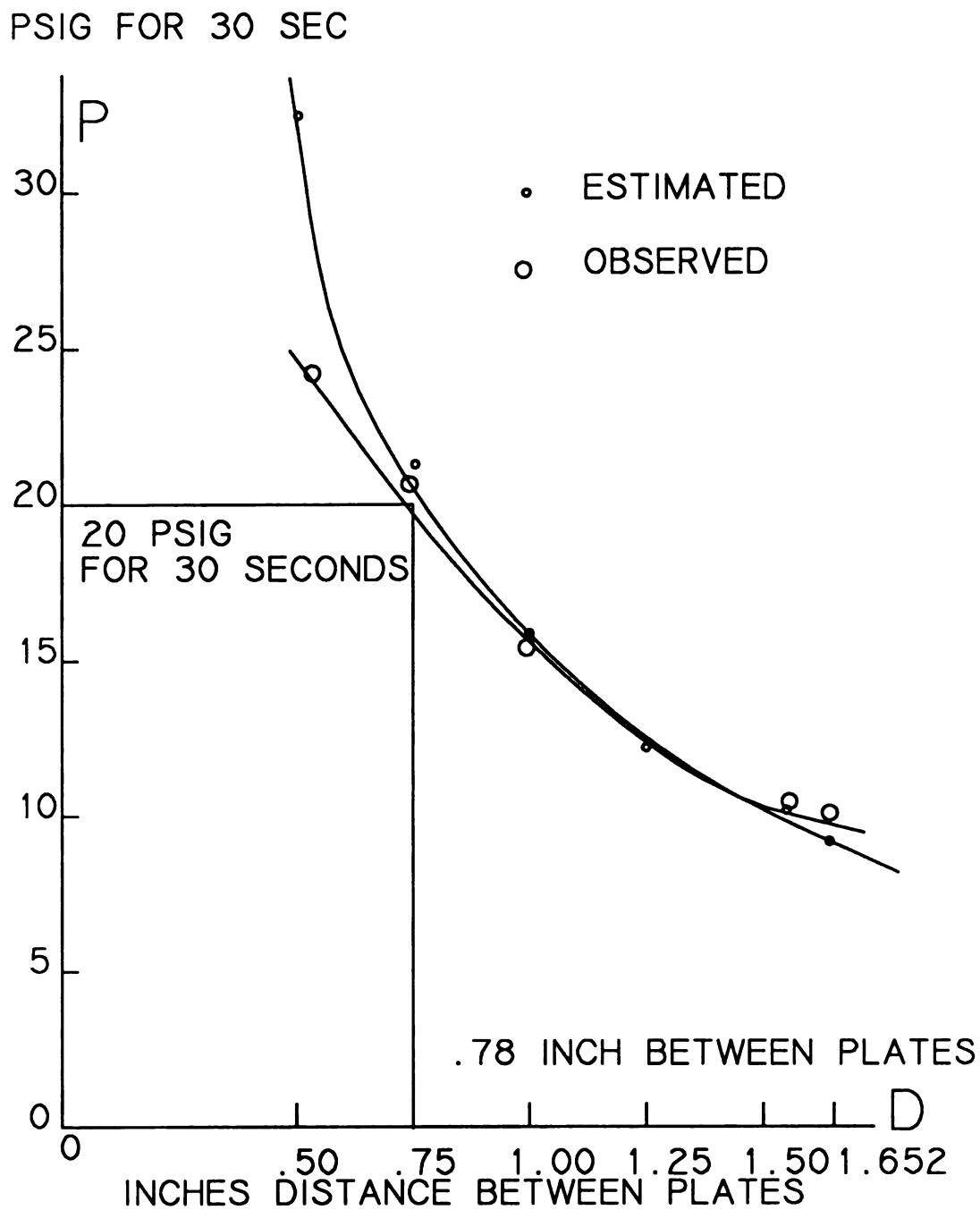


FIGURE 17 - PREDICTED VALUES FOR P
COMPARED TO OBSERVED
VALUES FOR P

same angle as would be associated with an unrestrained burst test. Test data recorded from restrained and unrestrained brik pak samples indicated that it is easier to separate "good" from "bad" when the angle was less than 180°. The lower the seal angle the greater the force required to cause fusion seal failure. "Good" seals require more force than "bad" seals under these conditions. The tensile test therefore is not as precise in separating "bad" from "good" as the burst test in which samples are restrained.

VII. SOURCES OF ERROR

1. Pressure gauges may be inaccurate and should be calibrated prior to conducting testing.
2. The tube connecting the sample and pressure gauge may contain liquid product or a foreign particulate which could block the passage of air. These tubes should be cleared periodically.
3. There may be a pressure leak in the system or surrounding the needle hole which delays the rate of inflation.
4. Unfolding heat sealed corners and draining their contents may damage packages.
5. Material properties may be affected if the packages are wet. Water will wick into the paper from cut edges.
6. The configuration of the restraining device makes recognition of back seal defects difficult to detect.
7. Minute holes may be insufficient to permit an airflow which is detectable by the Aro burst tester.
8. Samples which have been dropped on their corners sustain damage. These are not acceptable for burst testing. Similarly samples that have been compressed or impacted may sustain separation of the seal which cannot be determined without destructive testing.

VIII. CONCLUSION

A burst test may be employed in the manufacture of paperboard aseptic packages with fusion seals to measure the strength of seals and to predict their performance in distribution. The static method may be applied to moving lots to determine when conditions change and seal strength is lost. The dynamic method may be employed to determine the effect of machine adjustments on the strength of seals in moving lots. Stationary lots may be sampled and a dynamic burst test used to determine the average and standard deviation. Hypothesis tests may be employed to compare sample values to standard values thus permitting statistical confidence in the decision to accept or reject a lot. Restraining samples during burst testing causes burst failures of "good" fusion seals to occur at greater pressures than "bad" fusion seals. A greater separation in the burst pressures of strong and weak seals will be observed when a restraining device is employed when burst testing 250 ml brik paks. Unrestrained brik paks delaminate in the transverse seal area when they burst. However, restrained samples do not delaminate at burst and the defect is often left along with a narrow channel where failure has occurred at less than normal bursting pressure. Fiber tear is a visual indicator of burst seals. Seals which fail at higher bursting pressure display more fiber tear than those which fail at a low bursting pressure. Burst tests are deemed desirable for packages with fusion seals because the package is stressed uniformly and fails at the weakest point.

APPENDICES

APPENDIX A

TABLE 34
STATIC BURST TEST
30 sec hold at 20 psig

	<u>Pass</u>	<u>Fail</u>	<u>Total</u>
Known Bad	36	18	54
Unknown #1	32	22	54
Unknown #2	45	9	54
Known Good	51	3	54

TABLE 35
DYNAMIC BURST TEST

Maximum observed burst pressure (psig)

	<u>Mean (psi)</u>	<u>Standard Deviation (psi)</u>	<u>Sample Size</u>
Unknown Bad	22.8	4.064	54
Unknown #1	22.9	4.156	54
Unknown #2	23.8	2.923	54
Known Good	27.7	1.469	54

TABLE 36
KNOWN GOOD SAMPLES

<u>Sample Number</u>	<u>Maximum Burst Pressure (psig)</u>	<u>Burst Seal Top or Bottom</u>	<u>Length of Observed Fiber Tear (inches)</u>	<u>Percent of Burst Seal Showing Fiber Tear (percent)</u>
1	26	t	2.5	2.5
2	29	t	2.5	62.5
3	24	b	1.5	37.5
4	24	b	1.0	25.0
5	26	b	2.0	50.0
6	26	b	1.5	37.5
7	26	b	2.0	50.0
8	27	t	2.5	62.5
9	26	b	2.5	62.5
10	28	b	2.5	62.5
11	24	b	2.125	53.125
12	26	b	2.0	50.0
13	29	b	2.5	62.5
14	26	b	3.0	75.0
15	27	b	2.5	62.5
16	28	b	3.0	75.0
17	25	b	2.5	62.5
18	25	b	3.0	75.0
19	28	b	3.0	75.0
20	27	b	3.0	75.0
21	27	b	2.5	62.5
22	28	b	2.5	62.5
23	26	b	2.5	62.5
24	27	b	2.5	62.5
25	25	b	3.0	75.0
26	28	b	3.0	75.0
27	29	b	2.5	62.5

TABLE 36 (continued)
KNOWN GOOD SAMPLES

Sample Number	Maximum Burst Pressure (psig)	Burst Seal Top or Bottom	Length of Observed Fiber Tear (inches)	Percent of Burst Seal Showing Fiber Tear (percent)
28	26	b	2.0	50.0
29	28	b	2.5	62.5
30	26	b	2.5	62.5
31	27	t	2.5	62.5
32	26	b	2.0	50.0
33	26	b	1.5	37.5
34	26	b	2.0	50.0
35	26	b	2.0	50.0
36	27	b	2.5	62.5
37	27	b	3.0	75.0
38	28	b	2.5	62.5
39	27	b	2.5	62.5
40	29	b	2.5	62.5
41	24	b	2.125	53.125
42	28	b	2.5	62.5
43	27	b	2.5	62.5
44	28	b	3.0	75.0
45	27	b	3.0	75.0
46	28	b	2.5	62.5
47	29	b	3.0	75.0
48	28	b	2.0	50.0
49	28	b	2.5	62.5
50	26	b	2.5	62.5
51	29	b	3	75.0
52	28	b	2	50.0
53	24	b	1	5.0
54	24	b	1	25.0

TABLE 37
KNOWN BAD SAMPLES

<u>Sample Number</u>	<u>Maximum Burst Pressure (psig)</u>	<u>Burst Seal Top or Bottom</u>	<u>Length of Observed Fiber Tear (inches)</u>	<u>Percent of Burst Seal Showing Fiber Tear (percent)</u>
1	20	t	0	0
2	20	t	0	0
3	29	t	2	50
4	27	b	2	50
5	19	t	0	0
6	31	t	2	50
7	30	b	2.25	62.5
8	18	t	0	0
9	19	t	0	0
10	29	t	1.75	43.75
11	31	b	2.25	56.25
12	26	b	2.125	53.125
13	20	b	0	0
14	23	t	.5	12.5
15	27	b	2	50
16	21	t	.25	6.25
17	21	b	.35	6.25
18	28	b	2	50
19	25	b	1	25
20	24	t	.5	12.50
21	18	b	.125	3.125
22	26	b	1.125	28.125
23	23	t	.25	6.25
24	18	b	0	0
25	23	b	.25	6.25
26	18	b	0	0
27	23	b	.25	6.25
28	24	t	.125	28.125

TABLE 37 - (continued)
KNOWN BAD SAMPLES

Sample Number	Maximum Burst Pressure (psig)	Burst Seal Top or Bottom	Length of Observed Fiber Tear (inches)	Percent of Burst Seal Showing Fiber Tear (percent)
29	22	b	.125	28.125
30	27	t	.125	28.125
31	27	t	1	25
32	29	t	.50	12.5
33	26	b	.25	6.25
34	19	b	.125	28.125
35	19	b	0	
36	28	b	1	25
37	17	t	0	0
38	27	t	.5	12.5
39	24	t	.125	28.125
40	26	t	.25	6.25
41	24	t	.5	12.5
42	19	t	0	0
43	19	b	.125	28.125
44	23	t	.25	6.25
45	23	t	.125	28.125
46	24	b	.5	12.5
47	19	b	0	0
48	18	b	.5	12.5
49	19	b	.125	3.125
51	23	t	.125	3.125
52	18	b	0	0
53	18	t	.25	6.25
54	19	b	.125	3.125

TABLE 38
UNKNOWN SAMPLE #1

<u>Sample Number</u>	<u>Maximum Burst Pressure (psig)</u>	<u>Burst Seal Top or Bottom</u>	<u>Length of Observed Fiber Tear (inches)</u>	<u>Percent of Burst Seal Showing Fiber Tear (percent)</u>
1	22	b	.25	6.25
2	18	t	0	0
3	21	b	.5	12.5
4	28	t	2.5	62.5
5	22	t	1.25	31.25
6	29	b	2.5	62.5
7	21	b	1.0	25.0
8	22	b	.5	12.5
9	18	t	0	0
10	21	b	.5	12.5
11	22	t	.25	6.25
12	27	t	2.0	50.0
13	18	b	.25	6.25
14	29	t	3.25	81.25
15	22	b	.5	12.5
16	21		.25	6.25
17	23	b	.5	12.5
18	30	t	3.0	75.0
19	22	b	.5	12.5
20	22	b	.25	6.25
21	22	b	1.0	25.0
22	19	t	0	0
23	23	b	.5	12.5
24	22	t	.25	6.25
25	27	b	2.5	62.5
26	7	b	0	0
27	22	b	.5	12.5
28	23	t	.5	12.5
29	28	b	3.0	75.0

TABLE 38 - (continued)
UNKNOWN SAMPLE #1

Sample Number	Maximum Burst Pressure (psig)	Burst Seal Top or Bottom	Length of Observed Fiber Tear (inches)	Percent of Burst Seal Showing Fiber Tear (percent)
30	21		.25	6.25
31	24	b	.5	12.5
32	22	t	.5	12.5
33	16	b	0	0
34	29	t	2.75	68.75
35	23	t	.5	12.5
36	22	t	1.5	37.5
37	21	b	.5	12.5
38	26	t	2.50	62.5
39	22	t	.75	18.75
40	26	b	1.5	37.5
41	18	b	0	0
42	26	t	2.5	62.5
43	25	b	2.25	56.25
44	22	b	1.0	25.0
45	21	b	.5	12.5
46	22	b	.5	12.5
47	21	t	.75	18.75
48	30	b	2.75	68.75
49	22	t	.75	18.75
50	18	b	.25	6.25
51	27	b	3.0	75.0
52	22		1.25	31.25
53	30	b	2.5	62.5
54	29	b	2.25	56.25

TABLE 39
UNKNOWN SAMPLE #2

<u>Sample Number</u>	<u>Maximum Burst Pressure (psig)</u>	<u>Burst Seal Top or Bottom</u>	<u>Length of Observed Fiber Tear (inches)</u>	<u>Percent of Burst Seal Showing Fiber Tear (percent)</u>
1	23	b	1.0	25.0
2	24	t	1.25	31.25
3	25	b	.50	12.5
4	23	b	.125	31.25
5	26	b	1.50	37.5
6	19	b	0	0
7	20	b	.25	6.25
8	23	b	.25	6.25
9	29	b	2.125	53.125
10	25	t	1.25	31.25
11	23	b	.125	3.125
12	27	t	1.75	43.75
13	24	b	1.50	31.5
14	26	t	.75	18.75
15	23	b	.75	18.75
16	25	b	.50	12.5
17	26	b	1.0	25.0
18	23	b	.25	6.25
19	29	t	2.25	56.25
20	19	b	.25	6.25
21	23	b	.25	6.25
22	19	t	.25	6.25
23	20	b	.125	3.125
24	23	t	.50	12.5
25	19	b	.125	3.125
26	27	t	2.25	56.25
27	23	b	.25	6.25
28	26	t	2.0	50.0
29	24	b	1.25	31.25

TABLE 39 - (continued)
UNKNOWN SAMPLE #2

<u>Sample Number</u>	<u>Maximum Burst Pressure (psig)</u>	<u>Burst Seal Top or Bottom</u>	<u>Length of Observed Fiber Tear (inches)</u>	<u>Percent of Burst Seal Showing Fiber Tear (percent)</u>
30	29	t	1.75	43.75
31	23	b	.25	6.25
32	25	b	.50	12.5
33	19	b	0	0
34	25	b	1.50	37.5
35	20	b	.125	3.125
36	23	b	.25	31.25
37	29	b	1.75	43.75
38	23	b	.50	12.5
39	19	t	0	0
40	25	b	1.0	25.
41	23	t	.25	6.25
42	27	b	1.5	37.5
43	24	b	.25	6.25
44	23	b	.125	3.125
48	27	b	2.125	53.125
49	23	b	.50	12.5
47	20	t	.125	3.125
48	28	b	1.75	43.75
49	23	b	.50	12.5
50	29	b	2.25	56.25
51	23	b	.50	12.5
52	25	b	2.0	50.0
53	23	b	.50	12.5
54	20	t	.25	6.25

TABLE 40
Percent of Seal Showing Fiber Tear As a Function of Burst Pressure

Burst Pressure		Percent of Surface Area In Failed Seal Showing Fiber Tear or Fusion	
<u>psig</u>	<u>n</u>	<u>psig</u>	<u>(%)</u>
7	1	0	0
16	1	0	0
17	1	0	0
18	13	2.9	3.9
19	16	4.7	9.5
20	8	2.7	2.6
21	11	11.9	5.9
22	20	18.6	10.4
23	31	13.5	11.9
24	16	27.0	13.9
25	12	39.6	23.6
26	27	46.8	17.7
27	21	54.8	17.3
28	19	60.5	12.9
29	18	57.1	15.5
30	4	67.2	6.0
31	2	53.1	4

Plate separation .625 inches.

TABLE 41
Burst Strength vs Area Showing Fusion and/or Fiber Tear

<u>7 psig</u>	<u>16 psig</u>	<u>17 psig</u>	<u>18 psig</u>	<u>19 psig</u>	<u>20 psig</u>
0 area	0 area	0 area	0 area	0	0
			3.125	0	0
			0	28.125	0
			0	0	6.25
			12.5	0	3.125
			3.125	28.125	3.125
			0	0	3.125
			6.25	0	6.25
			0	6.25	
			6.25	6.25	
				3.125	
				0	
				0	

TABLE 41
Burst Strength vs Area Showing Fusion and/or Fiber Tear - (continued)

<u>21 psig</u>	<u>22 psig</u>	<u>23 psig</u>	<u>24 psig</u>	<u>25 psig</u>
6.25 area	28.125 area	12.5 area	37.5 area	62.5 area
6.25	6.25	6.25	25	75.0
12.5	31.25	6.25	53.125	75.0
25.0	12.5	6.25	53.125	25
12.0	6.25	6.25	25	56.25
6.25	12.5	28.125	25	12.5
6.25	12.5	3.125	12.5	31.25
12.5	6.25	12.5	28.125	12.5
12.5	25.	12.5	28.12	12.5
12.5	6.25	62.5	12.5	37.5
18.75	12.5	12.5	12.5	25
	12.5	12.5	12.5	50
	37.5	12.5	31.25	
	18.75	25.	37.5	
	37.5	31.25	37.5	
	18.75	6.25	6.25	
	25	3.125		
	12.5	18.75		
	18.75	6.25		
	31.25	6.25		
		12.5		
		6.25		
		6.25		
		31.25		
		12.5		
		6.25		
		3.125		
		12.5		
		12.5		
		12.5		
		12.5		

TABLE 41
Burst Strength vs Area Showing Fusion and/or Fiber Tear - (continued)

26 psig	27 psig	28 psig	29 psig	30 psig	31 psig
62.5 area	62.5 area	62.5 area	62.5 area	62.5 area	50 area
50	62.5	75.0	62.5		
37.5	75.0	75.0	62.5	75.0	56.25
50	62.5	62.5	62.5	68.75	
62.5	62.5	75.	75.0	62.5	
50	62.5	62.5	75.0		
75.0	62.5	62.5	50		
62.5	75	62.5	43.75		
50	62.5	75.0	12.5		
62.5	62.5	62.5			
50	75.0	50.0	62.5		
37.5	50	62.5	81.25		
50	50	50.0	68.75		
50	28.125	50	56.25		
62.5	25	25	53.125		
53.125	12.5	62.5	56.25		
28.125	50.0	75	43.75		
6.25	75.0	56.25	43.75		
6.25	43.75	43.75	56.25		
62.5	37.5				
62.5	53.125				
37.5					
62.5					
37.5					
18.75					
25					
50					

APPENDIX B

AIR LEAK TESTING

Objective:

To detect holes by injecting air into hermetic package.

Method:

Puncture container wall with needle. Inject air while increasing at 1 psi/sec until reaching a standard pressure. The standard pressure used for testing should be less the normal unrestrained burst pressure for the package. Additionally, the container may be immersed under water and observed for emission of a steady stream of air bubbles indicating the location and relative size of the leak. A proximity meter may be employed to measure deflection in a container which is subjected to external air pressure.

Materials:

Compressed air with regulator
Needle, valve, hoses
Water
Transparent container to observe bubbles

Procedures

Select a sample package from the production line. Inject air to create internal pressure within the package without causing it to burst. Immerse the package in water and inspect visually for a stream of bubbles emitting from a common source.

Results:

Positive - a steady stream of bubbles is observed to come from the package at one or more locations.

Negative - no bubbles are observed to be emitted from the package.

False Positive - bubbles are emitted from the point at which the needle entered the package or bubbles clinging to the surface of the package release after the package was submerged in water.

False Negative - food particles block holes through which air might escape from a defective package or the air pressure used is insufficient to force air through minute holes in the package.

Discussion:

There are two methods. The first involves piercing a package to inject air or by cutting the seal area away from the package and clamping it to a fixture before immersing it in water. The second method involves creating high pressure around the closure or seal of a package and measuring deflection of the lid with a proximity device. Helium is sometimes used because the extremely small size of the helium molecules, relative to most gas molecules, allows helium to penetrate and permeate small openings more quickly. Devices for both applications are commercially available.

BIOTESTING**Objectives:**

To detect the presence of holes in hermetic packages by placing them in an agitated solution of fermentative bacteria in water for an extended period of time.

Method:

Obtain representative packages and submerge them in an agitated solution of active bacteria. The bacterial concentration should be 10^3 to 10^6 bacteria per cubic centimeter. The bacteria must cause fermentation of the product within the package if they penetrate and must not be pathogenic. Packages should be flexed during immersion to expose cracks and holes to incursion. The solution which surrounds the packages should be maintained at a temperature that permits rapid growth of bacteria within defective packages. Following biotesting, packages are incubated for two weeks at 95° to 100°F.

Materials:

Waterbath with temperature control and agitation
Solution of Aerobacter aerogenus or Eschericia coli.
Sample packages
Apparatus to flex packages
Incubator

Procedures:

Obtain representative samples

Mix active bacteria in water add samples

Procedures - Continued:

Agitate waterbath and flex sample for 3-14 days

Incubate samples for two weeks at 95° to 100°F Observe packages for swelling

Results:

Positive - gas formation within an infected package causes it to swell and distort.

Negative - the hermetic barrier of the package remains intact preventing bacterial infection of the product.

False Positive - the product was not commercially sterile prior to hermetic closure and incubation caused organisms contained within the sealed package to produce gas.

False Negative - fermentation occurred within a package but the gas produced vented through a hole in the package.

Discussion:

Biotesting is a method for uniformly conditioning packages in an environment which provides a high probability of infecting packages which lack hermetic barrier. Stressing the packages by flexing them while they are surrounded by a solution of viable microorganisms makes the test more rigorous. This test is time consuming and requires skilled personnel. A temperature controlled test apparatus and incubator are available only in

well equipped laboratories. These are seldom available at most food processing establishments. Because this is a cumbersome test, its use is generally limited to evaluation of new package designs and start-up of new equipment.

CHEMICAL ETCHING

Objective:

To chemically strip the layers of a hermetically sealed composite paperboard package to expose the sealant layer.

Method:

The outer layers of a package are removed by tearing, abrasion, and chemical action to expose the sealant layer intact. By photographing or xeroxing the package prior to etching, then comparing the etched seal with the photo, it is possible to develop an understanding of the hermetic significance of visually discernible defects.

Materials:

Water bath and heater with thermostat
Three each, one-liter pyrex glass beakers
Tongs
Running tap water
Graduated cylinder
Automatic stirring device (heated is preferred)
Paper towels
Drying oven equilibrated to 65°C (150°F)
Rubber gloves, protective goggles, apron
Fumehood with chemical resistant surface

Chemicals for etching of paperboard aseptic packages

1. Hydrochloric acid (HCL) concentrated 36 to 38 percent in water
2. Acidified solution of CuCl_2
3. Solution of bisodium carbonate (saturated) in water

Preparation Methods

1. Pour one liter of concentrated HCL into one liter of cold distilled water. Pour slowly as heat will evolve when acid and water mix. Stir until mixed completely. Cover to prevent evaporation.

2. Pour 0.5 liter of concentrated HCL into 1.5 liter of cold distilled water. Add 10 grams of CuCl_2 . Stir until completely mixed. Cover beaker and allow to warm to room temperature before using.
3. Pour sufficient baking soda (Na_2CO_3) into a container to make a saturated solution at room temperature. Some undissolved Na_2CO_3 should remain on the bottom of the beaker even after stirring.

Procedures:

For paperboard aseptic packages, Tetra Pak recommends the following method, equipment, chemicals, and procedure for acid teardown of Brik pak seals.

1. Cut transversal seal from package approximately one inch from the end. Multiple samples may be identified by notching the cut edge with a scissors.
2. Manually strip the paper from the sample to be etched.
3. Place the sample into hot hydrochloric acid solution at 65°C for five minutes.
4. Remove the sample using tongs and immerse it in bisodium carbonate solution to neutralize the acid.
5. Remove the sample from the sodium carbonate solution using tongs and rinse it in running tap water. Pull off the polyethylene layer which lies between the paperboard layer and the aluminum foil.
6. Using a glass stirring rod to manipulate the sample, drop it into the copper chloride solution so that it is completely immersed. Observe closely while stirring to assure that the heat of the reaction does not damage the polyethylene sealant layer as the foil is dissolved. Remove from solution.
7. Neutralize the sample by dipping it in the sodium carbonate solution followed by rinsing with water.
8. Press the sample gently between soft absorbent paper towels and place the sample in an oven at 65°C (150°F) until dry.
9. Apply an alcohol based ink solution to the inner and outer seal edges. (See section on dye testing for solution formula.

10. Observe the pattern of ink dispersion and check for leaks and channels within the fused seal area. An overhead projector is useful to enlarge seal samples and make accurate visual inspections.

Results:

Positive - a channel in a seal is made visible by etching and by dye.

Negative - a fused seal having no break in hermetic integrity is observed.

False Positive - dye on the outside surface of an etched seal is mistaken for a break in the hermetic barrier.

False Negative - dye does not penetrate a seal channel all the way through the seal.

Discussion:

The significant advantage of seal etching is the ability to relate visually observed package defects with the integrity of the hermetic seal. This in turn provides accurate information concerning seal integrity for paperboard aseptic packages. With a clear understanding of the relationship between visually observed defects and the associated etched seals, operators may be able to make appropriate machinery adjustments before seal problems develop into package defects.

Chemical etching to remove the polyester and aluminum foil layers from sealed retort pouches was developed by Ludlow Corporation in the late 70's, (Long 1985). This was further developed by the Army's Natick Laboratories and the NFPA as a tool for visually inspecting the fusion

portion of MRE Army rations. A solution made from 200 ml nitric acid, 200 ml potassium chromate and 200 ml water was heated in a vented hood and used to dissolve the polyester layer of the retort pouch. The acid was diluted by rinsing the pouch in tap water followed by a dip in a mild solution of sodium hydroxide to neutralize any residues. When immersed in a concentrated solution of copper sulphate without the protection of the polyester layer, aluminum foil will quickly dissolve. The remaining polyolefin is transparent. Wiping the etched area forcefully with a paper towel between soaking in the solutions makes this method quick and effective, but hands should be protected by chemical resistant gloves. The ability to examine blisters, wrinkles and seal contamination under the microscope was a large step toward classifying visual seal defects in terms of public health significance.

COMPRESSION TESTING**Objective:**

To detect holes in hermetic packages by externally applying mechanical force.

Method:

Place a filled and sealed food package on a flat surface and apply pressure while observing for leaks.

Materials:

Flat surface or conveyor belt
Sealed package
Heavy flat object or mechanical press
Timer

Procedure:**A. Static Method**

Place a sealed package on a flat surface then lay a weight having a flat surface upon it. Observe the effect the weight has upon the integrity of the package seals as a function of time. A similar test may be performed by applying a constant weight to a package moving on a conveyor belt. The speed of the moving belt determines the time of static compression.

B. Dynamic Method

Using a platten to continually increase the force applied to a package by two flat surfaces moving together at a constant rate. Observe the maximum force required to cause failure of the package.

Results:

Positive - holes form in the package or its seals or seams

Negative - no loss of hermetic integrity occurs.

False Positive - underfilled or weak walled packages deflect in a manner that simulates failure without loss of hermetic integrity.

False Negative - holes form in the package but food product closes off the holes disguising the defect.

Discussion:

Containers may be compressed, and to one degree or another, compression may be used as an indicator of abuse resistance and package integrity. A short list of common compression tests given below is taken from Federal Test Methods Standard No. 101C, 13 March 1980.

1. **Compressibility and recovery test for gasket materials.**

ASTM F.36-66 (1973) is appropriate for measuring the ability of gasket material to resist a compressive load applied to a localized area of the top surface and its ability to recover from such deformation.

2. **Compression set after constant deformation. Federal Test Method #2009.**

This test method indicates the residual deformation of low density (generally less than 7.5 lbs/cu.ft.) packaging materials after being compressed for a period of time in a clamp. Thickness of the material is measured before compression and after the recovery period allowed.

3. Compression set after cycling. Federal Test Method #2010.

This test method indicates the residual deformation of low density (generally less than 7.5 lb. cu.ft.) packaging materials after being cyclically compressed and released from compression many times. Thickness of the material is measured before compressing and after a one hour recovery period.

4. Compression test for shipping containers. Federal Test Method #5003 (ASTM D642-76).

ASTM D642-76 is appropriate for measuring the ability of shipping containers to resist compressive loads applied in a testing machine. Load may be applied top-to-bottom, side-to-side, end-to-end, or diagonally. The number of specimens (if other than three), loading manner, and end point should be specified in reference to this method.

5. Compressive force-displacement characteristics of cushioning materials. Federal Test Method #2011.

This procedure is intended to determine the relationship between a slowly applied compressive load and the resulting displacement of the material.

6. Compressive properties of rigid plastics.

ASTM D695-77 is appropriate for determining the compressive properties of rigid plastics when loaded in compression at relatively low speeds of testing. The use of a supporting jig adapts the method to the testing of plastic sheets, but the method is not applicable to plastic films.

One device for compression testing of water filled pouches was marketed by R.W.P. Flexible packaging in 1974 (Lampi, et.al. 1976).

"At least one European supplier uses and specifies internal pressure tests of the restrained pouch for package seal integrity. The company offers a procedure and equipment for a compression strength test in which a water-filled pouch is placed between two plates connected directly to an indicating hydraulic load cell, and a static loading is applied across the faces of the pouch. Pouches must withstand a force of 7.5 kg per 15 mm of internal seal length applied for 15 seconds." (Lampi, et.al. 1976)

Kraft uses an Instron Universal Testing Machine to compress retort pouches to study abuse testing for their A la Carte Program according to Paul Grabowski (1986). Kraft found that the vacuum test was inadequate in finding defects. However, a compression method proposed by the British Research Association recommended 700 pounds over the 23 square inch pouch surface (21.7 psig). (Lampi, et.al. 1976.) Compression at 1000 pounds (31 psig) would result in seal failure. Using these findings Kraft developed a static load on-line burst testing device for retort pouches consisting of parallel conveyor belts and rollers that apply a static force to pouches as they pass through the device.

This device initiates separation of the fusion seals (seal "creep" or "mooning") which is inspected visually following testing. Any separation in excess of 1/16 inch constitutes a defective package. The two test variables established for this testing machine are 1/32 inch deflection of the deadweight which rises when pouches are in the tester and the thickness of the pouch. The relationship between pouch area and static weight are

constant. A variation of this test may be used to compress plastic bottles with septum lids or semirigid and paperboard liquid filled aseptic boxes.

Packages are often manually squeezed to force product against the inner edge of a seal. This is normally used by line operators sealing pouches and Brik Paks. After squeezing, the operator/inspector examines the seal for separation at the inner seal edge. For Brik Paks this action is followed by visual inspection of six areas on the package after the paper has been stripped manually from the package. The four corners and two crossovers (back strip seal intersecting the two transverse seals) are carefully inspected. Line operators often refold the Brik Pak into a brik-shape before looking at the corners. Visual inspection to determine seal separation requires opening with a compression tester it may be possible to inspect all packages objectively with a go/no- go requirement. However, some problems continue to make this method imperfect. Overfilled containers may likely fail and create a sanitation problem within the tester. Underfilled containers which do not cause the dead weight to rise may pass through this device even with weak seals which could fail in distribution. As with burst testing, any product forced into seal defects may effectively plug holes resulting in defective packages as soon as bacterial action occurs. The method is insensitive to minute pinholes. Only catastrophic failure of a package in the test will trigger the detector into sensing a bad package.

Laboratory testing using compression devices is accomplished on aseptically filled paperboard boxes at International Paper. The compressive force required to rupture I.P. paper aseptic boxes is a routine laboratory test for the package manufacturer according to (DeGeronomo, 1986).

DISTRIBUTION (ABUSE) TESTING**Objective:**

To simulate conditions which result in the failure of defective packages in a manner similar to that observed in distribution. Normal packages would be expected to withstand this abuse.

Method:

Packages are subjected to vibration, compression, and impact at levels observed to be typical of the distribution system for which they are designed. Following the test, which is a conditioning regime, the packages are examined. Defects are quantified and described in relation to package failures observed in normal distribution. Corrective action is taken to eliminate fragility by engineering design changes in the package system.

Materials:

Packages to be tested
Vibration table
Compression (Platten) Device
Drop Tester
Laboratory at 72°F, 50% R.H.

Procedure:

A representative sample of packages is obtained and conditioned in the laboratory at 72°F, 50% R.H. for a minimum of 72 hours prior to testing. When these environmental conditions cannot be met the ambient conditions are observed and reported. The level of abuse to be simulated is different for each distribution system. The reader

may refer to ASTM 4169 for tables and explanations. The duration and frequency of vibration, load compression, and drop heights are predetermined values which simulate the distribution abuse for a particular mode of conveyance. During the simulation any failures are inspected and recorded. Following the simulation all package failures are examined and compared to similar defects collected from shipping tests or returned from distribution. Later the package design is altered to reduce fragility and the test repeated to confirm the effects of the changes.

Results:

Positive - a package loses hermetic integrity during any one phase of the testing protocol.

Negative - a package retains hermetic integrity through the test.

False Positive - a package appears to be defective, however, confirmational testing by biotesting, incubation, or dye penetration reveals no loss of the hermetic barrier occurred during the abuse test.

False Negative - a package which appears to pass the testing regime later exhibits failure and dye testing reveals a break in the hermetic barrier which is attributed to failure during the test.

Discussion:

The purpose of abuse testing is to simulate the forces that cause damage to defective packages. The objective is to reproduce damage similar to that observed in distribution but in a laboratory under controlled conditions. When done correctly the degree of damage may be correlated to

the amount of force. A correctly engineered package will withstand forces in the normal distribution cycle with an economically acceptable amount of failure. The result is a balance between cost and the failure.

The importance of abuse testing for low acid, shelf stable, food products whose integrity is not addressed by 21CFR 113, was addressed by the Canned Product Branch, Processed Products Inspection Division (PPID) of the USDA following the development and distribution for foods in retort pouches. The two documents commonly referred to are Test Cycles for Small Size Flexible Retortable Pouches (USDA, June 1982) and Test Cycles for Small Sized Semirigid Containers (USDA, August 1982). These documents and procedures apply to packages for retail sales when individual weight does not exceed 16 ounces (454 grams). Both documents describe abuse conditioning of packages in their shipping containers using laboratory tests prior to evaluation of container integrity. Destructive test methods and visual evaluations are employed in these tests.

ABUSE TEST METHODS

1. Paperboard conditioning ASTM D641-49, sections B,C,D
2. Vibration testing of shippers ASTM D999-75
3. Drop test for shippers ASTM D775-61 (objective B) Identify faces and corners of shippers in accordance with ASTM D775-61. The angle of fall is 15° off vertical with the impacting surface 90° to the direction of motion.

Abuse testing of retortable food containers is preceded and followed by tensile testing, ASTM D882 Method A or B and an internal burst test of 15 psig for 30 seconds with 1/2 to 5/8 inch restraint to determine the effects of abuse. A maximum 1/16 inch seal separation is permitted. For small

semirigid containers, the package seals do not lend themselves to tensile testing. Interlaminar bond strength of the lidstock is therefore substituted. Instead of peeling seals using a pull tester, the lidstock is separated using chloroform, pulled back to expose fresh interlaminar and tested following ASTM D882 Test Method For Thin Plastic Sheeting. No minimum value is given in Test Cycles for Small Semirigid Containers (pg 4, 1982). However, in Test Cycles for Small Size Flexible Retortable Pouches, an average value of 1.5 pound per inch (with no sample testing less than 1 pound per inch following ASTM D882-67) is required (pg 1,2, 1982). A modified burst test is employed by restraining the sealed package to no more than 10 percent expansion. Air pressure is forced into the sealed container which is held submerged in water $23^{\circ}\text{C} + 2^{\circ}\text{C}$). If no bubbles are observed after 60 seconds at 5 psi or no pressure drop is observed on the air pressure gauge the package is judged to be hermetically sealed. (Test Cycles for Small Size Semirigid Containers, pg 6, 1982.)

When comparing flexible pouches to metal cans using abuse testing, Schultz (1983) used ASTM 775-68 obj. B. Both fluid and semisolid food products were tested. Results of this testing is shown in TABLE 42.

TABLE 42
ABUSE RESISTANCE OF RETORT POUCHES AND METAL CANS
(SCHULZ, 1973)

	<u># of Containers</u>	<u>Failures</u>	<u>% Failures</u>
<u>Fluid Food</u>			
Metal Can	1,440	32	2.22%
Flexible Pouch #1	1,440	30	2.08%
Flexible Pouch #2	720	5	0.70%
<u>Semisolid Food</u>			
Metal Can	720	4	0.56%
Flexible Pouch #1	720	2	0.28%
Flexible Pouch #2	720	4	0.56%

ASTM D-4169 is a popular method using laboratory test methods to simulate distribution requirements.

"This method describes standardized procedures for evaluating the ability of packages to withstand the abuse of physical distribution. Since physical distribution procedures vary widely, it is up to the user to determine which test sequence is pertinent to a given application. It should be recognized that (in) the real world, distribution varies considerably and that packages are subjected to abuse that is worse than anticipated. When this happens the result is often container failure. Passing any shipping test does not guarantee performance. It only indicates that the container should perform satisfactorily under a specified amount of abuse. The primary criteria for evaluating performance after physical distribution is the ability of the package to maintain a barrier to microbial penetration. Visual appearance is, of course, also important. However, this is a subjective evaluation and is up to individual users to define accepted limits." (ASTM F2.5, 17 April 1985).

Both ASTM committees F-2 and D-10 agree that ASTM D-4169 is acceptable as a procedure for evaluating the ability of packages to withstand the abuses of physical distribution. This test was developed by ASTM

committee D-10, Ed Belmont of Delmonte Corporation, Chairman.
(Belmont, letter to F-2 and D-10 committee members 25 Nov 1985.)

Schulz (1975) and Lampi, et.al. (1976) observed the effects of abuse testing on container integrity tests.

"Seals examined at the time of creation can meet tensile and burst criteria without fusion, yet after a short (24-hour-plus) storage period, such seals fail when subjected to simulated handling tests such as vibration and drop cycles."

Abuse testing to simulate distribution requirements are not to be used in place of on-line Q.C. testing for hermetic integrity. Neither are these tests meant to be used as a routine step in on-line Q.C. testing.

"Abuse conditioning for preparation of meat and poultry semirigid packages is meant to gather background information on people submitting process filings (to FDA and USDA). It is not meant for on-line Q.C. or normal production testing." (Polvino, NFPA, 1985.)

The question is always raised by persons not familiar with abuse simulation defining how testing relates to the real world. The publications of the ASTM skirt this issue by demonstrating that the damage caused by testing is representative of the damage seen in the distribution system. Similarly the methods of Test Cycles for Small Size Flexible Retortable Pouches skirt this issue. However, both documents also describe a shipping test which may be conducted when laboratory equipment is not available.

SHIPPING TEST

The following conditions apply when performing an actual shipping test.

A. Containers and Shippers

1. The immediate containers and the shippers will be prepared as described below. In addition, the contents of the containers must support bacterial growth.
2. Test shipments will consist of an undefined number of shippers.

B. Shipping

1. Each shipper shall be marked to identify its position on a pallet or load. Diagram or describe the load configurations.
2. Shipments are made via commercial truck lines or rail lines and shall be part of a normal car load or truck load shipment, or shipped in a manner which simulates a typical commercial shipment.
3. Shipment will be made to a minimum distance of 500 miles (800 km).

C. Container Examination

The test shipment will be examined for leaking, swelling or otherwise defective containers which will be excluded from the test. The remaining normal-appearing containers will be incubated for 14 days at 95°F (35°C) and examined for spoilage. (Test Cycles for Small Size Semirigid Containers, pg 6 and 7, 1982).

The concluding statement to both documents leaves the user with the impression that there is a legal mandate to perform testing and that some form of regulatory approval is granted for packaging.

REPORTING RESULTS

A complete report, which includes a detailed description of all test procedures and test results, will be submitted to the Food Safety and Inspection Services (FSIS). Send to:

**Canned Products Branch
Processed Products Inspection Division
MPITS/FSIS/USDA
Washington, DC 20250**

(Test Cycles for Small Semirigid Containers (pg 7, 1982) and Test Cycles for Small Size Flexible Retortable Pouches (pg 6, 1982).

While testing must be conducted to ensure packages will withstand the rigors of distribution, the legal mandate is not contained in these test methods. The Food, Drug and Cosmetic Act requires that foods distributed through interstate commerce be safe and wholesome. With the exception of 21CFR 113 defining double seam integrity and the recommended guidelines for bursting strength, tensile strength, and seal separation, no specific guidelines exist.

On June 1988, Bob Miller, Ph.D., Director of Processed Products, Inspection Division USDA informed members of the NFPA/FPIG that the results of abuse testing may be submitted to USDA/PPID for evaluation. Submission of valid test results indicating adequate abuse resistance may result in USDA's approval of low-acid, shelf stable foods in flexible packages with seal widths less than the 1/16 inch minimum. This approval

mechanism establishes a 1/16 inch minimum seal width for most flexible packages for shelf stable low-acid foods as well as signals the acceptance of distribution (abuse) testing as a viable method to gain USDA approval for new package designs.

DYE TESTING**Objective:**

To detect small holes in package seals and materials using dye solutions with low surface tension.

Method:

Dye is applied to a cleaned package at the suspected location of failure and observed to pass through to the outside.

Materials:

Pigment
Solution with low surface tension
Sink
Scissors or knife
Oven to dry sample packages
Paper towels
Magnifying glass or low power microscope

Procedure:

A package is opened, emptied, washed, and dried by wiping or by oven drying. A low surface tension solution containing dye is applied on one side of the package wall at the suspected location of loss of hermetic barrier. The solution moves by capillary action through the hole and appears on the opposite side of the package wall. After drying the dye the package is cut with a scissors to closely examine the hole.

Results:

Positive - dye penetrates a hole in a package.

Negative - dye does not pass through the package (wall or seal).

False Positive - the solution dissolves the packaging material creating a hole in the package.

False Negative - the solution penetrates through holes in the hermetic barrier layers but fails to reach the outside of the package where it would be visible.

Discussion:

The benefits of dye testing to identify or verify the existence of package defects are well known.

"These tests are used for identifying potential microbial pathways. The dye will tend to accumulate in cracks with pinholes making them more readily visible. Further analysis and/or previous experience may be used to establish the microbiological significance of the suspect areas of the package." (ASTM, F2.5, Aseptic Packaging Guide, unpublished, 28 May 1984.)

Dye testing may be conducted to locate package defects and the significance of the defect verified by biotesting. Conversely, packages showing gas formation following biotesting or following incubation should be tested with dye placed inside the cleaned and dried open packages. Testing therefore, may be presumptive or used for verification as a quality control method.

"Dye tests for package integrity should be tested. A dye test will be performed concurrently with the biotest for comparison. Those (ASTM) members supplying containers for biotests will perform dye tests concurrently used, for their own quality control." (ASTM F2.5, Meeting on Aseptic Package Integrity, 1 Nov 1984, St. Charles, IL.)

The steps involved in dye testing are shown in TABLE 43.

TABLE 43
STEPS FOR DYE TESTING FLEXIBLE AND SEMIRIGID PACKAGES

1. Cut package with knife or scissors so that the seals remain intact. Rinse with water.
2. Dry with paper towel, hair dryer, heat gun, or oven at 65°C.
3. Apply 2ml of dye solution to the critical points (seals, creases, folds, suspected defect area).
4. Let package stand for a suitable (usually specified) length of time, then dry.
5. Examine the package closely while unfolding, bending, or pulling plies apart to expose inner surfaces and layers of material.
6. Those packaging faults that show penetration to ink are untight and have the potential to cause unsterility.

Step number four is critical in dye testing. A serious problem relating to flouresceine dye testing of retort pouches for MRE rations arose in 1985 and continues in 1987. The following is an example of a test proposed for metal can seam defects being applied to flexible packaging with unforseeable consequences. The B.A.M. recommends flouresceine dye testing for metal cans based upon information provided by American Can (1975).

"Flouresceine dye testing has been used for many years to detect minute double seam, lap, and sideseam leakage paths in all types of containers. The flouresceine test is especially useful for examining sanitary - style containers that are normally packed with some internal vacuum. Experience has shown that, in many cases, flouresceine dye can detect minute leakage paths on suspected cans that do not leak under air pressure. Flouresceine testing of most types of containers under vacuum simulates actual packed condition, i.e., with ends pulled inward." (BAM, 24.10, 1984).

The dye recommended by the BAM is Zyglo dye solution ZL-413 available from Magnaflux Corporation, Chicago, Illinois. (BAM, 24.10, d.1), 1984). The dye used to examine retortable pouches by the U.S. Army is Zyglo dye solution ZL-54, a water washable penetrant. The problem involving misapplication of a zyglo test for cans to flexible packages was described in a communication from the NFPA to the FDA.

"Flouresceine dye solution (Zyglo, ZL-54, Water Washable Penetrant) has shown the ability to dissolve the plastic material in flexible pouches in just 2 hours. Further, control tests were reported showing the solvent alone did no damage, but pigment causes the problem." (Denny, NFPA, letter to Jackson, FDA, 11 Dec 1986.)

Members of the NFPA-FPIG subcommittee on Flexible Pouches rejected the Zyglo dye test for retortable pouches because "false positives (detectable holes created by the dye) are obtained if the dye remains longer than two hours on the pouch." Instead of dye testing the subcommittee recommended the Burst Test contained in the BAM and the "Squeeze Test" (manual compression). Roger Genske of American Can reported that the dye may follow pathways through pinholes in the aluminum foil before attacking the polypropylene sealant. Cleve Denny of the NFPA stated that the contact time for Zyglo dye testing of retort pouches currently does not exceed 60 minutes.

Despite the problems associated with Zyglo and retort pouches, dye testing has many advantages. Paperboard aseptic containers are dye tested by machine operators during production as an on-line test. This is also repeated by Q.C. operators in the laboratories as a confirmation. When defects are detected by electroconductivity testing, their location often can be confirmed by dye tests, (BAM 24.53, 1984). Defective pulltab applications on

paperboard (aseptic) rollstock may be inspected by painting Rhodamine B in isopropanol followed by wiping with paper towels. After a few minutes the pull tabs are removed and inspected for leakage. Similarly, operators inject dye under the backing strip of aseptic paperboard packages. These are visually inspected for dye penetration under or through the polyethylene backing strip. Dye testing may be conducted on containers which show pressure loss during air leak or burst testing with pressure or vacuum.

"For detection of container leakage caused by minute body pinholes and perforations, and/or defective side seams, air pressure testing is the most convenient and conclusive. it is also helpful in locating position of double seam leaks. During pressure testing, the container is subjected to pressures that may distort double seams, and this may either produce false leakers or seal off minute leakage paths. For this reason, air pressure testing method should be used in conjunction with flouresceine test or penetrant dye test to trace actual leakage path through double seams." (BAM, 24.07, 1984)

Pressure testing may be useful for rigid containers, but not always practical for driving dye under pressure through defects in semirigid or flexible packages. Minute pinholes do not always show dye penetration immediately in laminate structures. Sometimes as much as 24 hours is required for dye penetration through aseptic paperboard packages. When pinholes are dye tested, it is often necessary to examine both surfaces, then cut the sample in a cross section to determine if wicking has occurred through one or more layers. Alcohol, having a low surface tension, and the addition of surfactants to further induce wetting, improves penetration. (DeGeronomo, I.P., 1986.) However, isopropyl alcohol can make polyethylene brittle and contribute to delamination and a false dye test. Dye

tested samples must be rinsed out and completely dried before opening. (Brik Pak Manual.)

Many problems exist with dye tests. Few packers using dye testing as a quality control method agree on which dye or method is best. Popular pigments include rhodamine B (red), methylene blue, and flouresceine. Carriers may be methanol, isopropanol or a mixture of the two. Surfactants may or may not be added. The length of time dye is in contact varies from a few minutes to 24 hours. International Paper conducted dye tests in Canada using rhodamine B in methanol, isopropanol, and with or without surfactants. Different visual artifacts were observed on similar packages and uninterpretable results were obtained. (DeGeronomo, 1986.) Possibly the flouresceine dye test contained in the BAM could be applied to all plastic food containers as an integrity test.

ELECTESTER**Objective:**

To determine changes in viscosity of shelf stable liquid foods following incubation of filled packages.

Method:

If all factors are constant, shock waves will dampen at different rates in liquids of different viscosities. Thus it is possible to incubate shelf stable liquid foods and nondestructively test each package to identify those which have been subjected to microbial activity.

Materials:

Packages filled with liquid food, incubated
Electesting device
Fixture to hold test packages

Procedure:

Representative samples are removed from the production line and incubated at 100°F for 14 days. Microbial activity resulting from loss of hermetic integrity will cause changes in the viscosity of liquid food products. Packages are placed on a fixture with the largest flat surface of the package facing downward. The package is rotated 90° horizontally and back to its original position very rapidly. This is done only one time. This motion creates a shock wave. The fixture holding the package is precisely balanced to minimize outside interference and minimize dampening. The shock wave moves back and forth within the package. The motion is sensed and displayed.

The electester is an oscilloscope with alarms alerting the operator to vibrations which dampen more quickly or more slowly than normal values for a specific liquid food product.

Results:

Positive - a wave dampens more quickly or slower than normal.

Negative - the rate of wave dampening is within the range established by testing "normal" liquid product which did not display microbial spoilage during incubation.

False Positive - the range of acceptance is too narrow erroneously identifying "good" product as spoiled.

False Negative - the range of acceptance is too broad erroneously identifying spoiled product as "good".

Discussion:

The Electester is a non-destructive testing device developed for 250 ml brik paks by Tetra Pak in Lund, Sweden. In operation, an individual container is placed in a form fitting receptical with the longest dimension laying horizontal and the shortest dimension standing vertical. Upon command, the package is quickly rotated 90 degrees in a horizontal plain, rapidly to the original position. When the package reaches the 90° point and the direction of rotation reversed, a shock wave is sent through the liquid contents of the package. This shock wave is sensed by the electester which measures the rate at which the wave dampens. The peak to peak amplitude decrease is an index of product viscosity.

Changes in viscosity occur when liquid foods spoil. Bacterial fermentation creates acid which lowers the pH causing proteins to denature. As the liquid becomes thicker the shock wave dampens sooner. By calibrating the electester to shock waves associated with known "good" product, it is possible to manually test thousands of 250 ml brik paks for those few "bad" packages displaying shock waves which dampen more quickly. The wave cycles remain constant for all commercially sterile samples once calibrated. The variable being measured is the ratio of wave height A (standard) to wave height B (sample). This value is always less than or equal to 1.0 as shown below. (Electester Model 8020 Operation Manual, pg 8, no date.)

$$\text{Amplitude Ratio} = \frac{\text{Amplitude B}}{\text{Amplitude A}} = < 1.0$$

Normal product will display a range of viscosities. Samples laying outside of the pre-established range with which the electester is calibrated will set off alarms alerting the operator. These are set aside and later tested again to ensure that no false-positive samples occur. It is possible for some "good" packages to be rejected as "bad" packages. Verification testing includes incubation of "good" and "bad" packages with the anticipation that the product contained in the "bad" packages will ferment. Spoiled packages may be destructively tested. A pH meter may be used to verify spoilage in lieu of taste testing product which may prove fatal.

Wave period will change from product to product, but within the range of one product's production lot, very little change is anticipated. Wave dampening is much more variable. The sensitivity of the electester is increased when the wave period is relatively constant and is established at approximately 50 percent of the value obtained for wave dampening as

shown in the Amplitude Ratio above. By calibrating the electester with a series of known "good" samples, the wave period may be limited to a range of $\bar{x} \pm .50$. This is defined below.

$$V = \frac{\bar{x}_i - i}{\bar{x}_i} \times 100\%$$

where $\bar{x}_i$ = the mean of wave periods in milliseconds

i = the wave period of a known "good" sample in milliseconds

V = variance

By limiting acceptance to 1/2 the normal variance some harmonic vibrations, outside interference, and other frequencies which possess wave lengths different from the primary shock wave may be reduced.

There are 142 model 8020 Electesters in the world, many of which are used to test brick paks with liquid products for changes in viscosity which relate to spoilage. The advantages of this method are that it is non-destructive and it will identify spoiled product. The disadvantages include the low production rate for manual testing and the requirements for incubation or storage of the product prior to testing.

ELECTROCONDUCTIVITY**Objective:**

To detect holes in hermetic packages by flows of electrical current.

Method:

A hermetically sealed package by definition does not possess holes. Plastics are generally poor conductors of electricity. Consequently, plastic food packages without holes will form an effective barrier to mild electrical current and this method may be used to detect minute breaks in these food packages. The presence of holes indicates that the hermetic barrier has been lost.

Materials:

1% sodium chloride in water (brine solution) Scissors
9 volt battery
Light bulb, 9 volt
Three lengths of wire, 12 inch each
Plastic bowl large enough to submerge package.

Procedure:

A sample food package is obtained and one end cut off with a scissors. Brik paks and pouches may be cut on all but one edge at the equator and folded 180° on the uncut side to form two equal halves. The samples are washed to remove all food contents and dried plugs which may occur in holes. Oven drying is recommended prior to immersion. Wiping the cut edges with a paper towel may be sufficient. Wet edges may result in false positive test results. The samples are placed in a bowl containing brine and are partially filled

with brine so that they float upright and almost completely submerged. The conductivity meter is placed with one probe inside the sample and the other external as both probes are submerged into the brine solution.

Results:

Positive - current flow indicates a break in the hermetic barrier.

Negative - no current flow indicates a hermetically barrier exists.

False Positive - aluminum foil conducts electricity. A break in or pinhole partially through the inner layers of a package may expose the foil layer resulting in a false positive test result. Dye testing will confirm the presence of holes. Moisture may form a bridge over the cut edge of a package creating a false positive.

False Negative - dried product may occlude minute holes in a package. If plugs do not rehydrate quickly they will not conduct electricity when dry package(s) are immersed.

Discussion:

This method is fast and objective. If all samples displaying conductivity are dye tested to confirm the existence and location of holes this method will prove to be quite reliable. Food plugs which fail to rehydrate quickly are rare. Because of the low incidence of false positives and negatives this method is considered objective.

Either a voltmeter or an ammeter will register an electrical current. The sensitivity must be in millivolts or milliamps. An electrician's pocket potentiometer (VOM) works very well. Many processors use a KM-66 Kyroitso AZ1-Star powered by a 9 volt battery with a test range of 0-50 mA

and a 56,000 ohm resistor available from Tetra Pak. Item number 90243-110. Conductivity is proportional to the concentration of free ions in water. Therefore, this test uses one percent sodium chloride in water to assure good conductivity. To test, the emptied, washed, and dried containers are partially filled. They are then floated, partially submerged, in the one percent brine solution so that only 2 or 3 cm is above the water level. No correlation has yet been made between hole size and amperage. There is no standard method for creating small holes of predetermined size in plastic packages. Some plastics will reclose when punctured. Bernard, (1987). Ralph Hygax of Ross Laboratories reported that aperture plates available for obtaining hole sizes starting at 2 microns have been evaluated. (ASTM F2.5, 1984). Researchers at Natick Laboratories used fine music wire to make holes in retort pouches and workers at NFPA used fine drill bits and a Moto Tool™ drill press to make holes in retort pouches for biotesting. Sizer and Arndt (unpublished, 11 Sept 1986) observed polyethylene partially reclosing over recently made pinholes made in aseptic paperboard cartons. Lasers may be used to make holes in thin brass sheets which are exposed to packages subjected to biotesting. However, brass is conductive and will not be expected to provide correct information when used for electroconductivity testing. Photoetching of thin metal plates is one way of creating minute holes. Plates are then positioned with epoxy glue over larger holes cut into a package.

The electroconductivity test is considered the best overall package integrity test for nonconductive packages. Polvino, (1986). At aseptic packaging establishments, the first paperboard cartons from the sealer and from the end of the pack line are tested in this manner. When machinery

is started at the beginning of a production run, the first 200 brik paks are generally tested for conductivity. (Brik Pak Manual.). Thereafter, samples are taken by the operator and Q.C. personnel, and tested for conductivity throughout the production shift.

There are a number of problems with electroconductivity as a test method. It is not normally possible to test above the level of the liquid. Cold seals, i.e. those lacking fusion are not detectable by this method. (Sizer in BAM, 24.52, 1984.) Dried plugs of product may not rehydrate quickly enough to be conductive. False positives, caused by breaks in the sealant layer covering the aluminum foil, conduction over wet package edges, and thin spots which lost their insulating properties have been observed by DiGeronomo, (1986). Opening the folded flaps on sealed brik paks has been noted to create microleaks. The procedure for electroconductivity testing recommends cutting a container in half and not unfolding the flaps, (Brik Pak Manual, P.L.-5, no date).

There are three ranges of response for package defects tested using the electroconductivity method. These are shown in TABLE 44.

TABLE 44
ELECTROCONDUCTIVITY RANGES

<u>Microamps</u>	<u>Interpretation</u>
0	Not conductive - a good package less
0-30	Weak positive - a questionable package
30-50	Strong positive - a bad package

Packages showing no conductivity are presumed to be tight and possessing a barrier to microbes. Packages with greater than 0 microamps are suspected of being defective. The need to retest samples with weak conductivity is one of the disadvantages of this destructive test method and this problem was recognized by the authors of the Bacteriological Analytical Manual.

"If ammeter reading is unsteady and greater than 0 microamps, wipe cut edge thoroughly. Let package stand in bath for 5 minutes. Measure once more. If reading is still greater than 0 microamps, package has leakage. If deflection is quick and precise, and reading is greater than 0 microamps, package is untight." (BAM, 24.53, 1984)

In the case of brik paks (Tetra Pak, Inc.) pseudo positives may occur on a daily basis during production. Pseudo positives, showing greater than zero, but less than 30 microamp readings, may be the result of excessive die pressure or insufficient die clearance during the scoring process in the manufacture of rollstock for paperboard aseptic packages. Two defects are possible. First, a tight fit between the scoring dies caused by incorrect die placement or unexpectedly thick paper may result in thinning of the sealant coatings adjacent to the aluminum foil. This is depicted in FIGURE 18. (DiGeronomo, I.P., 1986.) When thinned, polyethylene is less of an insulator. The package will sometimes show very weak conductivity. The second scoring defect relates to stretching and breaks within the laminate structure. This defect is shown in FIGURE 19. According to Tetra Pak this is less of a cause for brik paks than for paperboard aseptic cartons made by International Paper.

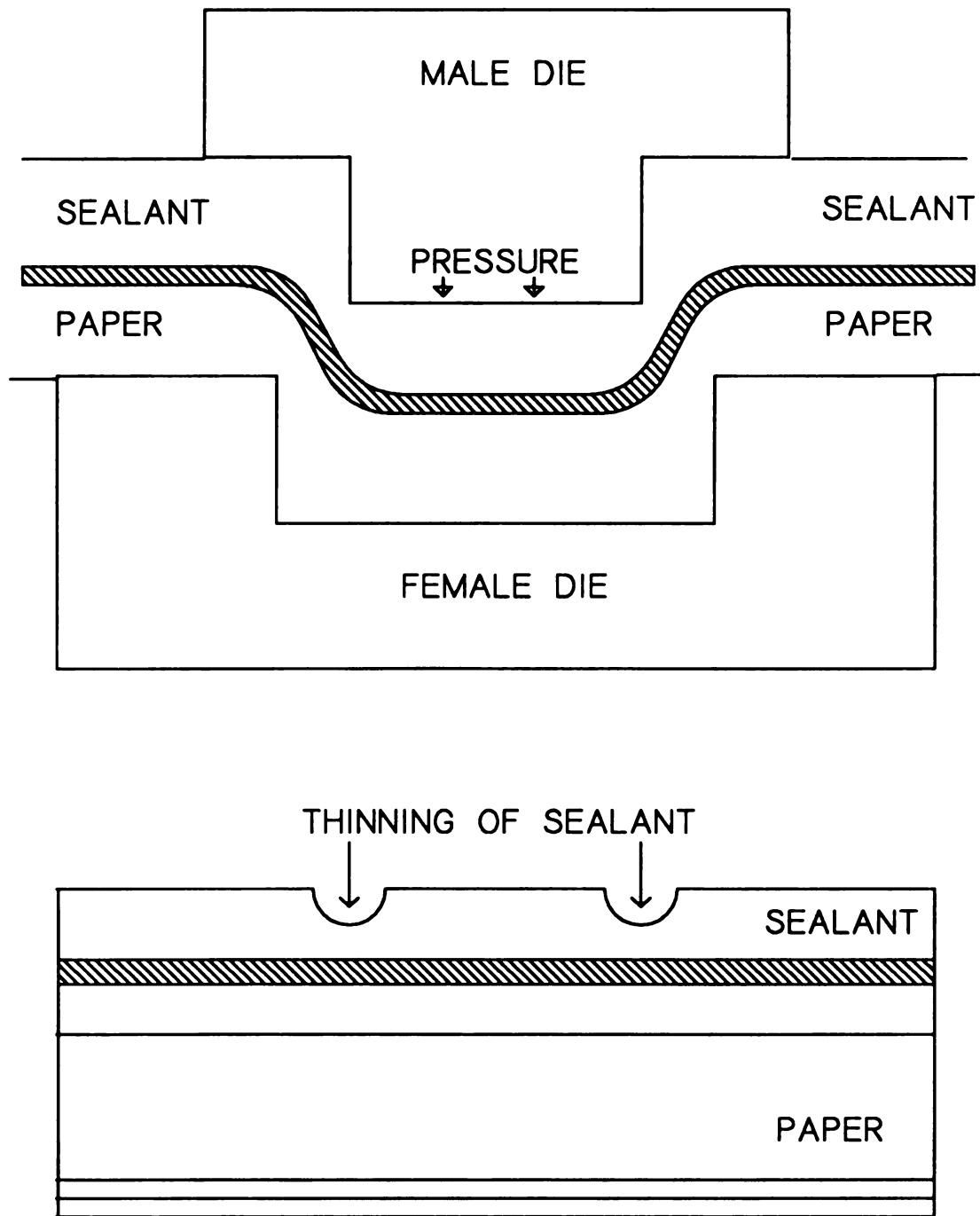
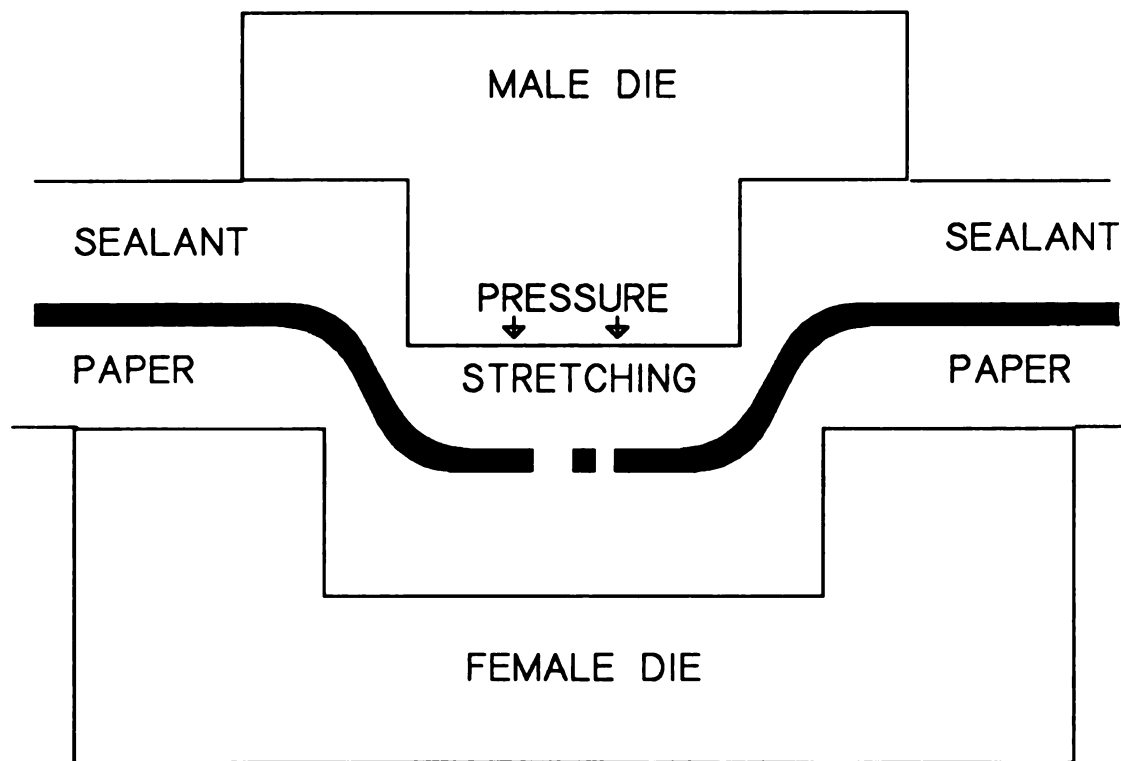


FIGURE 18 - THINNING OF SEALANT DUE TO SCORING DIE PRESSURE



DAMAGE DUE TO STRETCHING

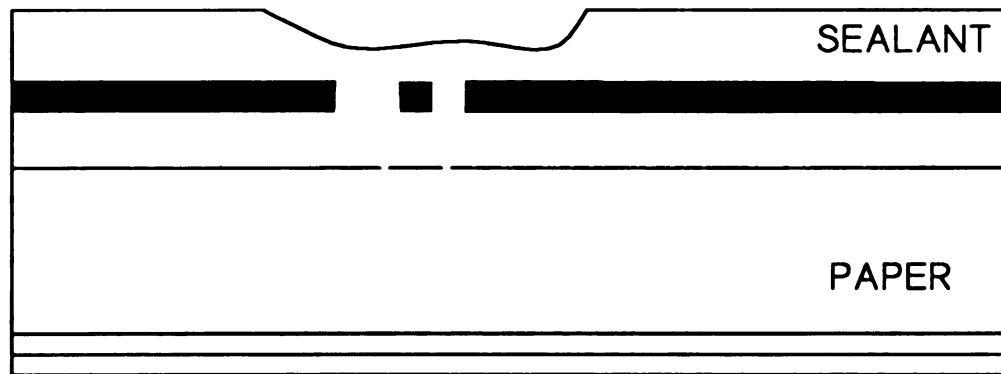


FIGURE 19 - STRECHING OF SEALANT DUE TO SCORING DIE PRESSURE

"False positives are less likely with Tetra rollstock because Tetra creases scores prior to laminating the paper. IP makes the scores and creases on the fillers. Since the IP paper is not precreased it is more likely to crack when cartons are formed."

One technique to locate this type of defect involves use of flexible probes with a piece of brine soaked gauze held on to one of the two testing probes by a rubber band. One probe is immersed in the brine solution outside the container while the second with a moistened pad is wiped along the inside surface. When the defect is located, the ammeter will indicate completion of a (weak) circuit.

Pseudo positive electroconductive defects are found at the K-creases in Tetra Paks by this method. Corner defects may be evident as well. Strong current flow will be observed by the microammeter when a break in the sealant layer exists, and the current is carried by the aluminum foil from the inner brine solution to the brine solution outside the package. This may be caused by scratches on the sealant surface.

The electroconductivity test is only presumptive. When conductivity is observed, it must be verified as a break in the hermetic seal or as a false positive. There are two methods for verification. One method is to dry the sample in an oven and retest using the electroconductivity meter. After drying, it sometimes is necessary to let the sample float, partially filled in the reservoir to permit capillary incursion of brine into micro leaks. If on the second test a package shows no conductivity, the false positive is sometimes attributed to conduction over the wet edge of the sample during the first test. Dye testing is used to confirm the presence of micro holes in the package.

Dye testing as previously described makes holes in packages visible. The dye is usually coated on the inside of the package. However, when breaks occur in the sealant layer, and the aluminum foil layer remains intact, the alcohol used in the dye dissolves the edges of the polyethylene and promotes delamination. This makes the defects difficult to inspect visually. Verification of all packages showing greater than 0 microamp is recommended by the BAM 24.53 (1984).

GAS DETECTION**Objective:**

To detect holes in hermetically sealed packages using detectors tuned to detect only the gas contained within that package.

Method:

A hermetically sealed package by definition does not possess holes. The package must be a barrier to the test gas so that it does not permeate through the package walls at a rate that may be detected by the leak sensing device. Gas concentrations may be detected by impact to a sensor. The sensor may be a heated element in which resistance varies in relation to gas molecules removing heat as they impact. Many gasses may be used: oxygen, nitrogen, carbon dioxide, natural gas and hydrogen are examples.

Materials:

Tank of gas

Method for trapping test gas within hermetically sealed food package

Gas detector: gas chromatograph or mass spectrometer

Procedure:

Gas obtained from air fractionation or storage tanks may be used to displace headspace gasses within food packages prior to closure. Air may be displaced by diffusion, forced displacement by pressure or vacuum, or by entraining the gas within the product being filled. After closure the gas concentration within the package must be higher than its concentration outside of the package. If there is a

break in the package the gas will move from its higher concentration within toward a lower concentration in the surrounding atmosphere. The detector must sense concentrations of the test gas which are higher than the normal background level.

Results:

Positive - detection of gas concentrations greater than the normal atmospheric concentration indicate a break in the hermetic barrier of a sample package. Confirm with dye testing to locate the hole in the sample package.

Negative - no detection of test gas concentration greater than the normal atmospheric concentration.

False Positive - detection of gas concentrations in excess of the normal background level may be the result of an increase in the test gas concentration in the testing area.

A test of the background concentration should be conducted prior to and following sample testing. Packages with a high permeability or large surface area may lose gas.

False Negative - internal gas concentration may be reduced by absorption by the product, reaction with a component inside the package, permeability if over an extended storage period, or by leaking out through a break in the package wall.

Discussion:

There are many gas detection systems adapted to package testing. The Oxtran™ oxygen analyzer by Mocon and the Permatron™ carbon dioxide detector are found in many laboratories. This section focuses upon

the helium leak detector by Varian, with particular application to its leak detection capabilities in paperboard, aseptically filled and sealed food packages.

Stan Alexander, of General Mills developed a testing apparatus for aseptic paperboard cartons using the Mocon Oxtran™. This apparatus may be epoxied to the package, and oxygen transmission into the package measured without leakage at the probe site. Bob Ater of Combibloc drafted a recommended practice for whole package testing based upon this method. (ASTM F2.5, subcommittee proceedings, 1 Nov 1984.) Carbon dioxide may be measured by infrared spectroscopy and can be readily quantified. ASTEC of Cedar Rapids, Iowa is working on a method to use CO₂ leakage as an integrity test for aseptic packages containing aluminum foil according to Tom Taggert at Lyons Magnus (1985).

Aluminum foil is a barrier to gas. If permeation occurs through a composite structure containing aluminum foil, the leakage can occur in only three places. First, pinholes in the aluminum foil may act as permeation windows. The permeation rate may be related to the area of pinholes and the permeation constants of the remaining layers of the packaging material. Pinholes occur in aluminum foil with increasing frequency as the material is made thinner. TABLE 45 shows the relationship between pinholes in aluminum foil and gauge thickness.

TABLE 45 (SHIELDS, 1985)
WVTR (FLAT) OF PLAIN BARE ALUMINUM FOIL

<u>1 GAUGE. = .00001 INCH</u>	<u>GM MOISTURE/100 SQ. IN./24 HR.</u>
25	.17
30	.07
40	.06
50	.045
60	.030
70	.02
80	.01
90	.005
100	.001

The second source of permeation in sealed packages relates to the tightness of seals. Stretching of seals in pallet sized aseptic bag in box products caused areas of product oxidation adjacent to the seals. Plastic fitments of Scholee Bags containing fruit purees showed similar areas of oxidation. Ralph Gygax of Ross Labs presented information on a literature search he made on gas detection methods for package leak testing at a meeting of ASTM, F2.5 in St. Charles, Illinois on 1 November 1984. He listed 11 test methods with sensitivities to detect from 10^{-3} to 10^{-11} cc/second gas permeation. (ASTM F2.5 Aseptic Subcommittee Report, 1 Nov 1984.)

The third source of gas movement through hermetically sealed package is holes and cracks.

"The ideal leak referred to in kinetic theory is a circular opening in a wall, whose diameter is at least 10 times the wall thickness (like a manhole, in effect). In the real world, most leaks are tortuous, sometimes multiple, paths of great length compared to cross section - more like long irregular wormholes." (Introduction to Helium Mass Spectrometer Leak Detection, p.25, 1980.)

Leak rates vary and some of the specifications recommending maximum tolerable limits are shown in TABLE 46.

TABLE 46
EXAMPLES OF VARIOUS LEAK RATE SPECIFICATIONS FOR
VARIOUS PRODUCTS AND INDUSTRIES
 (Introduction to Helium Mass Spectrometer Leak Detection page 8, 1980)

<u>PRODUCT OR SYSTEM</u>	<u>LEAK RATE SPECIFICATION ATM. CC/SEC</u>	<u>COMMENT</u>
Chemical Process Equip.	10^{-1} to 1	High process flow rates
Torque Converter	10^{-3} to 10^{-4}	Retention of liquid
Beverage Can End	10^{-5} to 10^{-6}	Retention of CO ₂
Vacuum Process System	10^{-5} to 10^{-7}	Dynamic System
I.C. Package	10^{-7} to 10^{-8}	
Pacemaker	10^{-9} or lower	Implanted in human body

The detectability of leaks - aside from the sensitivity of the detector depends on three factors: first, the pressure gradient, second, the flow rate through the hole, finally, the distance the detector is located from the hole. Gas flows from high to low concentrations seeking to fill any given volume at a single concentration with all parts in equilibrium. When a concentration of detectable gas inside a closed container is greater than the concentration outside, any flow escaping through a flaw in the container

may be localized. After it exits the container, the opportunity to detect gas depends on the distance the detector probe is held from the leak and the speed with which the probe is moved over the leak. (Forant, no date) Thus, two steps may be taken in finding the source of the leak. First, by limiting the surrounding air mass of a leaking container, any increase in detectable gas concentration within the outer vessel may be detected as a function of time. Second, upon identifying a defective container, a further investigation using a sensitive "sniffer" probe may locate the hole through which the gas escapes from the container. The device used for leak testing of many packages is the Varian model 938-41 Porta-Test helium leak detector.

This device has the reported capability of sensing helium concentrations as low as 10^{-10} (Felder, 1986). The principles of operation for the Varian model 938-41 are shown below.

1. At atmospheric pressure, molecules move as a liquid in response to pressure and atomic repulsion.
2. Partial pressure causes similar molecules to separate by equal distances. Helium molecules are uniformly spaced (at STP) at an atmospheric concentration of 5.2×10^{-6} .
3. In a vacuum, atomic forces are too weak to influence molecular distribution of gas molecules. Thus, it is possible to have voids and clusters of helium molecules inside a diffusion pump chamber.
4. Within a diffusion pump helium molecules are collected in hot oil where they have no apparent attraction or repulsion for one another. The oil has great affinity for helium molecules.
5. Diffusion pumps, however, have a limitation and it is impossible to capture 100 percent of the molecules in the vacuum chamber. Some gas molecules will escape through the top of the diffusion pump and move on to the detector portion of the device.

6. Molecules escaping from the diffusion pump are restrained by baffels placed in their path. Molecules moving randomly pass by the baffels after many rebounds from the walls and collisions with other molecules.
7. All molecules next pass through an electrically charged gate. Those molecules, atoms, and ions with a charge are collected by either the positive or negative plate. Noble atoms and molecules are possessing a charge pass through the gate.
8. After being sorted by the gate, all gas particles with a neutral charge (hydrogen, tridium, duterium, helium, nitrogen, oxygen, carbon, hydrocarbons, etc.) are themselves charged by bombardment of electrons from a hot glowing coil of electrified wire.
9. Neutral molecules passing through the charged gate continue in a straight line pass the glowing coil as it emits electrons. The loss of one electron following bombardment gives the formerly neutral particles a positive charge. This is shown below:

$$\text{He}(0) - e(-) \text{ --- } \text{He}(+)$$
10. Continuing in a straight line, the charged particles enter a magnetic field. The field causes particles to change their direction of motion. The field is calibrated to cause helium molecules with a positive charge to bend 90 degrees.
11. A metal plate with a very small slit lays in the path of the charged helium molecules. All other positively charged particles strike the plate and are scattered. Only positively charged helium molecules pass through the slit. The Varion model 938-41 detector may be described as a "tuned mass spectrometer" because it is designed to measure helium only.
12. Charged helium molecules strike the detector.
13. The impact is sensed, and amplified. The resulting signal is used to activate circuits for lights and alarms. The strength of the detector signal is proportional to the concentration of helium being taken in through the sniffer probe.

The ASTM has approved three methods involving helium leak detection.

1. ASTM E 493: leaks using the mass spectrometer leak detector in the inside-out testing mode.

2. ASTM E 498: leaks using the mass spectrometer leak detector or residual gas analyzer in the tracer probe mode.
3. ASTM E 499: leaks using the mass spectrometer leak detector in the detector probe mode.

Helium is injected into a small container and sealed. The container is then placed into an evacuation chamber. Using method E 493, the mass spectrometer senses the atmosphere of the evacuated chamber for helium. Method E 498 is used for rigid containers or vessels that can be mechanically evacuated. Method E 499 uses a probe to test one side of a container while helium gas at atmospheric pressure is flooded on the opposite side of the surface being tested. This method works with containers of all sizes.

Helium leak detection was evaluated by Sizer and Arndt (unpublished, 10 Sept 1986). Helium gas from tanks was used in place of nitrogen gas and injected into water before being packaged in 250 ml brik paks, using an AB9 form/fill/seal aseptic packaging machine. Hundreds of packages were produced. By varying settings on the AB-9 form-fill-seal machine, Brik Pak pilot plant personnel were able to create package defects typical of those seen under adverse production conditions. The following observations and conclusions are noted in TABLE 47.

TABLE 47
HELIUM LEAK DETECTION FOR BRIK PAKS

1. Helium can be detected leaking from holes in brik paks using the Varian model 938-41.
2. Water, product and foam can block holes preventing escape of helium gas.
3. Cold transversals (non-fused end seals) can be detected. However, pin tears with product in the hole cannot be detected. Fin tears (torn flaps) can be detected.
4. The closer the detector probe is positioned in relation to a leaking hole the higher the concentration of helium that will be detected. At 3 cm from a hole, helium can be detected.
5. A vacuum chamber could increase detectability of helium by creating a greater partial pressure for the gas to flow more readily.

Three difficulties would have to be overcome before helium gas detection could be usable as an on-line, non-destructive test method.

- a. Moisture droplets plug holes preventing the free flow of helium gas.
- b. A greater pressure differential is required than can be attained at atmospheric pressure.
- c. Foam inside the packages requires up to seven days to settle.

Gas detection methods offer a unique potential to test packages without creating extraneous and undesirable conditions associated with dye, electro- conductivity, burst testing or chemical etching. Helium, being a "noble gas" does not enter into chemical reactions and therefore will likely be safe in contact with food products. Helium may be injected in the same manner as nitrogen or carbon dioxide when used for modified atmosphere packaging.

INCUBATION

Objective:

To determine whether or not a package has lost hermetic barrier by holding containers at the ideal growth temperature for sufficient time to assure growth of spoilage organisms.

Method:

Hermetic integrity is the condition barring entry of microorganisms to a food product. By incubating we create the ideal growth conditions for spoilage organisms. The growth of microorganisms indicates either insufficient processing or the loss of hermetic barrier.

Materials:

- Sample packages
- Incubator with thermostat and recording thermometer
- Device for opening packages for visual inspection of product.
- pH meter
- Sanitary disposal of product
- Safe disposal of spoiled product.

Procedure:

Obtain representative sample packages.

Place packages in incubator for recommended period of time at recommended temperature.

Visually inspect packages for evidence of spoilage.

Open and inspect all (or some) of the packages following visual inspection. Check product suspected of spoilage with pH meter. Never taste incubated product if spoilage may have occurred.

Conformational dye testing to determine the presence of holes may be conducted on some packages.

Dispose of product.

Culture spoilage organisms to determine identity.

Results:

Positive - spoilage has occurred and is evident.

Negative - spoilage has not occurred.

False Positive - chemical reaction or enzymatic activities alter the products characteristics without microbial activity.

False Negative - the spoilage organism was present but does not grow under the conditions of incubation.

Discussion:

When all packages exhibit microbial spoilage this may be the result of under processing. When some packages display microbial spoilage this may be the result of package failures. The incubation period and temperature must be within the range required by the spoilage organisms or significant growth may not take place. Changes in the product must be detectable. Fermentation producing gas and lactic acid is a good spoilage indicator. Gas formation causes sealed packages to swell and lactic acid reduces the pH, which in turn may alter viscosity and appearance of the product. The spoilage organism(s) should be identified. Wide spread spoilage by a single species of microorganism indicates under processing. Knowledge of the support requirements of the spoilage organism may be valuable in redesigning the critical control points of the process. Spoilage by many different species may indicate environmental contamination resulting from defective packaging. For additional information refer to the Bacteriological Analytical Manual (BAM).

LIGHT**(VISIBLE LIGHT)****Objective:**

To detect holes in hermetical packages by the sensing of transmitted or reflected light.

Method:

Light may be detected by photo cells and used to open and close alarm circuits. Light may be transmitted through cutouts which receive foil pulltabs and an internal polyethylene barrier strip. Reflected light may be used to detect the angle of lid deflection formed by vacuum within metal.

Materials:

Packages capable of maintaining vacuum with a lid which deflects under vacuum.

Sealing device capable of creating a vacuum internal to the package.

Light and photocell with alarm device to detect defective packages.

Procedure:

This is a nondestructive test which has been developed to operate on-line. Packages which do not respond to the limits of acceptance are rejected. Evaluation of the sample following rejection may reveal the cause of rejection.

Results:

Positive - a package possesses a defect such as low vacuum or the absence or presence of a cutout.

Negative - a package conforms to the acceptance criteria.

False Positive - the testing device is defective.

False Negative - the testing device is turned off.

Discussion:

Most U.S. food processors using metal cans employ reflected light to detect loss of vacuum. By timing the light flash to coincide with the position of the moving can lid it is possible to isolate a consistent target location. By positioning a photocell at the point where the reflected light will impact when the lid is deflected by vacuum, a clear signal is received for cans which meet this critical control requirement.

Cutouts may be detected in a similar manner. A timing device is required to verify that the signal coincides with the positioning of the cutout. Receiving the light impulse at the required instant indicates acceptance. Similarly, when pulltabs are applied the light should not reach the sensor. Receiving the light impulse at the required instant indicates rejection.

LASER LIGHT**Objective:**

To detect holes and seal defects in hermetic packages by precise measurement of distance using lasers.

Method:**A. Holography, Wagner et.al., (1981).**

"The hermetically sealed food cans are tested in a chamber under either vacuum or pressure, in which a predetermined amount of stress is applied to cans. The surface of the can is viewed for the presence of fringes as the can ends deform in response to the applied stress. A hologram that shows the image of fringes is recorded by a reflected laser beam of a subject illuminated by a portion of the laser light (split beam). A hologram (recombined beam) of cans within the test chamber is recorded on a video tape and may also be exposed and developed in place with a liquid gate film holder. The pattern of fringes that occur on the surface of the can indicates the relative size of leak. To locate the leak in the can the stress applied to a pair of cans set side-by-side in the testing chamber. The stress is slowly varied, the seam area is photographically enlarged, and fringe control techniques are applied." (BAM 6th Edition, 1984, pp 24.27 and 24.88).

When conducted outside of a sealed chamber this method requires an absolutely still room with no movement of air during the test. The splitting of the laser beam, reflections from similar objects at different contour levels, creates interference patterns when the light recombines.

B. Timed Reflection

Using precise measurement it is possible to measure the distance from the point of emission to reflection and reception. This method was investigated by Natick Laboratories to detect fold-over wrinkles in retort pouch seals with limited success.

Materials:**A. Laser**

Vacuum Chamber (see BAM 6th Ed. 1984, pp 24.27, 24.88)

- B. No information, contact U.S. Army Natick Laboratories,
Natick, Mass. Young 1984, Wagner et.al. 1981.**

MACHINE VISION**Objective:**

To detect holes in hermetic packages by computer evaluation of photographs with predefined visible patterns. This system is designed to eliminate visual inspection of packages.

Method:

A video image is digitized. The photograph is divided into a grid and density of each cell is coded. Both greyscale and color density may be evaluated. The computer compares the coded patterns of the grid with patterns stored in the memory of the computer. Some systems evaluate the video images one at a time. Other systems use parallel computers to evaluate the video image with many different standards in shorter time.

Materials:

Video imaging system
Computer(s) with stored images for acceptance criteria
Strobe light (optional)
Packages

Procedure:

Packages are positioned in front of the video camera as they move along a conveyor belt. A strobe light stops the motion and highlights the inspection surface. Images are processed by the computer which produces an accept/reject signal. Rejected packages are ejected from the packaging line.

Results:

Positive - a package having a recognized defect is detected and the package ejected from the flow of production.

Negative - a package having a recognized pattern consistent with the acceptance criteria is approved by machine vision and continues past the rejection point down the flow of production.

False Positive - the system cannot distinguish between two similar patterns and mistakes a cosmetic characteristic as a rejectable defect.

False Negative - the system is incapable of recognizing defects which should be rejected.

Discussion:

Visible light - video imaging followed by digitizing and computer analysis has proven to be an effective method for recognition of gross objects. Machine vision systems are scanning packages and containers for cap positioning, label placement, fill heights, seals, dents and defects, Swientek (1987). However, a survey of 63 manufacturers of machine vision systems for the food industry (Food Processing, 1987) failed to identify any with the capability to identify seal defects in fusion seals. The problems of machine vision are further compounded when laminates overlay the fusion seal (RDA, 1984).

"Automatic detection of heat seal defects is not, at present, commercially developed. The potential to automatically detect package seal defects through the use of infrared radiometric scanning, television cameras associated with computer analysis, and through thickness measurement all have potential for the future." (Downes, 1984).

**Metal cans are checked visually for seam damage, seam thickness, overlap, etc . . . "But a systematic inspection of seals is not so easy."
(Arema and Schram, 1980.)**

PROXIMITY DEVICES**Objective:**

To detect holes by measuring changes in deflection of a hermetically sealed package as a function of time.

Method:

The position of a package containing metal may be established by the strength of a magnetic field using a galvinometer. By comparing two readings of the same container as a function of time we may determine whether or not the package has altered its shape.

Materials:

Package containing metal, capable of deflection
Two galvinometers

Procedure:

This is a passive detection method which does not contact the package. A test sample is placed on a conveyor belt. The belt positions the package a specific distance from the first galvinometer. A measurement is recorded as the package passes. A few minutes later the same package moves past the second galvinometer again at a specific distance from the sensing point. Slight changes in the profile of the package results in the package being rejected from the line of production.

Results:

Positive - there was a measurable difference in the profile of the package as it moved from the first to the second sensor.

Negative - there was no measurable difference in the profile of the package as it moved from the first to the second sensor.

False Positive - a change in the package profile resulting from impact or from packages which are out of order on the production line.

False Negative - the sensor cannot detect slight changes in the container shape because insufficient time elapsed while the package traveled between the sensors.

Discussion:

Sensing devices which measure changes in the strength of a magnetic field have been used for years in metal detection devices at many food processing plants. Two applications of this technique for detecting packages with holes in them have been shown effective. The Taptone model 4014-1A Automatic Profiler from Benthos is designed for aseptic paperboard boxes with gable tops or brick shaped, (Andres, 1982). The device relies on a partial vacuum within the container to maintain a concave profile on the front of the package. Each package passes two proximity meters as they move down a conveyor at a constant speed. If either detector measures a strong signal indicating a package is convex, it is ejected. A hole in these packages permitting air to enter will allow the package to relax and bulge slightly as the product contained inside settles. Slight changes in profile are sensed by tying the two detectors electronically. A slight bulge developing during the time required for the package to move between the detectors will give a stronger signal at the downstream detector. These

packages are then ejected from the line. (Carlson 1981). Andres (1982) reported that while only 10 inches of conveyor length is required, the longer the product is held, the smaller the hole size that can be detected. The only limitation is that the side of the container with a seam must be away from the profile probe.

By adjusting the proximity detector, it is possible to define a profile for a container to be used as a reference standard by the proximity detector circuitry. As will be described in the section on "sound", a dud detector senses only a precise target area. The advantage of this detector operating by comparison of signals from a sample to a standard, is its ability to sense a profile.

"Assuming the production process is controlled, the curvature will be consistent. If vacuum is lost, and thus the curvature, the "profile" changes. This results in a proportional readout of the degree of vacuum. The vacuum profiler senses this profile change, and rejects the container with the defective profile." (Taptone promotional information, no date).

A Taptone model 4104, installed to sense button caps on glass bottles inside shrink wrapped shipping trays, was installed at New England Apple in Littleton, Mass.

"Using electromagnetic waves, the sensors pick up the proximity, or closeness of the cap. These readings are used by the device to calculate the curvature of each cap. Should curvature not match one of the three preset standards, the detector engages an air operated reject mechanism. The entire case is pushed off the line." (Food Engineering, 1984).

According to Benthos (promotional information, no date), the following advantages are attained with the Taptone model 4104 vacuum profiler.

1. Vacuum can be measured even with product on the lid.
2. The device is not affected by can height changes, double seam variations, or embossed end codes.
3. There is no contact with the can except for rejecting.
4. No spacing of cans is needed and multiple heads may be used to test up to four rows of cans in a shipping case as it passes beneath the sensor wand.
5. This detection system is usable with any metallic surface that flexes with vacuum or pressure.
6. This device simultaneously rejects low vacuum or swollen cans from normal vacuum cans
7. Fully automatic system.

The Seal Integrity Tester (S.I.T.) by Taptone is designed for testing flexible membranes which are heat sealed onto food packages. These membranes must contain a metal barrier material. According to Benthos the parent company of Taptone, this system -

"can be customized to accommodate practically any container size, shape, or material construction - including sealed plastic caps/trays, sealed glass jars, sealed metal containers, and sealed paperboard canisters." (Rice, 1986).

This device operates by placing a sealed container in a receptacle, applying 80 to 100 psi air pressure to only the outside edge of the fusion seal perimeter and measuring for minute expansion of the septum using the proximity device. If the flexible septum does not move during the test the

container is presumed to be sealed completely. However, the sensitivity is limited to .125mm (.005 inch) lid deflection which is affected by leak size, applied gas pressure, container headspace volume, and viscosity of the test gas. (Taptone SIT promotional information, no date.)

The Puffer by Taptone is a second device for continuous on-line testing of packages having flexible lids to increase volume within the headspace of sealed containers. The bulging lids are scanned as they exit using an analog proximity device. The field is interrupted in a linear manner as the foil passes through the magnetic field. Liquid products which form a meniscus occasionally block the flow of escaping air from minute holes. A roller device which depresses the expanded lid of heated containers has increased the sensitivity of this device significantly and overcomes limitations brought about by fill volume and headspace variations, (George Coolidge, Benthos, 1988).

The Seal Integrity Analyzer by Seal Integrity Systems is a laboratory model nondestructive package tester that forces air or helium at pressures up to 300 psi external to the seal area. This device uses a proximity meter to measure the position of the package seal before and after pressure is applied. The application of external forces to packages enables more precise separation of good and bad samples than is possible by passive sensing. This system may be developed into an on-line nondestructive test method by Pacific Engineering of Richland, Washington.

SOUND**(ULTRASONIC)****Objective:**

To passively sense air moving through small orifices in packages possessing an internal vacuum by monitoring ultrasonic wavelengths.

Materials:

Packages possessing internal vacuum
Microphone, amplifier, frequency filter

Procedure:

Packages moving on a conveyor belt shortly after being vacuum sealed are passively inspected by adjacent microphones for the sound of leaking gas. Specific frequencies are monitored to eliminate background "noise".

Results:

Positive - a package exhibits "whistling" indicating a leak is present permitting air to enter the package.

Negative - no sound is emitted within the specific range of frequencies being monitored.

False Positive - background noise occurred within the frequency range being monitored .

False Negative - small food particles or water droplets block escape of air or reduce the flow rate so that neither the frequency or amplitude are within the range of the receptors.

Discussion:

When metal cans and glass jars containing shelf stable foods are opened, a sound is produced by air rushing into fill the vacuum. Similarly, when containers under pressure contain small holes, the sound of escaping air is available.

"Acoustical leak detection, a variation of inside-out testing uses the sonic (or ultrasonic) energy generated by a gas as it expands through an orifice. Anyone referring to a large leak as a "whistler" is noting the acoustical energy produced. Acoustical leak detection is widely used in testing ductwork for leaks, and in testing high pressure lines. It requires modest instrumentation and is fairly simple and fast. Its sensitivity, however, is limited to about 10^{-3} std cc/sec." (Introduction to Helium Mass Spectrometer Leak Detection, p.13, 1980.)

Tom Taggart (1985) reported that ASTEC is developing a leak detector involving amplification of sound for aseptic cups with flexible lids. Bates (1988) reported on a method to employ ultrasound at the low end of the kilohertz spectrum 20 to 100 kHz as an on-line nondestructive test method. The human threshold for sensing high frequency sound is 16.5 to 20.kHz.

ECHO**Objective:**

To actively sense the frequency of echos from hermetically sealed containers.

Materials:

Transducer capable of transmitting on one frequency with sufficient amplitude to create echos in packages.

Food packages containing viscous or liquid products or packages with internal vacuum.

Procedure:

Filled and sealed packages on a conveyor belt or within sealed shipping cases are targeted by sound waves. If the package contains a vacuum the package walls are under tension. The impact causes portions of the package to tympanate sympathetically. The reflected echo occurs at a different frequency than the transmission. Conforming packages possessing internal vacuum reflect sound at a different frequency than packages which do not possess internal vacuum. The change in sound between good and bad packages is audible and serves as an indicator for operators conducting inspection and for automatic sorting of production.

A second sound sensing device common to the food industry is found in many establishments. The Benthos, Portable Taptone uses sound measurement to determine the tightness of safety button lids on glass jars of baby foods at Beech-Nut and Gerbers.

"Rather than do destructive package testing as an inspection procedure, workers were manually removing sample jars from the partitionless, wrap-around cases, and tapping the tops of them. A trained ear could readily detect vacuum loss and appropriate correctional measures could be quickly initiated. However, this procedure was inherently subjective and did not meet the highest standards of consistent reliability which the company sought." (Tapetone promotional information, no date).

The Taptone equipment installed at Beech-Nut senses vacuum, missing lids and missing jars in closed wrap-around shipping cases at 50 cases (24 jars/case) a minute, (Repko and Rice, 1984). While this device works well on button lids, there are problems applying the device to cans. Heavy gauge metal can lids with more than one tension bead do not resonate as well as flat lids or those with only one tension bead. Embossed lids are difficult to resonate so the coded (stamped) end of a can should not be presented to the detector. Tight shipping cases enhance the accuracy of the sensing device by accurately arranging cans within a defined target area, (Coolidge, 1988). While a wrap-around shipping case provides the best container in terms of tightness and uniform corrugated thickness on the top of the case, a tolerance of + or - 1/4 inch should be maintained in vertical and horizontal positioning for the sound response to be concise, (Tom Flagg, Benthos, 1987).

A portable taptone device has been used on 250 ml brik paks containing milk based products. When these packages spoil without gas formation they are difficult to identify without opening the packages. If the coagulated product resets against the top of the 250 ml brik pak and forms a film adhering to the package the sound waves lose amplitude when passing through. The portable Taptone amplifies the sound and the trained

operator may recognize differences in amplitude. It is possible to separate normal from coagulated milk based product in 250 ml brik paks by this method, (George Coolidge, Benthos, 1987). In 250 ml Brik Paks lacking headspace gas, this device may be capable of detecting the formation of gas bubbles, or a leak which permits air to enter the package. When a 250 ml Brik Pak loses its normal tightness the package is free to vibrate and gives a louder response to the portable Taptone than normal packages, (George Coolidge, Benthos, 1988).

TENSILE TESTING**Objective:**

To measure the tensile strength of a (1/2 or 1 inch) section of fusion seal.

Materials:

Sample packages to be tensile tested sample cutter, samples cut perpendicular to seal, specify sample width

Tensile testing device

Scissors

Procedure:

A representative sample is removed from the production line. The sample is cut open and the contents removed. Care is required not to disturb the fusion seal to be tested. The cleaned sample package is cut to produce a test strip. The test strip must be cut perpendicular to the seal to be tensile tested. Both ends of the test strip are secured in separate clamps. A screw drive moves one screw clamp away from the other (usually downward). This creates a 180 degree separation of the seal. The force required to fully separate the seal is observed.

Results:

Positive - a sample separates at a tensile strength less than the established standard.

Negative - a sample separates at tensile strength greater than or equal to the established standard.

False Positive - a sample separates at a tensile strength less than the established standard because of equipment miscalibration or greater separation speed of the jaws.

False Negative - a sample separated at a tensile strength greater than or equal to the established standard. However, a portion of the sample failed at a tensile strength less than the standard.

Discussion:

Tensile testing is an objective method for measuring the force required to separate bonded materials by pulling in opposite directions (180°). The objectivity stems from the restriction of variables. By using samples of identical dimensions held under a constant force, pulled at a constant rate, and tested under controlled environmental conditions to which the samples are equilibrated, a great deal of variation may be eliminated. The test for fusion seals is ASTM D882, volume 35, Fed. Std. 406, method 1013, (Mackson, et.al. unpublished 1981). The first application of ASTM D882 test for Tensile Properties of Thin Plastic Sheeting to a flexible package for low-acid foods was by Natick Laboratories while developing the retort pouch.

".... the tensile test can best be used for surveillance of the sealability of materials and as a spot check on sealing conditions and equipment operation. Tensile tests have utility as a quality assurance tool for assessing the inherent sealing qualities of flexible packaging films and should be a mandatory test." (Lampi et.al., 1976).

Lampi, however, recognized the limitations of this method and recommended that seals be tested by a burst test as well as the tensile test.

"By definition, the tensile test measures the total force or weight required to cause failure over the total width of each sample strip. However, the deflection of any channel or stress points and the effect of occluded particles or other small weak areas within the seal are obscured by the adjacent high strength areas. Tensile tests should therefore be supplemented by burst tests." (Lampi, et.al., 1976).

Early pilot plant production of retortable pouches under Phase I at Swift and Company in Chicago proved the usefulness of tensile testing for seal strength.

"Duxbury et.al. (1970) reported no problems in achieving tensile strength of 2.8×10^3 N/M (16 lbs/in) with a 12 micron/9 micron aluminum foil/75 micron modified polyolefin laminate - neither retorting nor three month storage had any important effect on the tensile strength. Similar results with other films have been noted at the Natick Development Center. The tensile strength for different materials (laboratory sealed) ranged from 1.9×10^3 to 3.3×10^3 N/M (11 to 19 lbs/in), yet all packages performed adequately." (Lampi, et.al. 1976).

Tensile testing cannot predict performance of flexible package seals within the retort during processing.

"Pflug and Long (1966) studied seal tensile strengths under retort conditions and concluded that the behavior of a seal under such conditions cannot be completely predicted by room temperature tensile tests. They affirmed the value of fusion as a more important criterion than tensile strength. Their material was polyester/foil/vinyl, but similar relationships are likely with other films." (Lampi, et.al. 1976).

Polvino (1986) evaluated test methods for retortable plastic containers with fusion and peelable lidstock. He determined that the deficiency of the ASTM D882 Tensile Test is that it tests only a portion of the seal. Tedio

Ciavarini (Natick Labs, 1982) stated that some MRE processors were permitted to drop the tensile test from their on-line quality control requirements because they had demonstrated a history of "conforming production", and had demonstrated through testing that they were capable of monitoring seal integrity with the burst test alone.

The advantage of the tensile test is that it eliminates many variables and focuses on the strength of the seal bond for a sample of the package. This test is useful in establishing the conditions of time, temperature and pressure. The disadvantages are shown in TABLE 48.

TABLE 48
DISADVANTAGES OF TENSILE TESTING ASTM D882

1. Tests a portion of the seal, not the entire package.
2. Difficult to test semirigid containers with flexible lids.
3. Does not predict performance at retort temperatures.

VACUUM TESTING**Objective:**

To cause the movement of air out of a sealed container through leaks by applying external vacuum.

Method:

Closed packages are placed inside a sealed testing chamber and vacuum created to cause movement of air through leaks in the packages. Deflection of the package may be measured as a function of time to determine whether or not leakage has occurred. Occasionally product may move outward through the holes. Poorly sealed lids may open. If the vacuum chamber contains water bubbles emitting from holes in packages may be observed.

Materials:

Vacuum chamber and vacuum pump
Water (optional)
Packages to be tested

Procedure:

Obtain representative sample from production line. Place them inside of the vacuum chamber. Evacuate chamber. Observe effect as deformation of the container or emission of air bubbles or product. When vacuum is released, observe packages to determine if they have regained their original shape or if atmospheric pressure causes them to appear slightly crushed.

Results:

Positive - a leak in the test package causes air or product to emit through holes in a container. The container ruptures or the lid separates due to a weak closure. When the vacuum is released the package appears distorted or crushed by atmospheric pressure.

Negative - the package distorts under vacuum but no loss of product or air is observed and when the vacuum is released the package assumes its original configuration.

False Positive - air clinging to the surface of the package is mistaken for bubbles emitting from a defect as vacuum is applied to a chamber filled with water.

False Negative - food particles prevent movement of air out of the container while vacuum exists.

Discussion:

Vacuum testing is a valuable indicator of hermetic integrity for metal cans and glass jars. Flexible and semirigid containers conform to external and internal pressures. By creating a vacuum external to a flexible package, we may create the pressure necessary to move gas through a small leak. This method requires that the package being tested contain some headspace gas. The ASTM Packaging Guide (17 April 1985) recommends vacuum testing of packages underwater. The basis of the test is that by reducing the external pressure, gas bubbles will escape and be visible. However, it is possible for a hole to be large enough to allow microbial penetration yet be too small to permit passage of air bubbles.

To conduct a vacuum test underwater, the following equipment is required as described in TABLE 49 (Toledo, 1973)

TABLE 49
EQUIPMENT FOR UNDERWATER VACUUM BUBBLE-LEAK TEST

1. Bell jar, glass or plastic with tight fitting lid
2. Water to cover the package within the bell jar
3. Vacuum pump
4. Vacuum gauge
5. Valve
6. Grease to attain tight gasketing of lid on chamber

Before conducting the test it is necessary to degas the water. This may be done by using boiled water which has been cooled to room temperature, or by subjecting the water within the bell jar to a vacuum for an extended period of time.

Both Rampart Packaging and Continental Can Company recommend vacuum testing of cups with fusion and peelable lids underwater with a vacuum greater than 6 psi but less than 8 psi. This is done in conjunction with burst testing of samples. Both tests are conducted every thirty minutes during production, Garrett, (1985).

Paul Grabowski of Kraft used a vacuum test device and determined that defects in the seals of retort pouches could not be reliably measured with this method, (Grabowski, 1985). An external vacuum chamber may be used to deflect a flexible lid upward from a rigid or semirigid container to determine seal integrity. Toledo (1973) reported that a micrometer placed atop the domed seal may be used to measure deflection. Lids which do not bulge sufficiently to move the micrometer the required distance are

rejected. This method may work with a microswitch or a proximity detector. Briggs, (1986) reported that Air Logic Systems has designed a vacuum leak-up device suitable for detecting holes as small as three microns in flexible lidstock on semirigid containers.

VISUAL INSPECTION**Objective:**

To detect defects in food packages by visual inspection.

Method:

Visual inspection of samples or of every sealed package produced.

Materials:

Conveyor belt or inspection table
Adequate lighting
Trained inspectors

Procedure:

Obtain samples from the production line
Visually inspect critical areas and all remaining surfaces of the package. Replace package on the production line

Discussion:

Visual inspection of food packages is the most relied upon procedure. In many instances it is inadequate.

"In a typical cannery or vegetable processing plant, about 50 percent of personnel are manual inspectors. Drawbacks to manual inspection include worker fatigue, subjective judgment, operator error, and labor cost." (Swientek, 1987)

During inspection of retort pouches at MRE processor plants, fatigue of inspectors has proven to be a significant source of problems. At peak efficiency, inspectors may cull 85 percent of defective packages from the

production line during the first inspection. The incidence of defective pouches increases during retorting and following a second visual inspection, in cartoning. The requirement for visual inspection, and lack of a reliable on-line nondestructive testing device for 100 percent inspection of production, has priced this package out of the commercial market. During the summer of 1986, all of the MRE rations of the U.S. Military were on hold by the Surgeon General while inspection was conducted at storage depots around the world (Prinky, (1987)).

Guidelines for inspectors for retort pouches, aseptic paperboard packages, plastic retortable cans with double seams and fusion seals issued by the NFPA/FPIG contain only visually detectable defects. The significance of unseen defects is proprietary to container manufacturers and food processors. Visual inspection therefore is limited in scope and application.

"Until the use of nondestructive instrumented techniques merit their expense, visual examination in addition to fusion testing will be necessary to assure the absence of heat creep, significant wrinkles (over one half the seal width), surface irregularities, and occluded matter in the seal area." (Lampi, et.al. 1976).

Metal cans are all uniform in shape and temper and are easily tested on-line using nondestructive techniques. Flexible packages utilize many different sealing techniques. Different sealing methods give different visual affects. In some instances two different types of flexible packages will have identical visual effects, but on one package it is cosmetic and on the other it may be critical.

While visual inspections of retort pouches rely on distinguishing a 1/8 or 1/16 inch clear path of fusion (Young 1984), processors of plastic retortable cups with fusion lids are looking toward new developments - embossed rings in the seal area.

"If you see 90 percent of (the) embossed ring in (the) seal (area) you have a good seal visually. Embossing gives an operator visual inspection (capability). Embossing does not increase the integrity of the seal. It is an excellent first line indicator that (the) heat seal operation is going correctly. When it is not present it bares scrutiny." (Marceg, 1987)

Bosch projects the embossing ring from the heat seal die. Continental Can Company also uses embossing with the heat seal die. Hunt Wesson uses 90 percent embossed seal as their visual inspection guide, but they prefer to see 100 percent of the image as their in-house visual inspection criteria. Both Genesis and Mahaffie Harder offer this feature in sealing dies used for semirigid retortable trays. Both Continental Can Company and Rampart Packaging believe that less than a 90 percent visually detectable ring is a cause for concern on the part of the processor. However, neither wants to see a requirement of 100 percent visually detectable embossing as a requirement of regulatory agencies. Embossing does not adversely alter the seal integrity, but it is an excellent visual indicator. Vacuum leak testing underwater and a pressure burst test are used as secondary indicators to test seals that do not display at least 90 percent of the embossed ring. According to the NFPA/FPIG subcommittee on plastic retortable and aseptic cups with peelable and fusion lids, the following conditions may be assured (NFPA/FPIG, 23 April 1987) TABLE 50.

TABLE 50
EMBOSSSED SEAL RINGS AS VISUAL INDICATORS

1. A non-uniform pattern may indicate an area without fusion. This, however, is not a reliable visual indicator.
2. Embossings in the seal area do not alter the strength of a fusion seal when measured by burst or tensile tests.
3. Extreme pressures causing thinning of the sealant layer to .001 inches does not decrease the strength of a seal as measured by burst or tensile tests.
4. The strength of a fusion seal is mainly in the dam which is extruded from the seal area and set up along both sides of the seal forming a hermetic barrier.

In 1986 Campbell's Soup Company was the only food processor using fusion sealed semirigid retortable containers. All containers were visually inspected the same as retort pouches. Bouncing a beam of light or sound off the septum is not considered a reliable inspection method. The retortable soup bowl with a fused flexible lid cannot be inspected like a metal can. U.S.D.A. requires the packer to indicate what line tests are used in lieu of 100 percent visual inspection and to state the reliability of the test method used. Reliability can be difficult to establish.

"There is no such thing as a perfect seal. However, this is only a problem when it goes out of the specification of the manufacturer." (Stephanovich, 1986).

For paperboard aseptic packages, the cetrelli bar impression serves as a visual indicator of the condition of the fusion seal. When the width of the impression is less than specified, the process must stop. Lack of visual impression is taken as a critical defect - absence indicates nonfusion or "cold seals". For International Paper, seal to seal variation will be plus or

minus 1mm in height (seal width). If the tool height is less than 1.5mm the sealer will be shut down, DiGeronomo (1986).

When seals are manually pulled apart, fiber tear is a significant visual indicator. Seal peeling is done on the production line for Combibloc, Tetra Pak, and International Paper aseptic containers. In the U.S., these three package types require either evidence of fiber tear, sealant - foil separation, or separation at the edge of seal fusion indicating that the package seal was stronger than the interlaminar strength of the packaging material. Visual inspection of peeled seals from these three package types, showing voids where plastic laminate once adhered to the foil layer on the inside of the container is also a reliable visual indicator.

Thermoformed containers for low-acid foods should be visually inspected at start up and every half hour following. Some processors elect to inspect and record their findings every 15 minutes during production. Destructive integrity tests should be conducted at start up, and not less than every four hours during production. Many processors conduct destructive testing every half hour, with Q.C. and the line operator alternating.

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