2001

To investigate the reasons for moisture ingress in ready to drink mixes

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To Investigate The Reasons For Moisture Ingress In Ready To Drink Mixes

By

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A Thesis

Submitted to the

College of Applied Science and Technology

Rochester Institute of Technology

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Department of Packaging Science

2001-2002

COLLEGE OF APPLIED SCIENCE AND TECHNOLOGY

ROCHESTER INSTITUTE OF TECHNOLOGY

ROCHESTER, NEW YORK

CERTIFICATE OF APPROVAL

M.S. DEGREE THESIS

The M.S. Degree thesis of Rahul Eklahare has been examined and approved by the thesis committee as satisfactory for the thesis requirements for the Master of Science Degree

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5/21/01

Title of Thesis : To Investigate The Reasons For Moisture Ingress In Ready To Drink Mixes

Acknowledgements

I would like to express my sincere gratitude to Professor Fritz Yambrach for his guidance and patience during the research of this thesis. He has been a terrific source of motivation to me.

A special thanks to Mr. Mike Bayusik of Ontario Foods who gave me an opportunity to work on this project. He provided all the support and encouragement to carry out the tests and research work.

I would also like to thank Professor Deana Jacobs to have provided all the assistance that I needed as well as the interest she took in my research.

Abstract

The objective of this study was to determine the reasons for moisture ingress in ready to drink mixes. The containers for this product were supposed to be moisture resistant. However, after a certain period it was observed that the product inside was prone to caking and lump formation due to moisture ingress.

The dimensions of the containers were compared with the actual dimensions and the containers were exposed to accelerated aging to simulate a longer duration of shelf life. Several recommendations were made for new design of containers which were also tested for moisture ingress. Some observations were also made at the packaging line. The results indicated that the existing containers were defective and the recommended containers supported all the hypotheses. There is good scope for further research in this field especially in the areas of environment testing at the packaging line and real world testing of the new containers

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Introduction

Business in the late 20th Century and the 21st Century is not restricted to manufacturing products and selling them to customers; it is focused more towards satisfying the customers' needs and desires. Customer is a king of sorts with different brands of products of the same type chasing him in every market, store, mall and shopping center.

Customer satisfaction drives companies to strive to make their products better and cost less. Companies invest thousands of dollars in developing quality parameters, quality controls etc. which would ensure cost reduction and cost control in the long run.

Increased competition has led to reduction in prices and lower margins. Business for most industries is based on low margins and high volumes. Companies cannot afford inefficiencies and losses during the entire production and distribution process or the supply chain.

For products which have a restricted shelf life and can be subjected to spoilage with inefficient handling, companies have to ensure that the products are sold within the specified time frame and are not spoilt in the supply chain.

However, in real world business, losses do occur, spoilage does occur, customers tend to get defective goods and to salvage their reputation companies have to accept the sales returns and bear losses. These losses run in to millions of dollars annually. They affect the margins and in some cases the bottom line of many organizations.

The 4 P's (product, price, place and promotion) of marketing as suggested by several marketing Gurus are not sufficient in today's market place to ensure a good business cycle. Another component, also a 'P', known as *packaging* plays a very important role in the marketing aspect as well as the product safety and durability aspect.

There is probably not a single product today which isn't packaged and sold. Packaging has an important role to play in maintaining the quality of the products till they reach the customers.

Packaging for food products, pharmaceuticals and medical devices is one of the most challenging fields today and is responsible for maintaining the shelf life, safety and quality of these products. Packaging minimizes or in some cases eliminates product spoilage due to moisture, microorganisms, light, temperature, gases in the atmosphere, dust, insects and rodents.

It is easier to investigate spoilage in a food or pharmaceutical product today by first investigating the package for any inefficiencies. If the package is found to be inefficient, modifications can be made and the problem is solved. If the package is found to be perfect, then a long process of investigating the product, its manufacturing process, its composition and environment has to be undertaken. In most cases, however, the problem is the package and the problem is solved in the initial stage itself.

The study here focused on a similar issue where a company had been affected by sales returns due to apparent spoilage of a product. The study was an attempt to investigate the package of the product as a possible reason for that spoilage. This study was restricted to the initial stage of investigating the package only and not the product. The following chapters give a detailed description of the problem and the steps carried out in the investigation of the package. Several tests and results obtained are also included for the benefit of the reader. The last chapter deals on scope for further research and on areas which were beyond the scope of this study.

The Problem and Its Setting

The product here was a ready to drink mix which essentially consisted of sugar. Sugar is hygroscopic and prone to moisture absorption and has to be packaged properly. The product was manufactured, packaged and then distributed to various retail outlets across the country. The distribution environments as well as the region-specific environments for retail varied considerably across the country. There were regions of high humidity where the product was prone to moisture absorption by moisture ingress through the package while there were low humidity regions where there would be little or no moisture absorption through the package. In certain cases the climatic conditions varied considerably during distribution and the product was exposed to varying conditions over a certain period till it reached the retail shelf.

The container was supposed to be moisture resistant. However it was found that over a certain period, there was lump formation of the sugar due to moisture ingress which was not acceptable to consumers and resulted in large scale sales returns. This meant that the container, designed for moisture resistance wasn't as effective.

The study was carried out to investigate the reasons for moisture ingress which resulted in lump formation. Results from the study were used to make several modifications in the package and process to make the product suitable for marketing and distribution.

Ready to Drink Mixes

The ready to drink mixes are a class of dry products which contain only 1-3% moisture and have e.r.h. values below 20% and in some cases below 10%. The humidity of ambient air is rarely in this low range and such products tend to absorb moisture freely from the air surrounding them. These products are of porous nature and have a high surface to weight ratio, thereby increasing the hygroscopicity. The uptake of moisture being so rapid that packaging should be done in an environment where a low relative humidity is maintained. These products are also susceptible to oxidative deterioration and must be packaged in containers which are impermeable to water vapor and oxygen. If these products are inadequately packaged, they cake (form lumps) and will not reconstitute in most cases, and can also lose much of their volatile flavoring components.

Several studies have been conducted on packaging for hygroscopic products and moisture ingress through containers. The reasons attributed to this are :

1) Dimensional instability. Improper cap and bottle assembly.
2) Material selection for cap and bottle.
3) Manufacturing process of the cap and bottle.
4) Machine variations.
5) Humid conditions at the packaging line. No special care taken to make the packaging line more efficient for packaging of such moisture sensitive products.

i) Moisture absorption in Snack foods and Bakery products has been of concern and several studies have been undertaken to investigate the reasons for moisture ingress through the package.

A study by 'Food Product Design' – Weeks Publishing Company and authored by Kimberlee J. Burrington who is the whey applications program coordinator for the Wisconsin Center for Dairy Research in Madison, WI –

Faulty seals in packaging (including MAP) result in a reduced shelf life due to moisture absorption. Proper means to test the integrity of the package at the packaging line is important.

The article states that 'water migration is a difficult process to control in a baked product, especially when products possessing different moistures and available water are being combined' and this can only be controlled by binding of water and good packaging.

ii) 2000 North Carolina Flue-Cured Tobacco Production Guide NC State University by M.D. Boyette -- Extension Specialist, Department of Biological and Agricultural Engineering

Measuring and Controlling Moisture in Cured Tobacco

Cured tobacco is hygroscopic. Hygroscopic materials have a physical affinity for moisture. In tobacco, this moisture is absorbed from the water vapor in the air surrounding the leaf. The absorption of water by cured tobacco leaves is a complex process and is dependent on many biological and physical factors. Biological factors include the properties of the leaf that vary with variety, cultural practices, stalk position, and weather. The Physical factors include ordering temperature and humidity, air velocity around the surface of the leaf, and quantity and arrangement of the leaves.

The article states that :

1) the rate of moisture absorption increases with increasing relative humidity
2) moisture absorption rates also increase dramatically with increasing temperature

Different methods of drying have been suggested for preserving tobacco but the important point here is the importance of maintaining environmental factors for preservation of hygroscopic products at every stage.

iii) A study by Pittsburgh Corning FOAMGLAS® for Hershey Creamery Company, Harrisburg, PA discusses insulation systems for products to prevent moisture absorption in cold storage. It highlights the following important points :

Materials with poor dimensional stability or which lack in compressive strength, would result in the insulation system compressing under load, warping, or expanding and contracting and eventually causing distortion. This distortion may result in water vapor or water penetration.

Thus reliability of materials is an important consideration in designing packages for moisture resistance.

Sub-problems

The following sub-problems resulted from the above discussion:

1) To determine whether the dimensions of the containers were as per the specified dimensions and within the tolerances
2) To evaluate moisture ingress through the package system (consisting of the body and the cap)
3) To evaluate the packaging line environment and limitations of the process

Approach to Statistical Analysis

There was an attempt in this research to deviate from the classic quantitative approach of statistical analysis to a qualitative approach.

The approach of this research was such that it was more focused towards a pass and fail analysis which led to another test which was again subjected to a pass or fail analysis and so on.

The methodology involved was called as Negative Case Analysis or Analytic Induction.

Analytic induction is a method of data analysis described by Florian Znaniecki (1934) who named the method and systematized many of the associated ideas. Analytic induction can be contrasted with defining and using terms in advance of research. Instead, definitions of terms are considered during hypotheses that are to be tested. Inductive, rather than deductive, reasoning is involved. This allows for a modification of concepts and relationships between concepts, which occurs throughout the process of doing research, with the goal of most accurately representing the reality of the situation.

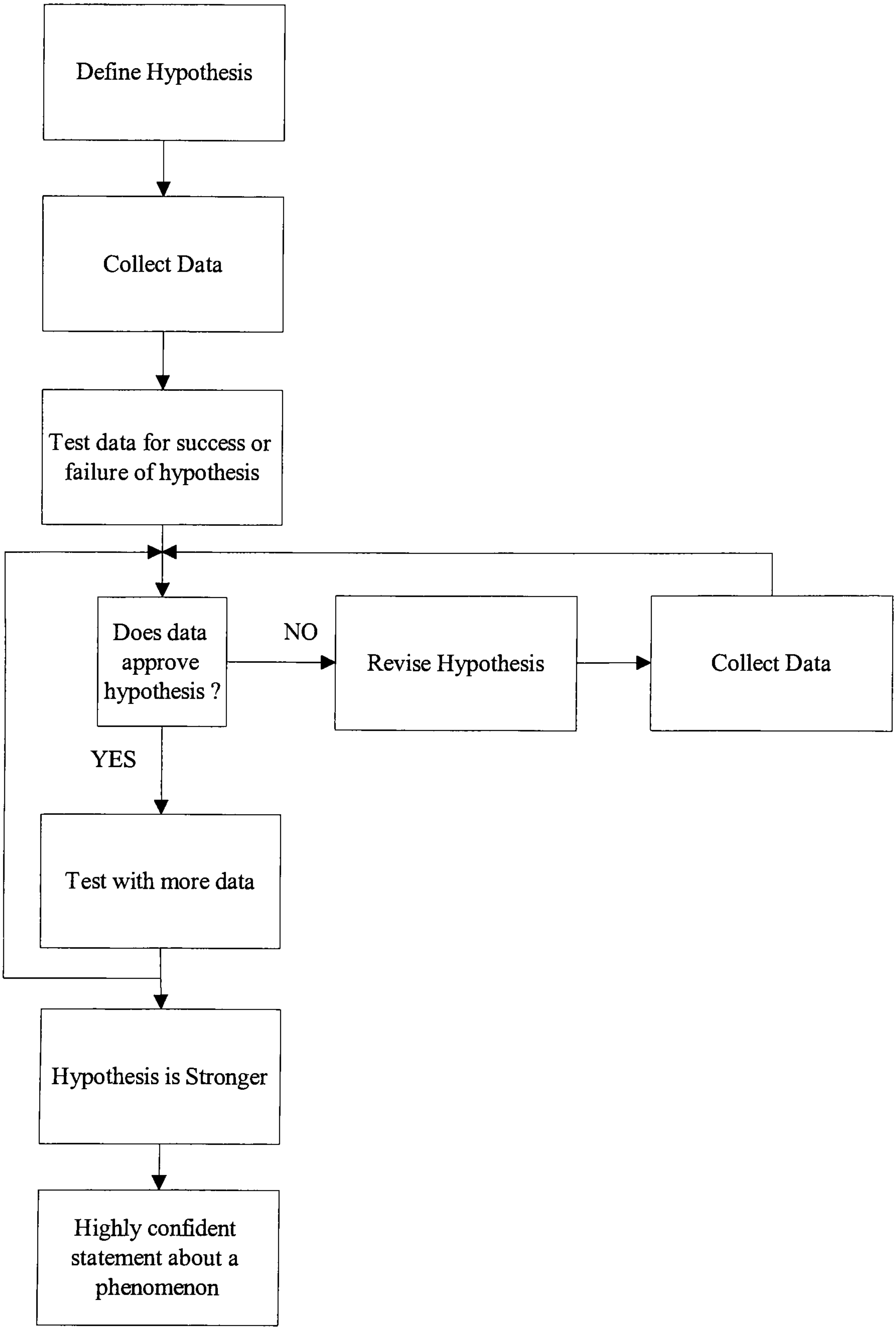
The goal of research is making universal statements that may need to be modified later if exceptions are discovered, but ultimately can reflect fairly exhaustive knowledge of what is researched. This is essentially a process of revising hypotheses with hindsight. As the researcher develops a hypothesis or explanation through an inductive process such as the constant comparative method, additional field data are generated and put to the test of the hypothesis. If the new data confirm the hypothesis, it becomes stronger. But if some aspect of the new data disconfirms the hypothesis, or relates to it ambiguously, the analyst redefines the hypothesis to accommodate the data.

The analyst keeps considering new data, and revising the hypothesis, until there are no more negative cases to account for. Ultimately, negative case analysis results in a highly confident statement about a phenomenon, one that even approaches universal coverage.

To summarize the above discussion, Analytic Induction Approach to Qualitative Research involves the following:

- Define a Hypothesis
- Collect additional data to test fit of the hypothesis
- Redefine questions or reformulate hypothesis based on further data collection and analysis
- Search for negative case to disprove hypothesis

The approach to Negative Case Analysis (Graphical Representation)



Hypotheses

The sub problems defined earlier lead to the following hypotheses which are to be tested one after the other.

The failure of one hypothesis leads to the next sub problem and further research has to be carried out to test the second hypothesis until there is a confirmation to the hypothesis. However, if the first hypothesis is a success or there is a step taken to correct the problem which confirms the acceptance of the first hypothesis, further research is carried out to make the arguments stronger and ultimately to result in a more efficient and refined approach.

1st Hypothesis (Null) : There is no significant difference between the actual and specified dimensions

2nd Hypothesis (Null) : There is no significant moisture absorption by the Dessicant

3rd Hypothesis (Null) : There are no limitations in the Packaging Line Environment and the Capping Process.

The Package

The container for the ready to drink mix was an HDPE blow molded bottle with an injection molded PP cap.

Previous studies have shown that injection molded containers tend to be dimensionally accurate and there is no significant variation. The injection molding process is reliable and maintains dimensions as per the desired tolerances.

A few caps were measured as per the drawing dimensions and were found to be stable and within the tolerance limits.

However, on the other hand the blow molding process is challenged by several factors that greatly affect the quality in bottle blow molding process; important among them being the presence of condensation during molding. The condensation is caused by high humidity and is the number one concern among bottle blow molders.

Lewis Ferguson (Chairman, Blow Molding Division, SPE) in his book 'Blow Molding Design Guide' has laid down several rules for extrusion blow molding of HDPE and one rule states that abrupt changes in the cross-section and profile should be avoided. Also, it is mentioned that HDPE containers shrink slowly and change size for many hours after molding. In designs where dimensional stability is of importance the mold has to be rotated and volume control inserts have to be employed to displace the difference in size. HDPE is more prone to heat warping.

Testing of the first Hypothesis

Dimensions of a sample size of 32 bottles showed serious dimensional instability, especially in the areas supposed to impart the required hermetic seal and moisture resistance. The table no. 1 shows the observed dimensions.

Factors responsible for the inaccuracy were due to :

1) An improper mouth or opening of the container - the opening was unsupported and prone to warping after molding resulting in an oblong opening. On capping there was room for moisture ingress and this contributed the most to the problem.
2) Abrupt changes in the cross-section - technically this was undesirable.

TABLE 1 : Critical Dimensions of the Containers
(Shaded portions indicate that dimensions are out of tolerance limits)

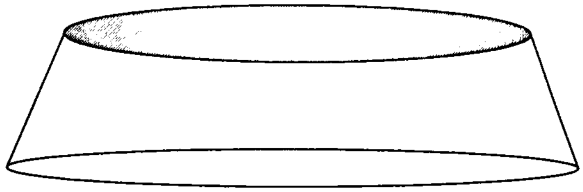
	T Dimension			E Dimension			Mouth O.D.			Mouth I.D.			Wall Thickness	
Cont. No.	3.508"(+/-0.01")			3.368"(+/-0.01")			2.76"(+/-0.01")			2.67"(+/-0.01")			0.045"+/-0.01"	0.791"
1	3.519	3.507	3.48	3.339	3.315	3.328	2.795	2.72	2.762	2.69	2.7	2.65	0.044	0.82
2	3.484	3.499	3.47	3.319	3.348	3.338	2.752	2.752	2.774	2.731	2.67	2.642	0.034	0.791
3	3.482	3.465	3.478	3.364	3.337	3.3	2.755	2.772	2.74	2.738	2.658	2.69	0.038	0.796
4	3.466	3.484	3.476	3.332	3.345	3.328	2.76	2.772	2.74	2.73	2.668	2.66	0.038	0.796
5	3.468	3.47	3.48	3.33	3.36	3.3	2.77	2.79	2.762	2.658	2.672	2.69	0.038	0.805
6	3.52	3.5	3.5	3.37	3.326	3.343	2.752	2.79	2.745	2.7	2.69	2.668	0.036	0.815
7	3.522	3.478	3.477	3.395	3.38	3.346	2.8	2.712	2.71	2.65	2.7	2.67	0.039	0.796
8	3.48	3.462	3.48	3.372	3.368	3.32	2.79	2.72	2.77	2.72	2.65	2.66	0.042	0.792
9	3.48	3.45	3.476	3.354	3.338	3.328	2.79	2.724	2.74	2.7	2.668	2.69	0.038	0.794
10	3.45	3.42	3.478	3.335	3.36	3.338	2.754	2.77	2.765	2.671	2.669	2.66	0.039	0.791
11	3.484	3.516	3.478	3.371	3.342	3.345	2.7	2.76	2.742	2.672	2.65	2.6	0.038	0.805
12	3.484	3.46	3.47	3.364	3.348	3.328	2.79	2.772	2.762	2.66	2.67	2.64	0.036	0.81
13	3.492	3.499	3.476	3.36	3.395	3.336	2.79	2.78	2.776	2.69	2.71	2.66	0.038	0.794
14	3.498	3.486	3.48	3.35	3.35	3.343	2.758	2.76	2.76	2.677	2.68	2.69	0.045	0.8
15	3.508	3.5	3.458	3.352	3.36	3.343	2.74	2.77	2.762	2.69	2.677	2.66	0.036	0.83
16	3.53	3.512	3.448	3.377	3.368	3.328	2.8	2.795	2.715	2.7	2.73	2.69	0.038	0.818
17	3.49	3.5	3.476	3.36	3.37	3.328	2.74	2.76	2.77	2.7	2.66	2.68	0.036	0.81
18	3.472	3.499	3.47	3.342	3.31	3.328	2.775	2.76	2.765	2.69	2.7	2.67	0.034	0.805
19	3.498	3.478	3.48	3.37	3.348	3.343	2.8	2.79	2.742	2.68	2.7	2.69	0.038	0.794
20	3.46	3.47	3.422	3.36	3.38	3.32	2.775	2.77	2.8	2.67	2.68	2.69	0.038	0.795
21	3.48	3.498	3.5	3.354	3.37	3.336	2.77	2.78	2.742	2.69	2.68	2.69	0.039	0.8
22	3.47	3.49	3.462	3.35	3.37	3.348	2.77	2.77	2.76	2.66	2.27	2.666	0.038	0.802
23	3.48	3.5	3.478	3.35	3.342	3.343	2.76	2.762	2.745	2.69	2.7	2.65	0.038	0.815
24	3.492	3.46	3.47	3.336	3.368	3.328	2.75	2.76	2.71	2.69	2.7	2.652	0.038	0.792
25	3.52	3.5	3.48	3.33	3.36	3.338	2.75	2.77	2.745	2.68	2.69	2.68	0.038	0.791
26	3.5	3.48	3.448	3.37	3.35	3.338	2.72	2.74	2.71	2.668	2.69	2.67	0.036	0.796
27	3.501	3.52	3.558	3.38	3.39	3.343	2.78	2.762	2.778	2.66	2.67	2.68	0.037	0.796
28	3.48	3.485	3.476	3.39	3.37	3.343	2.754	2.76	2.742	2.69	2.73	2.652	0.04	0.796
29	3.48	3.476	3.47	3.364	3.366	3.346	2.79	2.8	2.77	2.69	2.63	2.642	0.042	0.805
30	3.468	3.478	3.48	3.348	3.36	3.345	2.77	2.76	2.765	2.678	2.66	2.668	0.038	0.81
31	3.48	3.47	3.438	3.36	3.37	3.338	2.78	2.77	2.762	2.67	2.68	2.66	0.036	0.81
32	3.475	3.48	3.476	3.385	3.39	3.343	2.78	2.775	2.756	2.69	2.67	2.685	0.038	0.796

☐ Under Dimension ☐ Over Dimension

Note :

1) The Measurements were taken with the help of Callipers calibrated in Inches
2) All containers are not perfectly round. They are oblong in shape. Hence diameter obtained at one point may not be the same when dimensions are taken again
3) Wall thicknesses are different
4) Most of the dimensions don't match the specifications
5) The weights were taken on a normal mechanical weighing scale and all containers weigh 0.15lbs
6) The top surface is quite rough on most of the containers

The opening of the mouth is shown below :



Please refer to the Appendix for actual drawing.

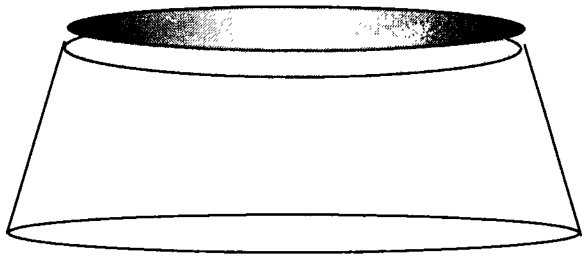
This observation proved that the first sub problem was valid. This negated the first hypothesis. As per the Analytic induction technique two points evolved :

1) Change the design of the opening of the container and test for Hypothesis 1 again
2) Test the existing container for second hypothesis along with the new one to get a comparison of data

Based on this limitation corrective changes were made in the design of the container and a new mold was developed considering the important technical factors and limitations of the blow molding process.

The new container had almost the same design with provision for the same caps to be fit on it. But the opening of the mouth was altered to suit the process and to maintain the tolerances as desired.

The opening of the mouth of the new container is shown below :



Please refer to the Appendix for actual drawing

A sample size of 50 new bottles, all from different molding lots, was studied for dimensional accuracy and the results were extremely good with only one bottle not conforming to the desired specifications. Hypothesis 1 in this case was satisfied. The next hypothesis had to be tested to strengthen the argument.

Table No. 2 lists the important dimensions of the new containers.

Solving the first sub-problem was not sufficient to confirm that the problem of moisture ingress was solved and production runs could begin. The containers were not studied for moisture ingress and the packaging line was also not observed. It was necessary to evaluate the same to prevent any problems in future which could be attributed to those factors and not the containers.

The discussions that follow lead to a testing of the second hypothesis on both the container designs.

Table NO. 2

		E Dimension	T Dimension
Container	2.693 +/- 0.015	3.368 +/- 0.015	3.508 +/- 0.015
1	2.708	3.362	3.5
2	2.694	3.362	3.502
3	2.704	3.37	3.5
4	2.708	3.368	3.494
5	2.708	3.352	3.5
6	2.694	3.37	3.5
7	2.896	3.356	3.502
8	2.708	3.372	3.499
9	2.706	3.371	3.496
10	2.708	3.362	3.494
11	2.703	3.356	3.496
12	2.707	3.368	3.497
13	2.706	3.371	3.5
14	2.705	3.371	3.494
15	2.708	3.357	3.51
16	2.707	3.353	3.5
17	2.7	3.362	3.5
18	2.708	3.362	3.5
19	2.706	3.363	3.5
20	2.7	3.362	3.498
21	2.708	3.37	3.498
22	2.698	3.353	3.5
23	2.698	3.358	3.5
24	2.698	3.37	3.499
25	2.708	3.358	3.493
26	2.71	3.364	3.492
27	2.708	3.371	3.493
28	2.702	3.37	3.492
29	2.703	3.371	3.498
30	2.706	3.352	3.496
31	2.708	3.353	3.498
32	2.708	3.362	3.5
33	2.702	3.364	3.5
34	2.703	3.353	3.51
35	2.702	3.358	3.494
36	2.698	3.378	3.497
37	2.701	3.362	3.494
38	2.707	3.37	3.497
39	2.708	3.362	3.494
40	2.708	3.355	3.497
41	2.706	3.371	3.498
42	2.707	3.372	3.5
43	2.703	3.353	3.494
44	2.703	3.364	3.5
45	2.708	3.353	3.499
46	2.708	3.355	3.5
47	2.693	3.355	3.493
48	2.706	3.38	2.493
49	2.708	3.371	3.5
50	2.708	3.372	3.498

Note :
1) Wall Thickness on each side observed to be 0.5"
2) The 'H' Dimensions were not possible to take accurately. However they are between 1.76 to 1.78 inches and are within the tolerances

Accelerated Aging

The next step involved the tests for moisture ingress using desiccant and without the actual product. The test was an accelerated aging test where the containers were subjected to a temperature of 90^0F and 90% Relative Humidity. The tests were carried out in a humidity chamber. Samples of both the old as well as the new revised designs were subjected to these tests to determine amount of moisture ingress for a specific period of time which would be used to predict the shelf life of the product.

The food industry and medical device industry has been involved in techniques to predict shelf life of polymer based devices. Developers of polymer based food package and medical products need ways to ensure a product life up to five or six years. Real time testing is impractical, thus indicating the need for accelerated aging techniques. The best known technique for predicting the properties using accelerated storage data is the Arrhenius equation.

Accelerated aging can be defined as a procedure that seeks to determine the response of a device or material under normal usage conditions over a relatively long time, by subjecting the product for a much shorter time to stresses that are more severe or more frequently applied than normal environmental or operational stresses (Karl J. Hemmerich).

The Arrhenius equation describes that the rates of chemical reactions, within the limits of certain restrictions, increase with temperature. This equation says that log rate ($\ln K$) is proportional to the inverse absolute temperature ($1/T$) multiplied by the reaction's energy of activation (Ea) divided by R, the universal gas constant :

$\ln K_T = A \, x \, Ea/RT$ or K_T = '$A \, exp \, (E_a/RT)$

The ratios of the rates at two different temperatures yields the Arrhenius shift factor

$A(T) = K_{TH} / K_{TL} = exp\ (E_a / R(1/T_L - 1/T_H)$

where K_{TH} and K_{TL} are the rates at the higher and lower temperatures and T_H and T_L are the higher and lower temperatures in Kelvin.

Accelerated aging is based on a thermodynamic temperature coefficient formulated by Von't Hof that states "for every 10 degree C rise in temperature the rate of chemical reaction will double." For polymers which have been previously characterized, a simplified approach for accelerated aging is based on conducting testing at a single accelerated temperature and then employing this rule. Typically Q_{10}=2 i.e. the reaction rate doubles with every 10^0C rise in temperature above the use or storage temperature (Karl J. Hemmerich).

However, this formula was based on rate kinetics of a single chemical reaction; not to packages with various kinds of materials. So, the direct extrapolation of this theory to the aging of packaging materials is to be used with caution. But the industry and the FDA believe the theory is useful in defining and justifying accelerated aging test programs.

General Test Protocol Guidelines

A number of miscellaneous factors must be considered in conducting an accelerated-aging study, regardless of the accelerated-aging protocol employed (General Aging Theory and Simplified Protocol for Accelerated Aging of Medical Devices – Karl J. Hemmerich www.devicelink.com/mpb/archive/98/07/002.html)

- When establishing the accelerated-aging protocol, the environmental conditions selected should not represent unrealistic failure conditions that would never occur under real-time, ambient-aged conditions. For example, where there is evidence that an aging effect occurs only in the presence of heat, one should perform aging under conditions of storage or use only.

- Proof testing can serve as a substitute for destructive testing. Proof testing requires all samples to be tested at the end of each test interval to a specified failure point, then returned to the aging environment for continued exposure. This method, however, is only applicable when the selection of proof-test values does not weaken or compromise the product properties being examined.

- When use of any accelerated-testing model produces a nonlinear plot, this may be an indication that multiple chemical reactions, complex reactions of a second or third order, or autocatalytic reactions are occurring at some, but not all, test temperatures. In these cases, elevated temperatures may negatively distort the performance of the material at operating or storage conditions. Under such circumstances, one should consider performing aging under conditions of storage or use (ambient).

- When possible, *accelerated testing* should be employed—testing of the device or material at high stress for a short period of time in order to deduce the dominant failure mode. Based on knowledge of the principal degradation mechanisms and stresses on or within the part, a significant enhancement in test-plan efficiency can be achieved through the use of excessive environmental stresses such as heat, oxygen, chemicals, or radiation. Often, radiation is the best stressor to use, since the degradation pathways are often similar to those induced by heat or oxygen. Irradiating a product at 100 kGy (10 Mrd) or more before initiating a formal test program can often root out the product's "Achilles' heel" and allow for improved targeted test design.

- All test units should consist of products constructed of the same components and subassemblies and manufactured by the same processes, methods, and procedures as those used for routine production.

Test Setup

The old and new containers were taken to the package filling facility. A measured amount of desiccant was filled in the bottles prior to capping. 24 bottles of the new design were capped on the packaging line with a capping torque of 25-30 lbs. 21 bottles of the old design were also capped on the packaging line. Another 24 containers of the new design were hand capped.

The desiccant was blue in color and had a property of turning pink on moisture absorption. This desiccant was selected so that it could give a visual indication initially.

Several limitations were observed at the packaging line which would be discussed later while discussing the third hypothesis.

After completion of this, the containers were weighed individually on an electronic weighing scale, which gave an accuracy of up to 2 decimal places. The containers were marked with the weight before the accelerated aging test and numbered for identification.

The containers were placed in three different corrugated cartons, a) The first one with the new containers which were machine torqued b) The second one with the new containers hand torqued and c) The third one with the old containers machine torqued.

The three cartons were then placed in a humidity chamber at 90^0F and 90% relative humidity for 7 days. The containers were tested once on the third day to observe any increase in weight and to note the readings to enable to determine rate of increase per day for any future study related to this product.

Humidity chambers are used to simulate a wide range of environmental conditions or to maintain a consistent environmental condition. Desiccant dehumidifiers are used for controlled pull-down of humidity or for maintaining a constant humidity for long periods of product testing and research or storage.

Results

Table No. 3 shows a detailed observation of increase in weights and time of tests etc. The following points were observed :

- Just 5 containers from the old lot maintained the weight they had initially i.e. did not absorb any moisture, while the remaining 16 containers showed a considerable increase in weight as can be seen from the table. A careful observation of the increase in weights shows that the rate of increase was between 0.02% to 0.08% in one week which was very high.

- The new containers which were machine torqued were highly consistent with their weights and there was no increase in the weight except for one which had an increase by 0.015% which was negligible.

- The new containers which were hand torqued did not show a consistent result with about 25% showing an increase in weight from 0.023% to 0.055% which was high.

- The graphs that follow show a visual adaptation of the moisture gain. Moisture gain follows a linear relationship and in most cases the slope is the same. An estimate of moisture gain after a period of 2 months can be made by following the line graph as the increase in moisture is going to be linear. Most charts for the old container show an initial constant weight and a sudden slope toward an increase of weight. Graphs for old containers 11 to 14 show a constant increase in weight and are terribly out of dimensional tolerances also.

- There are charts showing some variation observed on the new containers also.

Table No. 3

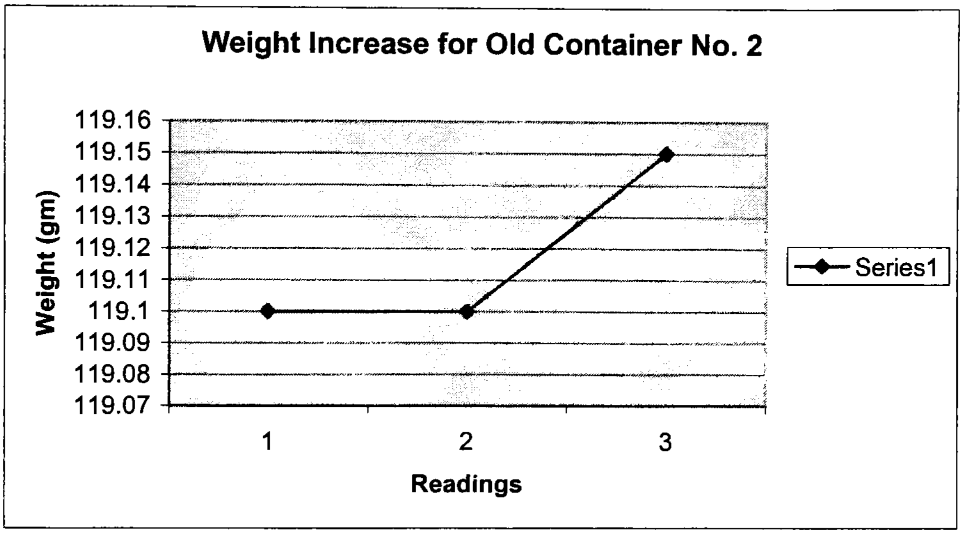
No.	New (Machine Torqued)			% Inc	No.	New (Hand Torqued)			% Inc	No.	Old (Machine Torqued)			% Inc
	3/23/01	3/26/01	3/30/01			3/23/01	3/26/01	3/30/01			3/23/01	3/26/01	3/30/01	
1	139.22	139.22	139.22	0	25	120.47	120.47	120.47	0	1	125.82	125.82	125.82	0
2	130.16	130.16	130.16	0	26	107.7	107.7	107.75	0.046	2	119.1	119.1	119.15	0.04
3	134.39	134.39	134.39	0	27	123.48	123.48	123.48	0	3	120.75	120.75	120.8	0.041
4	140.59	140.59	140.59	0	28	142.19	142.19	142.19	0	4	125.84	125.84	125.88	0.032
5	112.44	112.44	112.44		29	120.78	120.78	120.81	0.025	5	118.9	118.92	118.92	0.017
6	126.29	126.29	126.29	0	30	126.98	126.98	126.98	0	6	103.76	103.76	103.76	0
7	133.59	133.59	133.59	0	31	106.82	106.82	106.82	0	7	126.86	126.86	126.86	0
8	136.26	136.26	136.26	0	32	126.1	126.1	126.1	0	8	121.25	121.25	121.31	0.05
9	126.79	126.79	126.79	0	33	112.55	112.55	112.55	0	9	126.72	126.72	126.72	0
10	129.8	129.8	129.8	0	34	113.82	113.82	113.82	0	10	123.93	123.93	123.96	0.025
11	131.32	131.32	131.32	0	35	136.99	136.99	136.99	0	11	110.4	110.42	110.48	0.0725
12	134.85	134.85	134.87	0.015	36	124.37	124.37	124.37	0	12	114.5	114.52	114.57	0.06
13	122.85	122.85	122.85	0	37	107.72	107.72	107.72	0	13	122.57	122.59	122.61	0.0325
14	122.24	122.24	122.24	0	38	115.35	115.35	115.35	0	14	124.23	124.26	124.3	0.055
15	127.87	127.87	127.87	0	39	126.12	126.12	126.12	0	15	119.19	119.19	119.22	0.025
16	112.13	112.13	112.13	0	40	108.39	108.39	108.42	0.028	16	122.9	122.9	122.94	0.0325
17	122.63	122.63	122.63	0	41	109.82	109.62	109.88	0.055	17	126.53	126.53	126.53	0
18	135.17	135.17	135.17	0	42	123.37	123.37	123.41	0.0325	18	130.29	130.32	130.35	0.04
19	118.54	118.54	118.54	0	43	123.59	123.59	123.59	0	19	127.13	127.13	127.13	0
20	127.01	127.01	127.01	0	44	130.06	130.06	130.09	0.023	20	132.32	132.35	132.38	0.045
21	131.23	131.23	131.23	0	45	112.41	112.41	112.41	0	21	118.87	118.89	118.95	0.07
22	134.4	134.4	134.4	0	46	104.87	104.87	104.87	0					
23	129.78	129.78	129.78	0	47	119.49	119.49	119.49	0					
24	140.08	140.08	140.08	0	48	107.92	107.92	107.92	0					

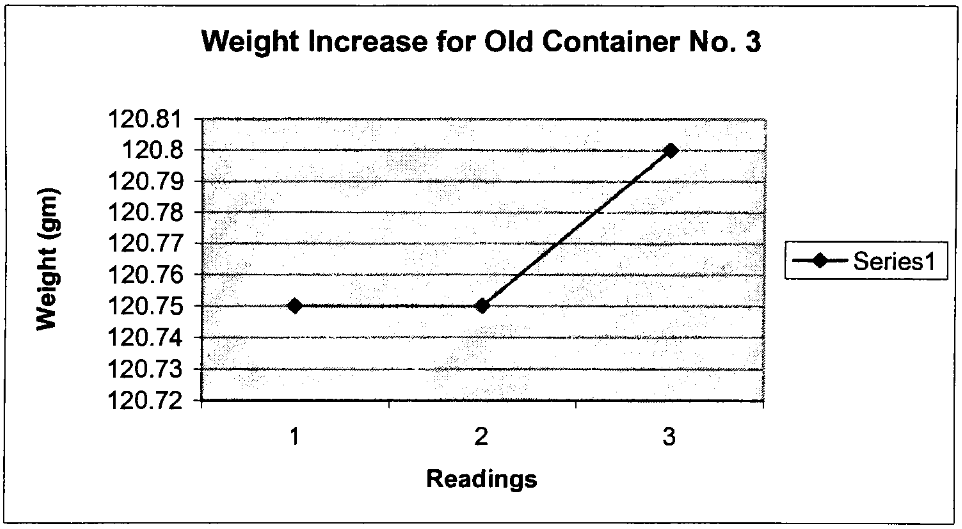
Weight Increase Observed

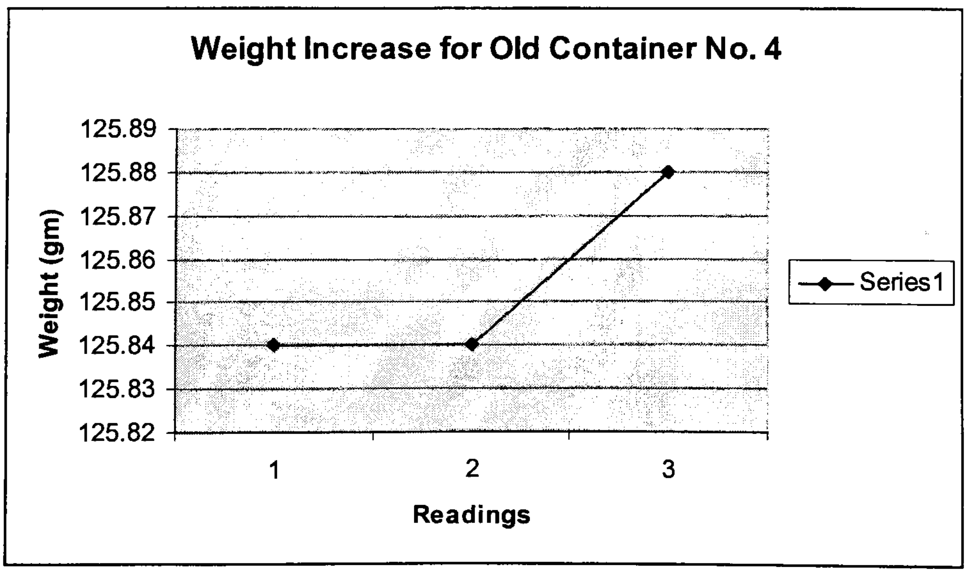
Note :

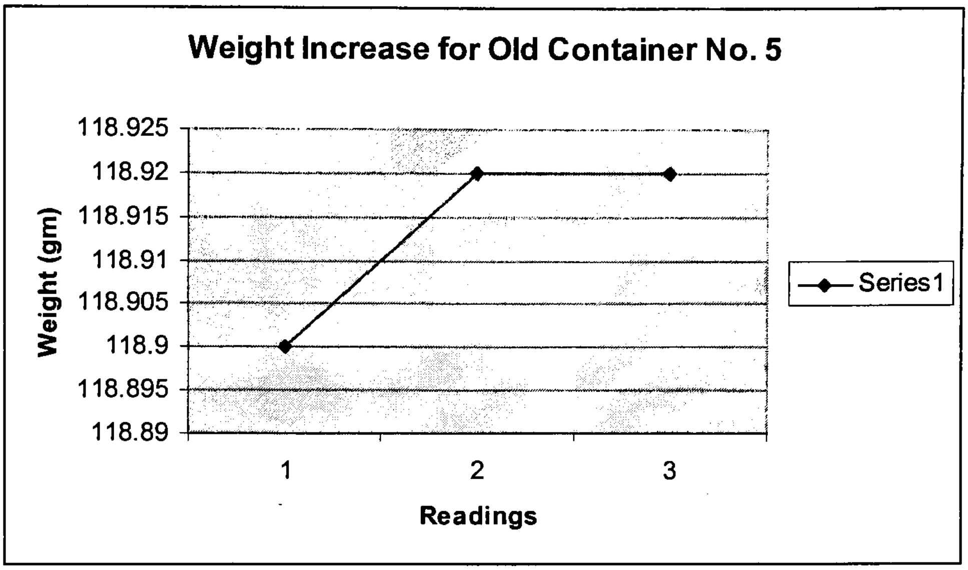
1) All weights are in grams
2) First reading is initial reading before placing the containers in the humidity chamber
3) Humidity chamber at 90% Relative Humidity and 90 degrees F
4) a) Containers placed in chamber at 11.05am on 23rd March 2001
   b) Containers removed at 11.05am on 26th March 2001 for first weight reading, in at 11.40
   c) Containers removed at 11.10am on 30th March 2001 for final weight reading
5) Caps machine torqued at 25-30 lbs (Observed to be a sufficient torque)
6) Hand Torqued containers had a torque range of 13 lbs to 28lbs

Weight Increase for Old Containers which were Machine Torqued

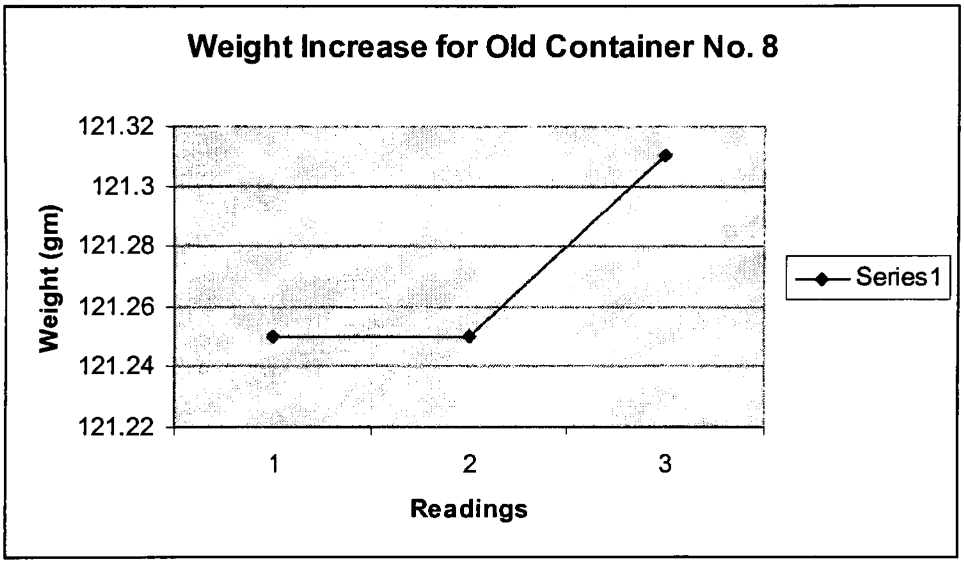




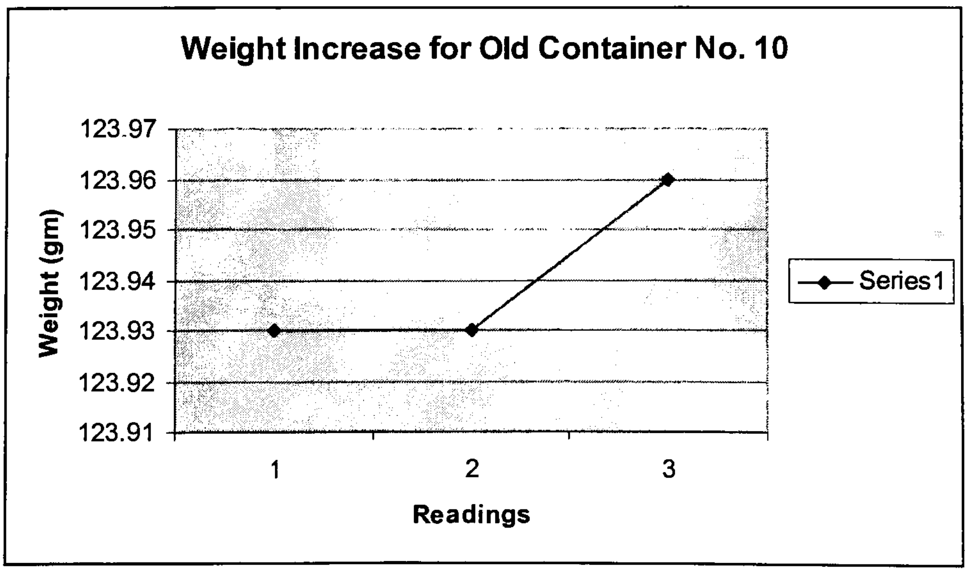




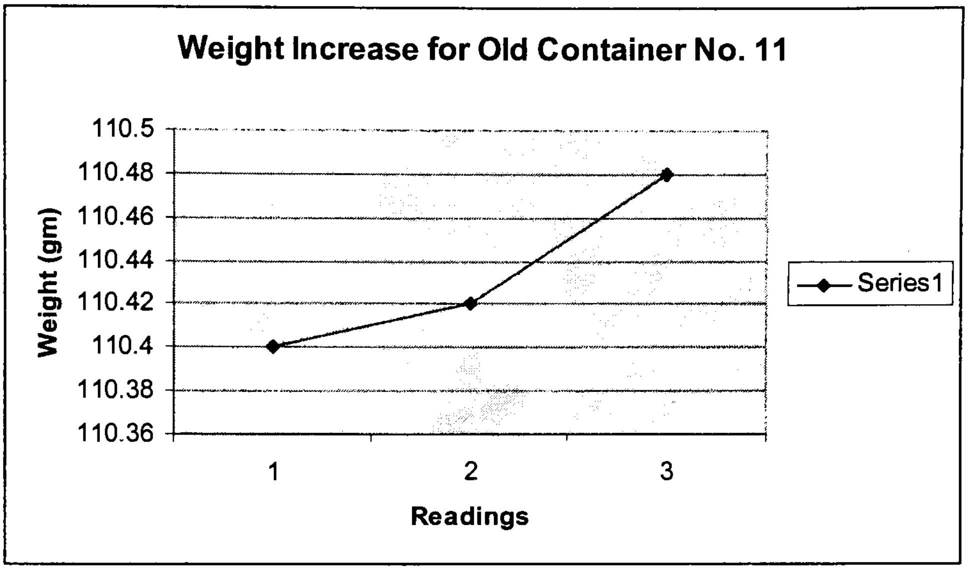
Weight Increase for Old Container No. 5
118.925
118.92
118.915
118.91
118.905
118.9
118.895
118.89
Weight (gm)
1
2
3
Readings
Series1

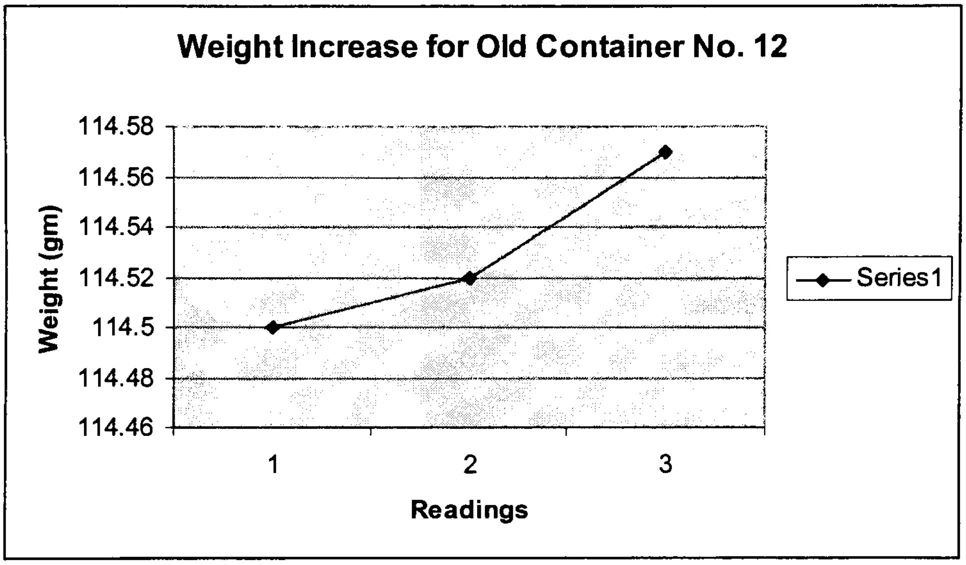


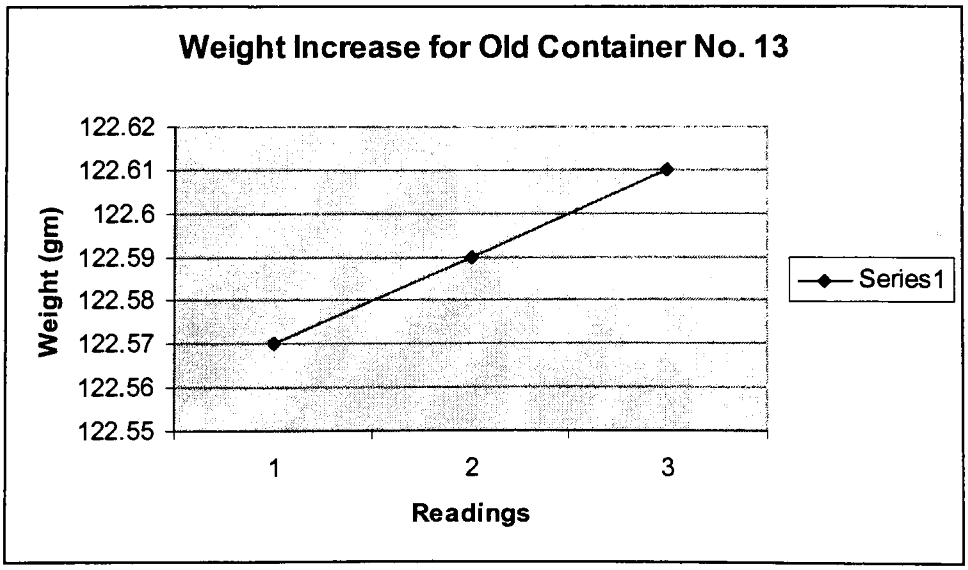
Weight Increase for Old Container No. 8
121.32
121.3
121.28
121.26
121.24
121.22
Weight (gm)
1
2
3
Readings
Series1

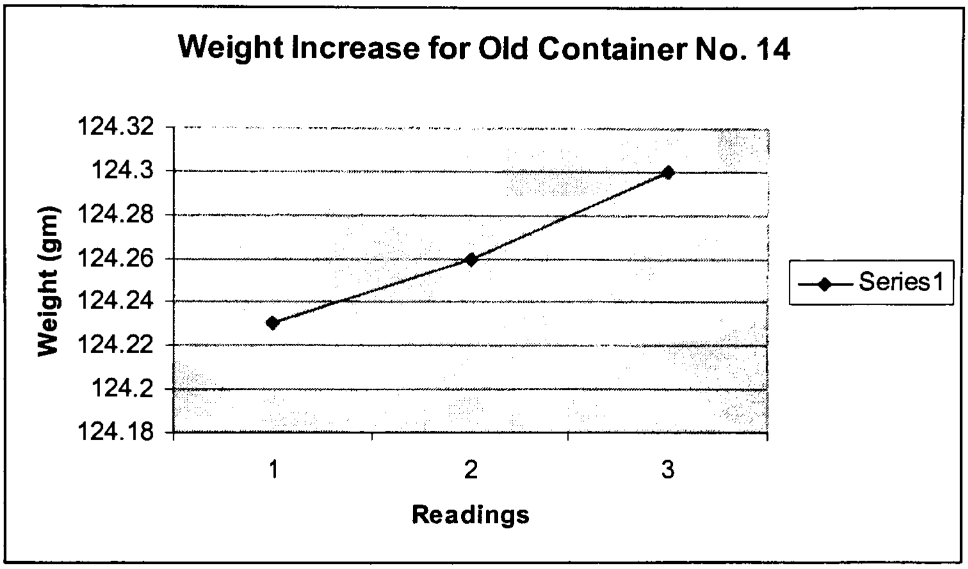


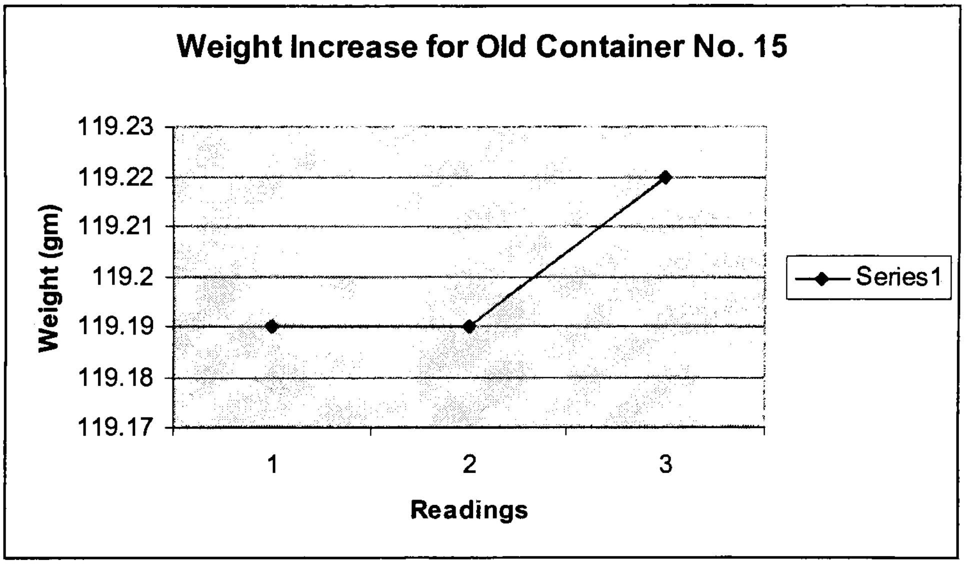
Weight Increase for Old Container No. 10
123.97
123.96
123.95
123.94
123.93
123.92
123.91
Weight (gm)
1
2
3
Readings
Series1

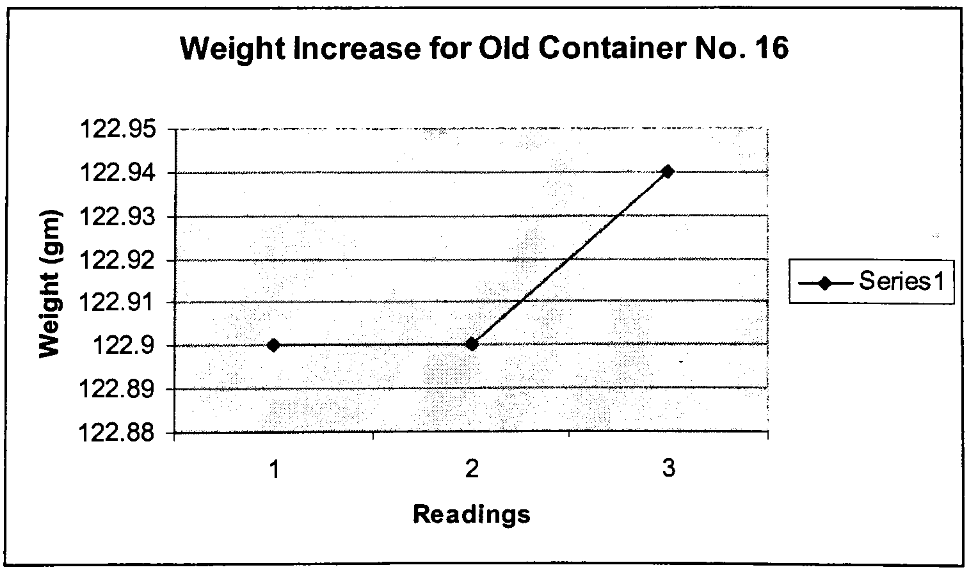

Weight Increase for Old Container No. 11
Weight (gm)
110.5
110.48
110.46
110.44
110.42
110.4
110.38
110.36
1
2
3
Readings
Series1


Weight Increase for Old Container No. 12
Weight (gm)
114.58
114.56
114.54
114.52
114.5
114.48
114.46
1
2
3
Readings
Series1

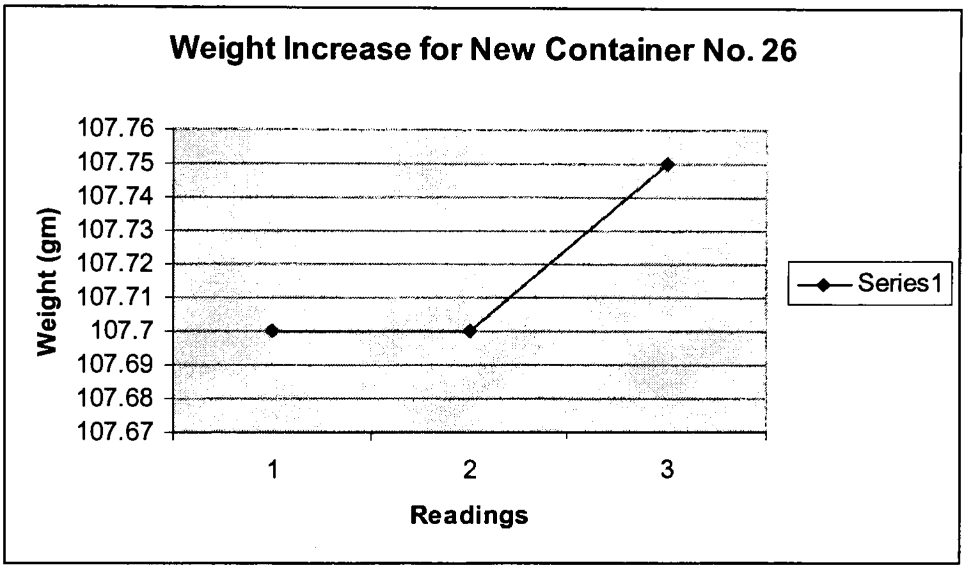

Weight Increase for Old Container No. 13
Weight (gm)
122.62
122.61
122.6
122.59
122.58
122.57
122.56
122.55
1
2
3
Readings
Series1

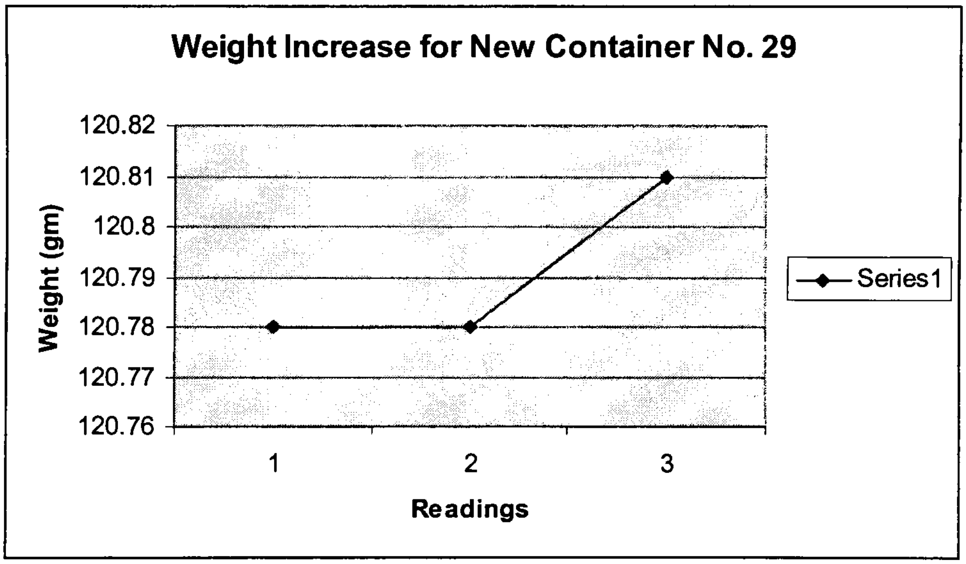

Weight Increase for Old Container No. 14
124.32
124.3
124.28
124.26
124.24
124.22
124.2
124.18
Weight (gm)
1
2
3
Readings
Series1

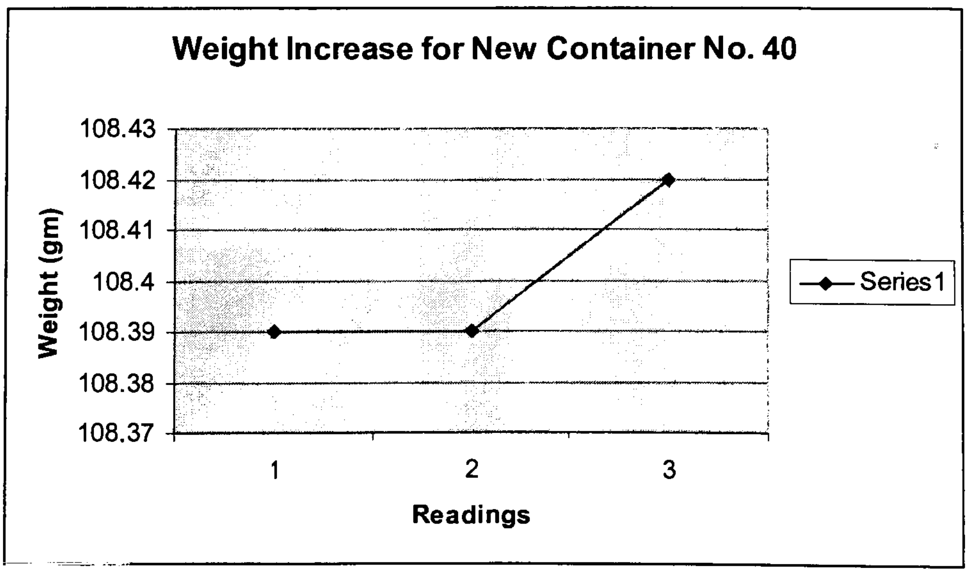

Weight Increase for Old Container No. 15
119.23
119.22
119.21
119.2
119.19
119.18
119.17
Weight (gm)
1
2
3
Readings
Series1

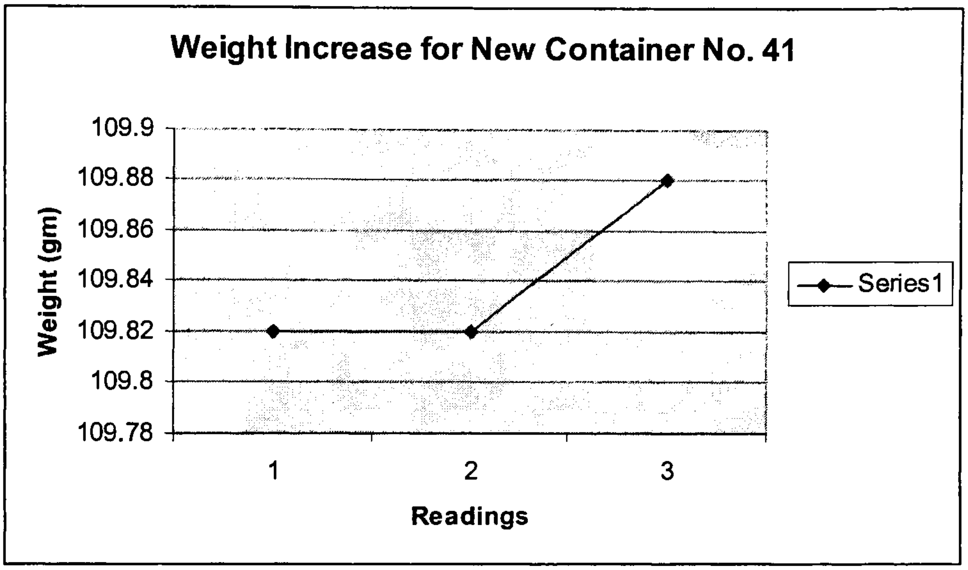

Weight Increase for Old Container No. 16
122.95
122.94
122.93
122.92
122.91
122.9
122.89
122.88
Weight (gm)
1
2
3
Readings
Series1

Weight Increase for New Containers which were Hand Torqued

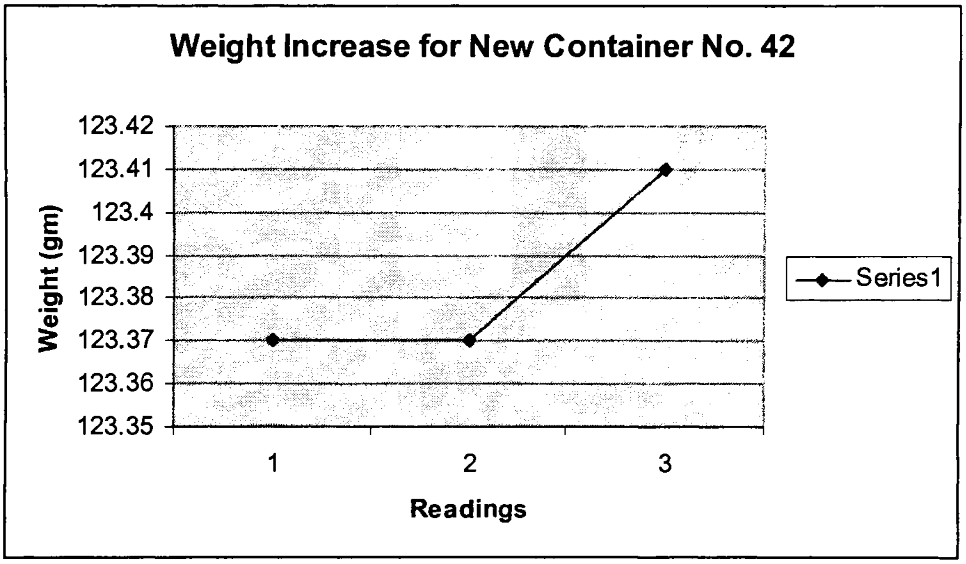




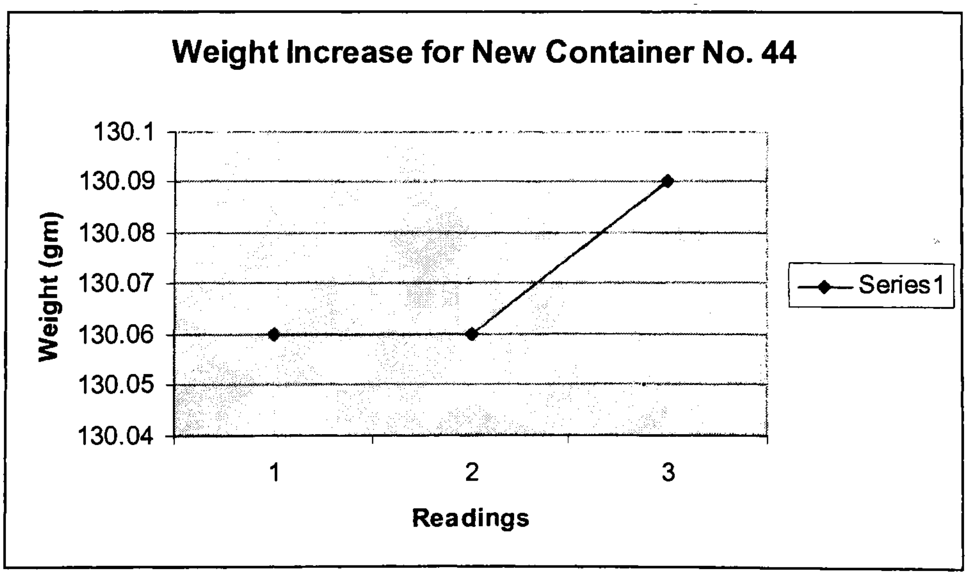




Weight Increase for New Container No. 41
109.9
109.88
109.86
109.84
109.82
109.8
109.78
Weight (gm)
1
2
3
Readings
Series1

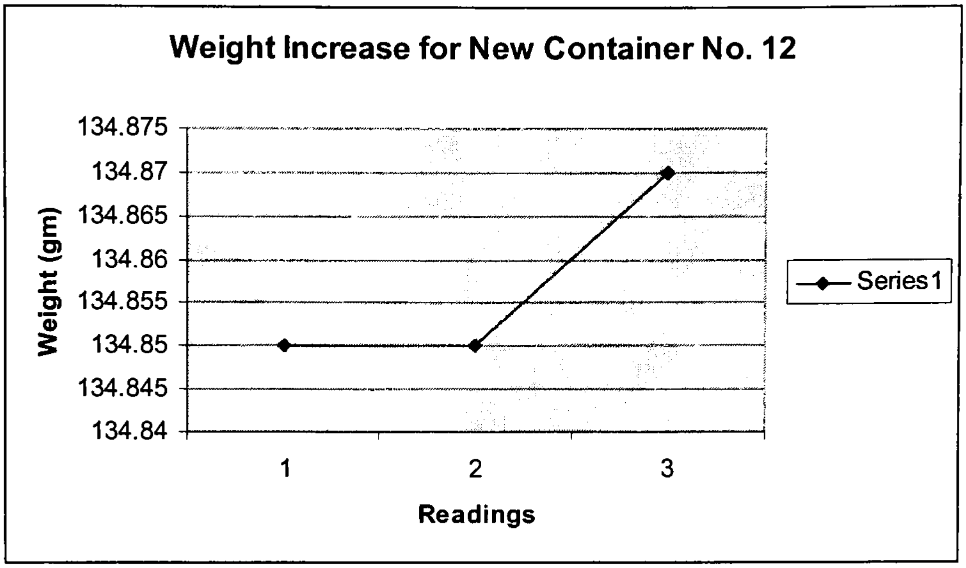


Weight Increase for New Container No. 42
123.42
123.41
123.4
123.39
123.38
123.37
123.36
123.35
Weight (gm)
1
2
3
Readings
Series1



Weight Increase for New Container No. 44
130.1
130.09
130.08
130.07
130.06
130.05
130.04
Weight (gm)
1
2
3
Readings
Series1

Weight Increase for New Container which was Machine Torqued



- Moisture absorption in some cases starts right in the beginning and then stops while in some cases it doesn't start initially but starts later. There is no uniformity observed in this phenomenon. This can be attributed to the following :
  a) Initial absorption might be due to presence of moisture in the inner environment and after a certain point of time it might be reaching a saturation point to stop further absorption
  b) Late start of absorption might be due to steady ingress after a point of time and further ingress and absorption.
  c) Self release of caps over a certain period could also be a reason for late start of moisture ingress.

Test of hypothesis 2 for the new containers was a success.
However, the test was negative for the old containers.

Another variable introduced during the tests, which hadn't been considered in the sub problems as well as the definition of hypotheses was the importance of the capping torque. The tabulated readings proved that a uniform machine torque on the new containers was more efficient than the hand torque on the new containers. This proved the importance of a specific capping torque necessary to prevent moisture ingress.

Conclusion

The failure of the first two hypotheses on the old containers proved that the approach taken during this research was correct and recommendation for the new design was appropriate in solving the problem of moisture ingress.

The success of the first two hypotheses on the new containers solved the first two sub problems as far as the container limitations were concerned. This also solved the problem of moisture ingress and prevented any caking of the mixes at the retail shelf.

It is also safe to conclude that on the basis of visual data as depicted from the graphs, the moisture ingress over a certain period of time can be estimated to determine the shelf life of the product. This is because there is a linear relationship followed. Literature has lot of information pertaining to the linear increase of moisture ingress, however it has been proved by the tests on a practical platform.

Some important observations at the packaging line necessitated the need to test the third hypothesis also. This would result in a long term solution for the problem of moisture ingress.

The Packaging Line Environment

In the Analytic Induction process of statistical analysis in this research the third hypothesis pertaining to the packaging line environment had to be tested. However, a detailed study on this was beyond the scope of this project

Observations at the Packaging Line

- It took typically 75 seconds for a bottle to travel on a conveyor from the filling line to the capping line. However, a delay or stoppage of conveyor increased this time to 120 seconds also.
- Desiccant left open at the packaging line turned pink in color in a duration of 120 seconds indicating presence of moisture in the environment.
- A change in color after 120 seconds implied that absorption of moisture started earlier. There must have been a certain percentage of moisture absorption in the drink mixes between the filling and capping lines.

There was no test carried out at the packaging line to estimate the percentage of absorption of moisture and time required. Also, it was very difficult to determine whether the moisture absorbed by the product in the packaging line contributed to caking of the product in due course.

Considering that the product is a class of dry products and tends to absorb moisture readily several precautions have to be taken at the packaging line.

To prevent any moisture absorption at the packaging line, hygroscopic products such as powders have to be processed in humidity controlled environments.

The pharmaceutical industry produces millions and millions of pills, tablets, capsules each year. This involves the processing of hygroscopic powders also.

Dehumidification is used to prevent moisture regain for powder processing, pneumatic conveying of bulk materials etc.

FDA guidelines and regulations have the impact of law concerning the manufacturing, package and storage conditions for pharmaceutical products. This data can be used to improve the efficiency of the packaging line for this product. Part 212 of the CFR cites a temperature of 72^0F (+/- 3^0F) and a relative humidity of 30-50% for manufacturing areas. Since personnel are gowned, the room temperature is often specified as 68^0F. Make-up air is required to replace exhausted air and is usually the largest load factor. It is recommended that make up air be dehumidified before mixing with return air.

Recommendations for further study

- Test the packaging line environment to acquire data to carry out an analysis and to test for other inefficiencies
- Study the new containers for a longer shelf life simulation to generate data for a quantitative statistical analysis
- Study the importance of capping torque on containers meant to prevent moisture ingress or provide a moisture barrier. Study the rate of self release of caps from the container and its contribution to moisture ingress after a certain period.

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