



119
058
THS

THREE



3 1293 10063 3894



This is to certify that the

thesis entitled

PREDICTION OF MOISTURE CONTENT OF PACKAGED
DRY FOOD PRODUCTS BY A CALCULATION
BASED ON SIMULATION

presented by

RANDY A. KLIMENT

has been accepted towards fulfillment
of the requirements for

M.S. degree in School of Packaging

Steven W. Giesly

Major professor

Date August 15, 1979



OVERDUE FINES ARE 25¢ PER DAY
PER ITEM

Return to book drop to remove
this checkout from your record.

MSU

X-17078

7-2-07
M-1-09

ADN 15-11-1

PREDICTION OF MOISTURE CONTENT OF PACKAGED
DRY FOOD PRODUCTS BY A CALCULATION
BASED ON SIMULATION

By

Randy A. Kliment

A THESIS

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

MASTER OF SCIENCE

School of Packaging

1979

ABSTRACT

PREDICTION OF MOISTURE CONTENT OF PACKAGED DRY FOOD PRODUCTS BY A CALCULATION BASED ON SIMULATION

By

Randy A. Kliment

The storage life of packaged dry food products may depend on a number of factors but one of the most important is the adsorption of moisture to some critical level. In real life the temperature and relative humidity are changing. The purpose of this study was to set up conditions where the temperature and relative humidity are constant during any storage interval but changed from interval to interval and predict the moisture content during these intervals. Actual experimental testing was conducted on three commercially available packaged dry food products to determine the moisture content during short storage intervals. Harsher condition changes were used between intervals than expected in real life. Each packaged product was subjected to at least one cycle, defined as subjecting the samples to conditions of low temperature-low relative humidity, transferring through storage intervals to conditions of high temperature-high humidity, and back down

Randy A. Kliment

again to low temperature-low humidity. A calculation based on simulation was then used to predict the moisture content of the three products during short storage intervals corresponding to the same conditions and time periods as used for the experimental tests. The calculated results were in good agreement with the actual experimental results.

Because of the extreme conditions the packaged products were subjected to, it is expected that this method can be used to calculate moisture content increase during actual field storage.

Dedication

This thesis is dedicated to my family, especially my parents, in gratitude for their encouragement and assistance during this and all other endeavors.

Also to Mr. John Barnes whose influence on personal development and career selection will always be remembered.

ACKNOWLEDGEMENTS

The author wishes to thank Dr. Steven Gyeszly for his considerable time and effort, encouragement, and guidance while serving as major adviser. Thanks are also due to Dr. Hugh Lockhart and Dr. Mark Ubersax for their criticisms and suggestions as members of the thesis committee.

Also, a special note of appreciation is extended to General Foods Corporation and in particular Mr. Harry Topalian. Without his personal involvement and commitment this thesis might never have been completed.

TABLE OF CONTENTS

	Page
LIST OF TABLES	v
LIST OF FIGURES.	vi
INTRODUCTION.	1
LITERATURE REVIEW	4
DISCUSSION OF CALCULATION BASED ON SIMULATION.	9
EXPERIMENTAL METHODS	18
A. Data Generation For Calculation	18
Initial moisture content.	19
Sorption isotherms.	20
Water vapor transmission rate	22
Surface area of the pouch	23
Fill weight of the package	23
Headspace volume of the package	23
B. Experimental Testing	24
Initial moisture content.	24
Test conditions of actual experimental testing	25
Calculation of experimental moisture content	31
CALCULATION	36
Calculation of Permeability Constant from WVTR.	36
Calculation of Slope and Intercept.	37
Determination of Moisture Content by Calculation Based on Simulation	37
RESULTS AND DISCUSSION	44
Experimental Error of Actual Testing	50
Experimental Error of Calculation Based on Simulation	51
SUMMARY	56
REFERENCES	58

LIST OF TABLES

Table	Page
1. Initial moisture content	26
2. Pouch surface area, headspace volume, and package fill weight.	26
3. Water vapor transmission rates	26
4. Test conditions, salt solutions, and equilibri- um moisture content results for sorption isotherms	27
5. Experimental test conditions; results of experi- mental and calculated moisture contents-- Product A	32
6. Experimental test conditions; results of experi- mental and calculated moisture contents-- Product B	33
7. Experimental test conditions; results of experi- mental and calculated moisture contents-- Product C	34
8. Permeability constant as determined from water vapor transmission rates	38
9. Permeability constant at temperatures used in experimental testing	38
10. Slope and intercept from sorption isotherms. .	39
11. Greatest percentage difference between calcu- lated and experimental data	44
12. Overall moisture content increases.	45

LIST OF FIGURES

Figure	Page
1. Adsorption isotherms--Product A	28
2. Adsorption isotherms--Product B	29
3. Adsorption isotherms--Product C	30
4. Permeability constant as a function of temperature--Package A	40
5. Permeability constant as a function of temperature--Package B	40
6. Permeability constant as a function of temperature--Package C	40
7. Experimental and calculated results-- Product A	41
8. Experimental and calculated results-- Product B	42
9. Experimental and calculated results-- Product C	43

INTRODUCTION

Stability of packaged dry food products depends greatly on the protection provided by packaging. The surrounding environment is detrimental to extended storage. The amount of protection provided by the package depends on the package's ability to act as a barrier between the internal and external environment.

The shelf-life of a product is the length of time a packaged product will remain of acceptable and saleable quality when subjected to conditions of the distribution environment. Dehydrated foods can deteriorate through several mechanisms such as lipid oxidation, non-enzymatic browning, enzymatic hydrolysis, degradation of proteins, and caking leading to toughening or insolubility. Stability is often determined by more than one environmental factor. The objective of packaging a dry food product then is to reduce or eliminate the rate of transport of water vapor and/or oxygen through the package wall into the internal environment. This is because the rate of deterioration depends on the conditions of the internal environment.

Typically, industry methods for selecting an appropriate flexible packaging material to insure adequate shelf-life and consumer acceptance have been based on actual

storage tests and experience. Actual long-term field storage is the most direct method of determining shelf-life but is expensive, time consuming, and impractical for immediate introduction of a new product. As an alternative, the food industry uses an accelerated test technique as a common shelf-life determining tool.

This technique uses accelerated testing conditions of temperature and humidity compared to normal distribution storage conditions. Several packaging structures are selected which have historically exhibited adequate protection for "similar" products. From these structures the one that gives the best protection at accelerated conditions is chosen as the packaging material for the product. This method results in wasteful overpackaging and severely impedes development of new packaging concepts.

Therefore a method is needed for calculating the amount of packaging protection required that will greatly reduce or eliminate overpackaging and also be more economical than the accelerated test technique.

The purpose of this thesis is to apply a calculation based on simulation to the determination of moisture content. The calculation has been used previously to predict shelf-life when storage conditions are constant (Manathunya 1976), but is applied here in time-steps in which the testing conditions are constant during any one interval and subsequently changed for succeeding intervals. This method has

application for prediction of shelf-lives of mass produced goods that are dispatched into the real world of consumers where the temperature and relative humidity fluctuate.

In designing a model to simulate fluctuating conditions, drastic changes were used that are more severe than those occurring in the course of a normal day. No attempt has been made to determine actual product shelf-life. The moisture content as a function of time due to changing storage conditions was determined experimentally and compared to calculated results for the same time periods and conditions.

LITERATURE REVIEW

Consumer acceptance of packaged dry food products depends in large measure upon their quality at the time of serving. These products may lose their appeal for a number of reasons, but one of the most important is the loss of consumer acceptance resulting from adsorption of moisture from the atmosphere. Packages that resist water vapor are used to prevent moisture adsorption by the product.

Deteriorative reactions of dry food products that depend on moisture content are of two general types. The first are reactions for which a definite critical moisture content can be established, below which the rate of spoilage is insignificant. Typical spoilage mechanisms which fall into this category are bacteria and mold growth, enzymatic spoilage reactions, recrystallization of sugars, and caking. The second general type of reactions proceed at all moisture contents but the rate of the reaction depends strongly on the moisture content. Many of the reactions which cause changes in texture, flavor, and color proceed in a manner which is not dependent on a critical moisture content.

Storage life prediction for a food-package combination or the prediction of the package protection required for a particular food have important application in food

packaging. The shelf-life of a packaged product in any geographical location can be estimated by actual field storage or by calculation from laboratory measurements of the package and product under known atmospheric conditions.

The most direct method is the actual field storage test because only two assumptions need be made. The first is that the location chosen for the storage test is typical of the larger general area and the second, that weather conditions during the test were normal for the time of year in that area. Careful selection of test locations will minimize errors resulting from the test location not being typical of the geographical area. Most field studies extend for a long period which will tend to lessen the effect of day-to-day weather variations. The storage life in a particular area will be greatly influenced by the time of the year that the products are placed into storage in that area. Thus, the accumulation of shelf-life data by actual field storage tests in all market areas for all seasons becomes a very expensive and time consuming procedure.

To reduce the testing time and the associated high costs of direct field storage, an accelerated test technique was introduced. This technique uses high testing conditions of temperature and humidity compared to normal distribution storage conditions. Easter (1953) described how to predict shelf-life from an accelerated laboratory test technique. Actual testing of the product is conducted under normal and

accelerated conditions until the product is deteriorated or unacceptable at the accelerated conditions.

The method typically involves the selection of several packaging structures which have historically exhibited adequate protection for "similar" products. From these several structures the one that gives the best protection as determined by the accelerated testing conditions is chosen as the packaging material for the product.

This method results in wasteful over-packaging and severely impedes development of new packaging concepts. In addition, the accelerated test technique assumes that reactions that determine shelf-life proceed in the same manner at accelerated conditions as they do at room conditions. An assumption is also made that "similar products will have similar room condition to accelerated condition time ratios. This assumption is not correct in most cases (Manathunya 1976).

A more scientific approach was introduced for the prediction of shelf-life that considered properties of the product, the package, and the internal and external environment. Several studies have been made in the past in which the storage life and/or package protection requirements have been calculated on the basis of certain properties of the food or package.

Oswin (1945) developed a method to predict a product's storage life based on adsorption of water by the food to some critical level of moisture content. Felt et al. (1945) extended this to the storage of cereals. Charie et al. (1963) used the same method for the prediction of shelf-life of several dehydrated foods. Mizrahi et al. (1970a) developed a simple mathematical model for predicting moisture content change and extent of nonenzymatic browning of stored dehydrated foods. This method could be applied to determination of the packaging material to be used for a desired shelf-life. Labuza et al. (1972) extended this work to additional food systems. Aguilera et al. (1975) and Davis (1970) have also done similar work for dried potatoes and Harrington (1973) extended it to storage of seed. Labuza (1968, 1971) has reviewed the area of maximum amount of moisture in a food in terms of stability.

Simon et al. (1971) introduced the same concepts to the prediction of the shelf-life of a product which undergoes oxidation. Quast and Karel (1972a,b) extended this further to products which deteriorate through two mechanisms. They studied potato chips which turn soggy from adsorption of moisture and become rancid from oxidation. Mizrahi et al. (1970b) and Karel et al. (1971) developed a mathematical model to study the same effects on dehydrated cabbage.

During the past several years, techniques have been developed to predict the self-life of packaged foods on the

basis of laboratory tests on kinetics of deterioration and on mass transfer properties of packaging materials.

Reviews of this subject have been published recently by Karel (1973, 1975), and Labuza (1972, 1973).

Mizrahi and Karel (1977a) developed a method for accelerated stability tests which does not require prior knowledge of the kinetic model of the effect of moisture on rate of deterioration. This isothermal "no model" method was later extended to include storage at different temperatures by Mizrahi and Karel (1977b).

More recently, Chirife and Iglesias (1978) and Boquet et al. (1978) have reviewed the major equations for fitting water sorption isotherms of foods. Resnik and Chirife (1979) studied the effect of moisture content and temperature on some aspects of nonenzymatic browning in dehydrated apple.

The scope of this research is concerned with predicting the moisture content of packaged dry food products at storage intervals by a calculation based on simulation. The storage conditions of temperature and humidity change from interval to interval but are constant during any particular interval.

DISCUSSION OF CALCULATION BASED ON SIMULATION

The moisture content--water activity of a food can be used to predict the storage stability of a food. The basis of this has been reviewed by Labuza et al. (1970) and Labuza (1971) from the standpoint of the solvent properties of water and the degree to which it is bound in food.

The control of water content of a food is a basic food processing technique based on reducing the water content to a point that will prevent microbial growth. This can be accomplished by drying or freezing in which the water is made unavailable. Other methods involve binding the water in a food by salting, sugaring, or by use of chemical agents as used for intermediate moisture foods.

By eliminating the possibility of microbial growth, the food stability depends on chemical reactions in the food. The rates of these reactions can be predicted as a function of the moisture content of the food. Very few reactions can proceed below the monolayer moisture content value which require the solubilization of reactants and an aqueous phase for reaction. Above the monolayer hydrolytic reactions increase with increasing moisture content. Thus, for prevention of these reactions it is best to keep dry

foods as close to the monolayer moisture content as possible (Labuza 1975).

Since rates of reactions and ultimate storage life depend on the moisture content of packaged dry food products, a mathematical model that considers the important aspects of the product, packaging material, and internal and external environments of the package can be used to predict the moisture content and shelf-life. Equations relating the total amount of water in a closed package as functions of the product, package, and environment have been reported widely in food packaging.

During experimental storage the internal conditions of the package are constantly changing in relation to the constant external environment. By using short time-steps and assuming the product in the package is in equilibrium with the internal environment during each time-step, the moisture content change can be simulated. This is referred to as the calculation based on simulation.

By knowing the instantaneous internal and external environmental conditions the change in the weight of the moisture in the package can be calculated for a short time-step. As the package gains or loses water the internal conditions change for the next time-step. Therefore by using short time-steps and assuming equilibrium the constantly changing experimental conditions can be simulated and the moisture content calculated based on this simulation.

The total amount of water in a closed package (M) is the sum of the weight of the water in the product (M_1) and the weight of the water in the headspace (M_2).

$$M = M_1 + M_2 \quad (1)$$

If W is the weight of the dry product in the package and m is the moisture content (g moisture/100g dry product) then $M_1 = m \cdot \frac{W}{100}$. (2)

Assuming equilibrium in the package (the product adsorbs the water which penetrated through the package very rapidly) the moisture content depends on the internal relative humidity at constant temperature: $m = f(H_i)$ where H_i is the internal relative humidity. Therefore a relationship between moisture content and internal relative humidity is needed. This relationship can be described by the sorption isotherm of a food.

The sorption isotherm of a food product is best described as a plot of the amount of water adsorbed or desorbed as a function of the relative humidity or activity of the vapor space surrounding the material. This amount of water is that which is held after equilibrium has been reached at a constant temperature (Labuza, 1968).

There have been numerous mathematical equations reported in the literature for describing water sorption isotherms of food products. Adamson (1960) and Gregg and Sing (1967) have reviewed the theoretical basis of the major

isotherm equations. Labuza (1968) has discussed the use of these equations within the food field. The commonly used equations have been summarized by Labuza (1975) and Iglesias and Chirife (1976a). Additional equations have been suggested by Iglesias and Chirife (1976b) and Caurie et al. (1976).

In this study the sorption isotherms were determined for each product. It was found that within the important range (from initial moisture content to the estimated maximum acceptable moisture content) the curve can be fitted by a straight line with good agreement. Therefore in the case of the three products used in this study the relationship between the equilibrium moisture content and relative humidity can be expressed by:

$$m = a + bH_i \quad (3)$$

where a and b are constant. Using these relationships

Equation (2) becomes $M_1 = (a + bH_i) \frac{W}{100}$ (4)

By using ideal gas law (water vapor is not an ideal gas but the error introduced in this case is negligible) the weight of the water in the headspace can be given by the equation:

$$M_2 = \frac{18V}{RT} P_s \frac{H_i}{100} \quad (5)$$

where

V is the headspace of the package
 T is the temperature
 R is the gas constant

p_s is the saturated water vapor pressure at T temperature
 18 is the molecular weight of water
 M_2 is the weight of the water in the headspace

From Equations (4) and (5) the total amount of water in a closed package at time (t) can be expressed as:

$$M(t) = \left[a + bH_i(t) \right] \frac{W}{100} + \frac{18V}{RT} p_s \frac{H_i(t)}{100} \quad (6)$$

and, the total amount of water in a closed package at time (t + Δt) can be defined by:

$$M(t+\Delta t) = \left[a + bH_i(t+\Delta t) \right] \frac{W}{100} + \frac{18V}{RT} p_s \frac{H_i(t+\Delta t)}{100} \quad (7)$$

The term Δt is defined as the time-step of the calculation.

The permeation of vapors and gases through polymeric packages can be described by Fick's laws of diffusion and Henry's Law of solubility. The basic equation commonly used by many authors for determining the quantity of moisture vapor penetrated across a flexible barrier material during a time period is:

$$\frac{dQ}{dt} = \frac{P}{x} \cdot A \cdot (P_e - P_i) \quad (8)$$

where

$\frac{P}{x}$ is the permeability constant of the film divided by the wall thickness, wt/area · time · Δatm

A is the package surface area

P_e is the external water vapor pressure at a temperature, atm

P_i is the internal water vapor pressure at a temperature, atm

Generally, it is easier to measure the relative humidity than to measure the water vapor pressure. The relation between the water vapor pressure and the relative humidity is given by Equation (9).

$$P = \frac{P_s}{100} \cdot RH \quad (9)$$

Substituting Equation (9) into Equation (8) and solving for Q gives (Manathunya 1976):

$$\Delta Q = \frac{P}{x} \cdot A \cdot \frac{P_s}{100} (H_e - H_i) \Delta t \quad (10)$$

where

H_e is the external relative humidity, %

H_i is the internal relative humidity, %

Δt is the time-step, and Δt must be very small

ΔQ is the weight of moisture penetrated through the container during Δt

The total moisture of the packaged product at any subsequent interval $(t + \Delta t)$ is the sum of the moisture at time (t) plus the moisture penetrated through the container during Δt . This can be expressed as:

$$M(t + \Delta t) = M(t) + \Delta Q \quad (11)$$

By knowing the internal relative humidity of the package at any time the moisture content can be calculated from the sorption isotherm curve and Equation (3). Therefore, the moisture content at time $(t + \Delta t)$ can be calculated by knowing the internal relative humidity at time $(t + \Delta t)$. Substituting Equations (6), (7) into Equation (11)

and solving for the internal relative humidity at time $(t+\Delta t)$ gives equation (12).

$$H(t+\Delta t) = H_i(t) + \frac{\Delta Q}{\frac{W_b}{100} + \frac{18V}{RT} \frac{p_s}{100}} \quad (12)$$

The calculation uses time-steps for determining the moisture content of the packaged product during any interval. An interval is defined as the experimental conditions of constant temperature and humidity that the package was subjected to for a time period. The conditions of each successive interval vary. The internal conditions of the package at the beginning of the first interval can be calculated by rearranging Equation (3) to:

$$H_i(o) = \frac{m_i - a}{b} \quad (13)$$

where

$H_i(o)$ is the initial internal relative humidity
 m_i is the initial moisture content
 a is the intercept of the sorption isotherm
 b is the slope of the sorption isotherm

By knowing the initial internal conditions of the package and the constant external conditions at the first interval, the weight of the moisture penetrated through the package for a time-step (Δt) can be determined by Equation (10) (the external conditions are constant during an interval with the internal conditions changing with time). The internal relative humidity will change as moisture penetrates or leaves the package. The internal relative humidity of

the package at time $(t+\Delta t)$ will depend on the amount of water vapor penetrated through the container wall during the time-step Δt . The internal relative humidity at $(t+\Delta t)$ can be determined from Equation (12). This new internal relative humidity at time $(t+\Delta t)$ will equilibrate with the food in the package and change the moisture content of the product according to Equation (3).

The change in time (Δt) in going from (t) to $(t+\Delta t)$ is defined as the time-step. At the end of each time-step the moisture content of the product will change as a function of the internal relative humidity. As the internal relative humidity changes the partial pressure difference $(P_e - P_i)$ will also change since the external relative humidity is constant. The weight of the moisture penetrated through the package (ΔQ) for the next time-step $(t+2\Delta t)$ will change as a function of this partial pressure difference change.

During experimental storage of the packages the internal conditions are constantly changing in relation to the constant external conditions. By selecting a short Δt for the time-steps and assuming equilibrium during each time-step, the internal environment change can be simulated and the moisture content calculated based on this simulation.

The length of the time-steps will affect the results of the calculation. The shorter the time-steps the more accurate the simulation of constantly changing internal conditions. Time-steps of 30 minutes, 15 minutes, and 10

minutes were evaluated. The corresponding moisture contents (g moisture/100 g dry product) at the end of the interval determined by the calculation for Product A were 4.0428%, 4.0427%, and 4.0427% respectively. Since these values are very close together, 30 minute timesteps were used.

Products B and C exhibited similar results. A program was written using Equations (3), (10), and (12) for a TI59 programmable calculator and the results of the calculation based on simulation were determined using this program.

EXPERIMENTAL METHODS

Three different commercially available packaged dry food products with low initial moisture contents were selected to be used for this study. All three products are mixtures of various ingredients with differing compositions. Thus their abilities to adsorb water vapor from the atmosphere are not the same and different shelf-lives can be expected. With a low initial moisture content (all three were different and covered a range) water vapor adsorption from the atmosphere to a maximum acceptable moisture content is the important criteria in determining shelf-life.

Product A is a gelatin product, B a dessert pudding, and C a coating mix. These products were tested in their existing packages which are paper/plastic laminates of varying degree of water vapor transmission. Packages are labeled A, B, C to correspond to products A, B, and C. By using existing packages produced on production equipment, variables attributed to pouch seals, other machineability defects, and rough handling are included in this study.

A. Data Generation For Calculation

The calculation based on simulation uses certain properties of the product, package, and environment to

predict moisture content. The initial moisture content, sorption isotherm, water vapor transmission rate of the packaging material, and surface area, fill weight, and headspace volume of the package are required.

Initial moisture content

Many methods are available for determining the moisture content of a food. Moisture content means the determination of the water held in the food. A common method of determining moisture content is to remove the water from the food and measure the weight change.

This can be accomplished by drying the food at some temperature for a time period and measuring the weight change. The air oven technique generally uses high temperatures for a long period of time. This high temperature can cause chemical reactions in the food affecting the change in weight and at the same time volatiles may be lost.

The vacuum oven technique uses a lower temperature and shorter drying time than conventional oven techniques. The rate of diffusion of water is faster by drying in a vacuum. However, there is still the possibility of weight loss due to evolution of volatiles from the food.

Other methods include freeze-drying at room temperature, drying with P_2O_5 desiccant, extracting or volatilizing the water in the food by means of an organic solvent, and methods based on the physical-chemical properties of bound and free water.

After reviewing the available methods, it was decided to use the vacuum oven technique. This method is widely used for food products because it is an efficient method.

Three samples of each product containing approximately 3 grams of product (each sample was obtained from a different package) were placed in a vacuum oven at 60°C for 4 hours. The average percentage of moisture content on a dry basis was determined by the loss of weight of the samples due to loss of moisture and expressed in units of $\frac{\text{g moisture}}{100 \text{ g dry product}}$. Results are in Table 1.

Sorption isotherms

The sorption isotherms were determined for each product by placing samples of known initial moisture content in contact with a range of relative humidities at three constant temperatures and measuring the weight gain or loss.

Constant humidities can be created by materials whose affinity for water regulates the water vapor pressure in the atmosphere surrounding the material. A saturated aqueous salt solution in contact with an excess of a definite solids phase at a given temperature will remain a constant humidity within any enclosed space around it. By properly selecting the salt to be used a wide range of humidities can be obtained and controlled.

There has been numerous data published relating relative humidity at a constant temperature for particular salts in a closed environment. The attainment of stable relative humidities is possible if certain procedures are followed.

Chemically pure salts and distilled water must be used as small amounts of contaminants can seriously affect the determination of the equilibrium humidity condition.

The preparation of the salt solution should be a slush with excess undissolved crystals. Too much water will produce a higher humidity than expected and solid crystals sticking above the surface of the solution can reduce the humidity.

The surface area of the solution should be as large as possible and the vapor space as small as possible. The air should also be circulated over the solution by some means.

The sorption isotherms for the three products were determined at three temperatures and eleven relative humidities by using saturated aqueous solutions of salts. The salts used and their corresponding relative humidities at the three temperatures are listed in Table 4.

Approximately 3 grams of each product were weighed into aluminum dishes then placed over a saturated salt solution in a closed container. Three 3 gram samples were used for each product at each condition.

Samples were periodically weighed on an analytical balance until no weight gain or loss was obtained indicating equilibrium had been reached. At the time equilibrium was attained, the equilibrium moisture content was calculated and expressed in units of $\frac{\text{g moisture}}{100 \text{ g dry product}}$. To obtain the sorption isotherms the average equilibrium moisture content of the three repeat samples was plotted against the corresponding relative humidity at three temperatures. Results are in Table 4 and plots of the sorption isotherms are in Figures 2, 3, and 4.

Water vapor transmission rate

The water vapor transmission rates were determined in the laboratory of the manufacturer that supplied the three products. Water vapor transmission rates were determined using ASTM Standard Method F372-73 which is a rapid procedure for determining WVT of flexible barrier materials in film or sheet form using an infrared detection technique. This method covers the use of the MoCon IRD-2 Infrared Diffusometer.

In this method a dry chamber is separated from a wet chamber of known temperature and humidity by the barrier material to be tested. The time for a given increase in water vapor concentration of the dry chamber is measured by monitoring the differential between two bands in the infrared spectral region, one in which water molecules adsorb

and the other where they do not. This information is then used to calculate the water vapor movement through a known area of barrier material.

The water vapor transmissions were determined at three temperatures--100, 92, and 83°F with use of $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ as the saturated salt solution. This solution gives a relative humidity of 90% at the three temperatures mentioned above. Results are in Table 3.

Surface area of the pouch

Surface area measurements were made by measuring the web width of each pouch structure and multiplying by the cutoff length. The average of three pouch area measurements for each packaging structure are listed in Table 2.

Fill weight of the package

The package fill weight for each product was determined by subtracting the pouch weight from the total package weight and averaging the values of three samples. Results are in Table 2.

Headspace volume of the package

The headspace volume of flexible packages is difficult to determine due to the fact that flexible packages do not have three independently fixed dimensions so the volume cannot be reliably calculated. Also, flexible packages can change shape and volume with changes in pressure.

Therefore the headspace volume should be calculated to standard temperature and pressure.

There is a method for determining the headspace volume resembling that of Griffin (1972) that uses Archimedes Principle, Boyle's Law, and the combined gas law. An attempt was made to use this method in determining the headspace volume but was found to be inadequate due to the nature of the pouch material.

The only alternative was to make an estimate of the headspace volume by determining the approximate dimensions of the headspace. By substituting various headspace values higher and lower than the estimated value into the equation for calculation of moisture content, it was determined that the headspace measurement had little effect on the calculated results. This was due to the fact that the water vapor that permeates the package equilibrates with the food inside the package faster than the permeation across the barrier. There is a small portion of the total moisture in the headspace compared to the moisture in the product. Results are in Table 2.

B. Experimental Testing

Initial moisture content

Initial moisture contents of Products A and B are reported in Table 1. It was necessary to redetermine the moisture content of Product C since there was a time lapse

between the previous determination as used for the sorption isotherms and the time when the experimental testing was conducted. This value is listed in Table 1.

Test conditions of actual
experimental testing

Three temperatures were established, two in controlled atmosphere walk-in cabinets and one in a controlled condition laboratory. Four relative humidities were set-up at each temperature. The relative humidities were established by placing beakers of saturated salt solutions with an excess of the definite solids phase in closed containers.

Five packages of Product A, B, and C were weighed on an analytic balance and placed over a saturated salt solution at a temperature for a certain time period. At the end of the time period the weight of each pouch was determined and the moisture content calculated.

After the pouches were weighed they were transferred to another previously set-up condition where the temperature, relative humidity, or both were changed. At the end of this second interval the pouches were again weighed, the moisture content calculated, and then transferred to another interval. In this manner the conditions were constant during any one interval but constantly changed from interval to interval.

Each product was subjected to at least one full cycle defined as transferring the samples from low temperature-low humidity through intervals up to conditions of

Table 1.--Initial moisture content.

	% Moisture - for sorption isotherms ($\frac{\text{g moisture}}{100\text{g dry product}}$)	% Moisture - for experimental test ($\frac{\text{g moisture}}{100\text{g dry product}}$)
Product A	0.725	0.725
Product B	4.042	4.042
Product C	3.555	3.690

Table 2.--Pouch surface area, headspace volume, and package fill weight.

	Surface area(cm ²)	Headspace volume(cm ³)	Package fill wt(g)
Package A	206	25	84.5
Package B	281	70	102.4
Package C	292	35	66.8

Table 3.--Water vapor transmission rates.

	Temp. (°F)	RH(%)	WVTR(g/100in ² ·24 hrs)
Package A	100	90	0.23
	92	90	0.12
	83	90	0.08
Package B	100	90	0.68
	92	90	0.37
	83	90	0.24
Package C	100	90	0.19
	92	90	0.10
	83	90	0.06

Table 4.--Test conditions, salt solutions, and equilibrium moisture content results for sorption isotherms.

Temperature (°F)	Salt(*) Solution	Relative Humidity (%)	Equilibrium Moisture Content ($\frac{\text{g moisture}}{100\text{g dry product}}$)		
			Product		
			A	B	C
90	(NH ₄) ₂ SO ₄	79.5	11.33	30.64	35.25
	NaCl	75.2	3.54	21.58	25.49
	NaNO ₂	62.9	1.55	6.35	7.87
	NaBr	55.5	1.04	5.01	6.52
	Mg(NO ₃) ₂	50.8	0.90	4.82	5.82
	KNO ₂	46.9	0.75	4.63	5.32
	K ₂ CO ₃	43.6	0.78	4.40	4.95
	CrO ₃	40.1	0.76	4.22	4.49
	MgCl ₂	32.2	0.64	3.90	3.80
	KC ₂ H ₃ O ₂	21.7	0.63	1.83	3.06
	LiCl	11.1	0.42	1.29	2.25
76	(NH ₄) ₂ SO ₄	80.0	6.01	32.73	37.96
	NaCl	75.4	3.11	13.11	15.08
	NaNO ₂	64.5	1.92	6.49	8.56
	NaBr	58.0	1.35	5.50	7.10
	Mg(NO ₃) ₂	53.1	1.20	5.21	6.48
	KNO ₂	48.2	1.06	4.81	5.52
	K ₂ CO ₃	43.8	0.86	4.90	5.10
	CrO ₃	39.5	0.82	4.47	4.44
	MgCl ₂	32.8	0.83	4.38	3.98
	KC ₂ H ₃ O ₂	22.9	0.68	3.96	3.37
	LiCl	11.1	0.59	1.75	2.68
62	(NH ₄) ₂ SO ₄	80.6	4.92	21.43	35.01
	NaCl	75.6	3.61	11.64	19.33
	NaNO ₂	66.2	1.90	9.99	9.27
	NaBr	60.5	1.48	5.53	7.65
	Mg(NO ₃) ₂	55.4	1.31	5.29	6.94
	KNO ₂	49.6	1.20	4.99	5.95
	K ₂ CO ₃	44.1	0.98	4.78	5.27
	CrO ₃	37.6	0.90	4.57	4.84
	MgCl ₂	33.3	0.80	4.18	4.22
	KC ₂ H ₃ O ₂	23.0	0.76	4.13	3.78
	LiCl	11.1	0.58	2.96	2.80

*Wink and Sears (1950).

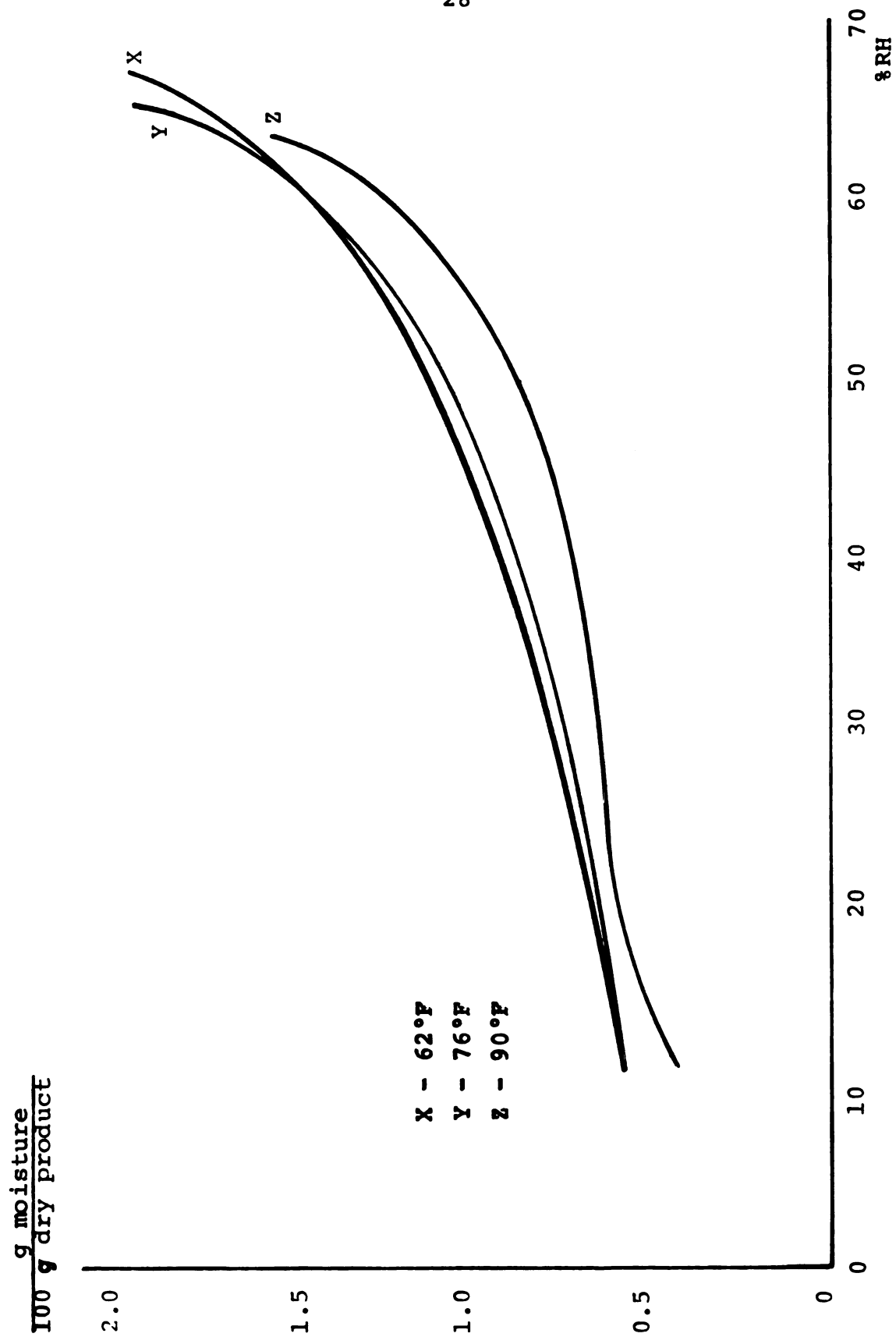


Figure 1. Adsorption isotherms--Product A.

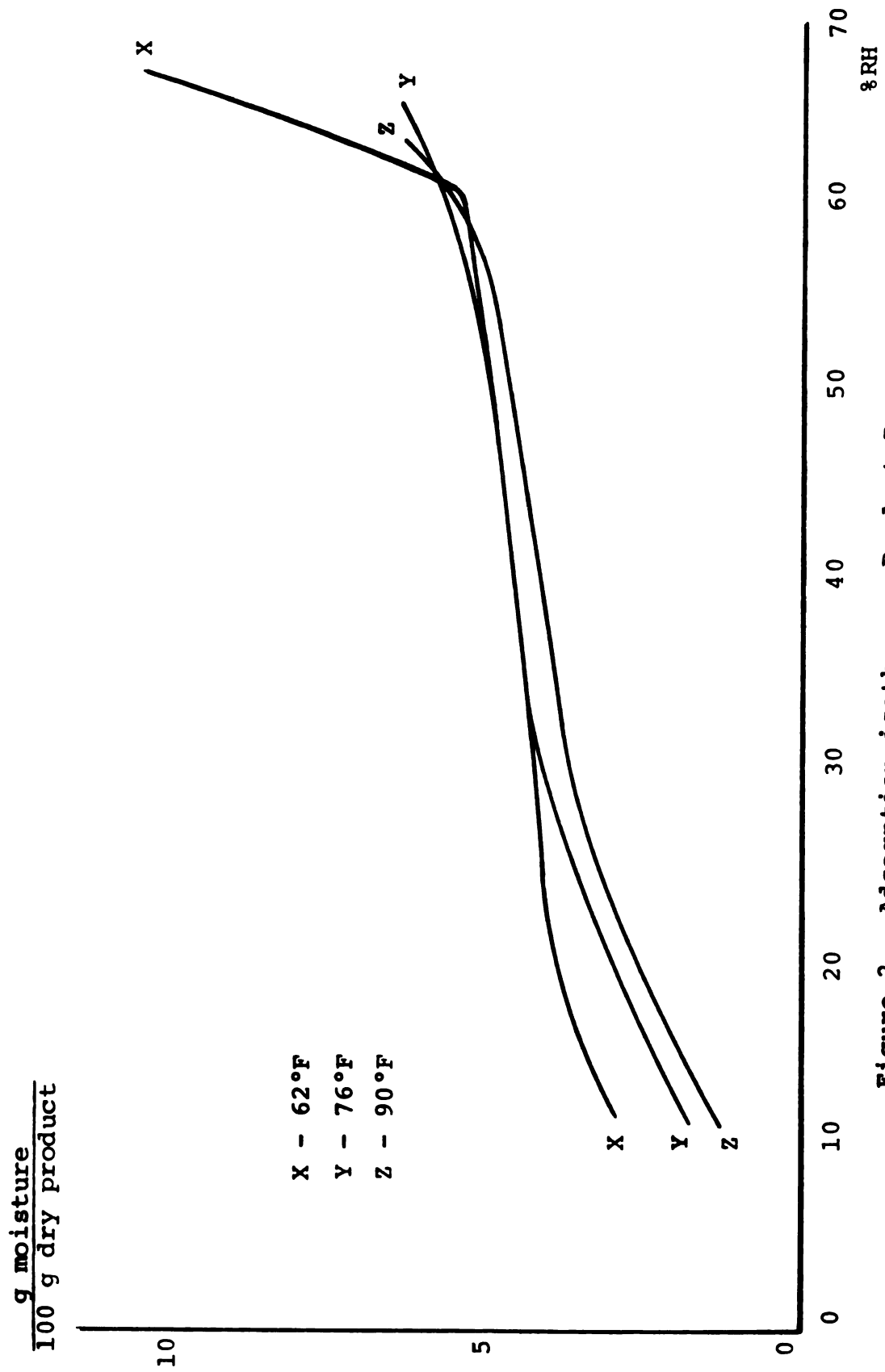


Figure 2. Adsorption isotherms--Product B.

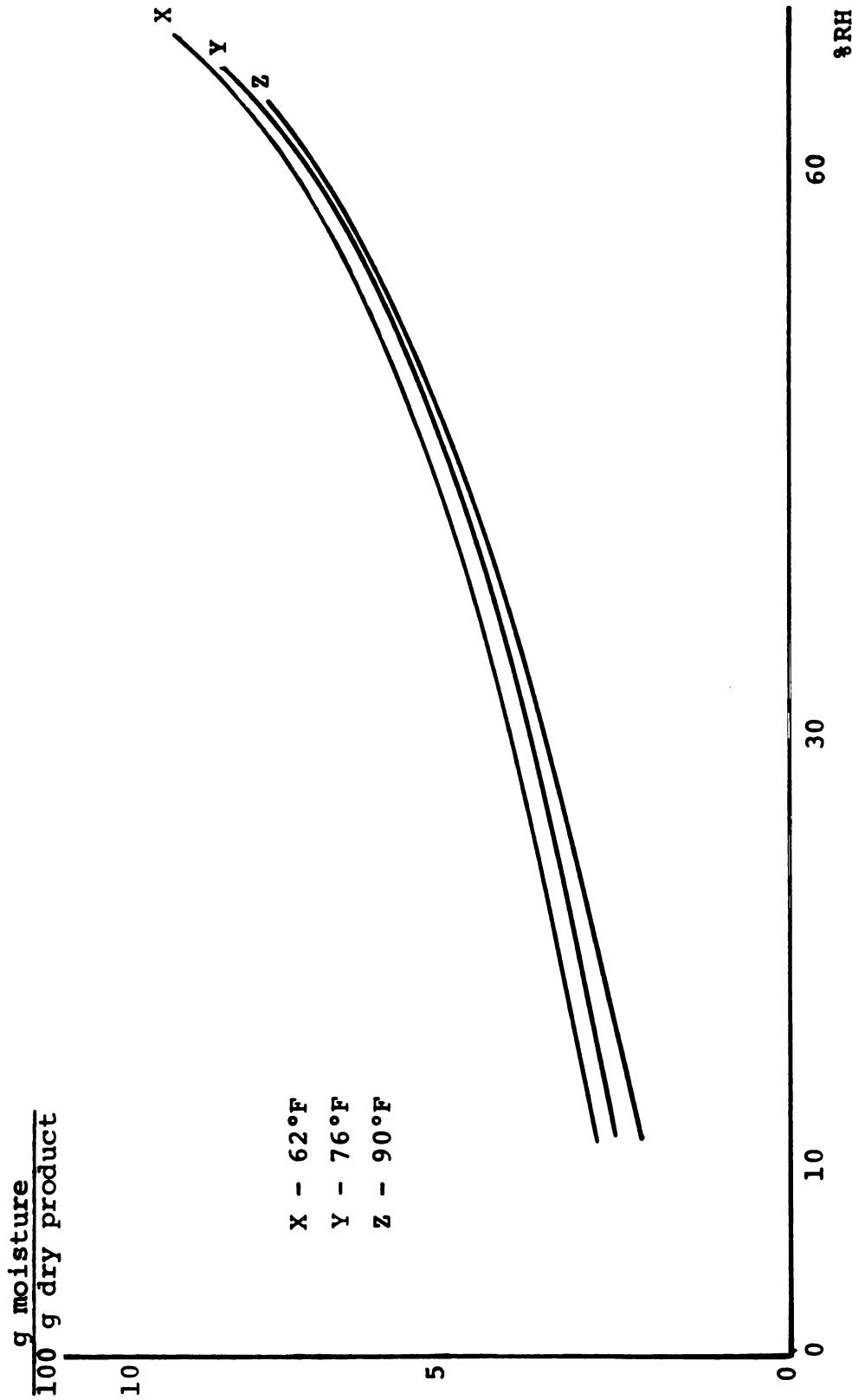


Figure 3. Adsorption isotherms--Product C.

high temperature-high humidity and back down again. Actual conditions that each product were subjected to and the corresponding time periods are listed in Tables 5, 6, and 7.

Calculation of experimental moisture content

The moisture content calculations at each weighing were based on first determining the dry weight of the product at time $t=0$ which has a moisture content equal to the initial moisture content. The dry weight at time $t=0$ is determined by:

$$D_o = \frac{W_o}{1 + \frac{m_i}{100}}$$

where

W_o is the weight of the product in the pouch at $t=0$, grams m_i is the initial moisture content of the product,

$$\frac{\text{g moisture}}{100 \text{ g dry product}}$$

D_o is the dry weight of the product at time $t=0$, grams.

The moisture content at any weighing can now be determined by:

Table 5.--Experimental test conditions; results of experimental and calculated moisture contents--
Product A.

Interval #	Relative Humidity (%)	Temp. (°F)	Total Time (Hours)	Average moisture content (% Dry Basis)	Low Moisture Content (% Dry Basis)	High Moisture Content (% Dry Basis)	Calculated Moisture Content (% Dry Basis)
0				0.725	0.725	0.725	0.725
1	37.6	62	17.83	0.732	0.731	0.734	0.728
2	60.5	62	22.50	0.738	0.736	0.740	0.729
3	80.6	62	26.50	0.759	0.754	0.762	0.731
4	32.8	76	31.75	0.739	0.737	0.742	0.731
5	43.8	76	40.42	0.737	0.734	0.740	0.733
6	53.1	76	44.42	0.741	0.738	0.744	0.734
7	75.4	76	49.25	0.756	0.753	0.759	0.737
8	46.9	90	53.17	0.735	0.730	0.741	0.737
9	55.5	90	62.50	0.738	0.734	0.743	0.739
10	62.9	90	66.92	0.744	0.739	0.751	0.741
11	75.2	90	71.25	0.758	0.751	0.765	0.744
12	79.5	90	75.58	0.767	0.763	0.771	0.747
13	75.2	90	90.83	0.784	0.772	0.797	0.757
14	62.9	90	94.83	0.780	0.776	0.794	0.758
15	55.5	90	98.83	0.776	0.765	0.789	0.759
16	75.4	76	113.33	0.805	0.791	0.821	0.766
17	64.5	76	117.50	0.809	0.796	0.827	0.768
18	58.0	76	136.50	0.813	0.798	0.832	0.773
19	48.2	76	158.0	0.818	0.798	0.850	0.777
20	80.6	62	162.58	0.844	0.829	0.864	0.779
21	66.2	62	167.91	0.846	0.830	0.867	0.780
22	60.5	62	172.08	0.843	0.827	0.865	0.781
23	49.6	62	181.25	0.792	0.775	0.810	0.782
24	37.6	62	184.58	0.789	0.768	0.812	0.783
25	49.6	62	188.41	0.789	0.773	0.808	0.784
26	66.2	62	204.08	0.847	0.830	0.868	0.787
27	80.6	62	208.58	0.850	0.838	0.873	0.789

Table 6.--Experimental test conditions; results of experimental and calculated moisture contents--
Product B.

Interval #	Relative Humidity (%)	Temp. (°F)	Total Time (Hours)	Average Moisture Content (% Dry Basis)	Low Moisture Content (% Dry Basis)	High Moisture Content (% Dry Basis)	Calculated Moisture Content (% Dry Basis)
0				4.042	4.042	4.042	4.042
1	37.6	62	4.75	4.052	4.047	4.055	4.043
2	49.6	62	9.42	4.053	4.049	4.058	4.045
3	66.2	62	21.59	4.056	4.052	4.064	4.054
4	80.6	62	25.84	4.070	4.062	4.079	4.058
5	48.2	76	29.92	4.039	4.032	4.047	4.062
6	58.0	76	34.09	4.044	4.037	4.055	4.066
7	64.5	76	45.84	4.059	4.049	4.075	4.083
8	75.5	76	49.92	4.063	4.052	4.080	4.090
9	55.5	90	54.25	4.049	4.035	4.068	4.095
10	62.9	90	67.67	4.069	4.054	4.094	4.117
11	75.2	90	71.92	4.082	4.064	4.110	4.128
12	79.5	90	76.25	4.094	4.076	4.127	4.141
13	75.2	90	92.58	4.121	4.094	4.163	4.180
14	62.9	90	96.50	4.118	4.091	4.161	4.186
15	55.5	90	100.83	4.119	4.090	4.161	4.191
16	75.4	76	116.41	4.161	4.129	4.211	4.216
17	64.5	76	120.49	4.161	4.128	4.213	4.221
18	58.0	76	124.24	4.162	4.128	4.215	4.225
19	48.2	76	142.91	4.165	4.129	4.220	4.237
20	80.6	62	164.99	4.221	4.174	4.284	4.258
21	75.6	62	168.99	4.218	4.172	4.282	4.261
22	66.2	62	172.66	4.209	4.163	4.275	4.264
23	49.6	62	185.41	4.209	4.163	4.274	4.268
24	37.6	62	190.24	4.205	4.159	4.271	4.268
25	49.6	62	194.57	4.212	4.165	4.280	4.269
26	66.2	62	207.15	4.222	4.172	4.291	4.278
27	80.6	62	211.40	4.227	4.176	4.297	4.281
28	48.2	76	215.40	4.20	4.148	4.271	4.283
29	58.0	76	221.32	4.199	4.146	4.271	4.289
30	64.5	76	230.24	4.211	4.155	4.284	4.300
31	75.4	76	234.24	4.218	4.160	4.294	4.306
32	55.5	90	239.07	4.202	4.144	4.280	4.311
33	62.9	90	244.07	4.206	4.145	4.284	4.317
34	75.2	90	254.65	4.237	4.172	4.326	4.339

Table 7.--Experimental test conditions; results of experimental and calculated moisture contents--
Product C.

Interval #	Relative Humidity (%)	Temp. (°F)	Total Time (Hours)	Average Moisture Content (% Dry Basis)	Low Moisture Content (% Dry Basis)	High Moisture Content (% Dry Basis)	Calculated Moisture Content (% Dry Basis)
0				3.690	3.690	3.690	
1	37.6	62	4.67	3.696	3.694	3.699	3.690
2	49.6	62	16.92	3.687	3.685	3.690	3.693
3	66.2	62	21.17	3.693	3.690	3.698	3.694
4	80.6	62	25.67	3.699	3.693	3.702	3.696
5	48.2	76	29.50	3.673	3.669	3.680	3.697
6	58.0	76	41.33	3.685	3.679	3.688	3.702
7	64.5	76	45.41	3.690	3.685	3.694	3.704
8	75.4	76	49.83	3.703	3.695	3.706	3.707
9	55.5	90	63.33	3.674	3.669	3.680	3.716
10	62.9	90	67.58	3.686	3.678	3.691	3.720
11	75.2	90	71.83	3.695	3.687	3.699	3.725
12	79.5	90	88.41	3.740	3.727	3.747	3.747
13	75.2	90	92.24	3.739	3.729	3.745	3.752
14	62.9	90	96.91	3.732	3.723	3.740	3.756
15	55.5	90	112.49	3.736	3.724	3.746	3.767
16	75.4	76	116.66	3.767	3.757	3.780	3.769
17	64.5	76	120.33	3.770	3.758	3.782	3.771
18	58.0	76	138.91	3.775	3.762	3.792	3.778
19	48.2	76	160.99	3.775	3.760	3.793	3.784
20	80.6	62	165.32	3.799	3.784	3.819	3.786
21	75.6	62	168.74	3.806	3.789	3.823	3.787
22	66.2	62	181.57	3.812	3.796	3.834	3.791
23	49.6	62	186.49	3.805	3.788	3.827	3.792
24	37.6	62	190.91	3.804	3.786	3.827	3.792
25	49.6	62	203.58	3.807	3.787	3.830	3.794
26	66.2	62	208.0	3.808	3.790	3.830	3.796
27	80.6	62	212.25	3.829	3.808	3.850	3.797
28	48.2	76	217.92	3.785	3.766	3.808	3.799
29	58.0	76	226.84	3.790	3.771	3.813	3.802
30	64.5	76	230.92	3.797	3.777	3.822	3.804
31	75.4	76	236.0	3.810	3.789	3.834	3.807
32	55.5	90	240.92	3.778	3.754	3.805	3.810
33	62.9	90	251.59	3.792	3.766	3.815	3.819

$$Mc_t = \frac{Wt - Do}{Do} \times 100$$

where

Wt is the weight of the product in the pouch at time t, grams

Do is the dry weight of the product at time t=0, grams

Mc_t is the moisture content at any weighing at time t,

$$\frac{\text{g moisture}}{100 \text{ g dry product}}$$

Results are in Tables 5, 6, and 7.

CALCULATION

The calculation based on simulation requires the permeability constant of the packaging material and a mathematical expression for the sorption isotherm.

Calculation of Permeability Constant from WVTR

As discussed previously the water vapor transmission rates were determined by an infrared detection technique at three temperatures and one relative humidity for each structure. These values were converted to permeability constant over thickness ($\frac{P}{x}$) having units of $\frac{\text{gms}}{\text{cm}^2 \cdot \text{hr} \cdot \Delta \text{atm}}$, by assuming that the partial pressure of water vapor in the dry chamber is zero atm. This assumption is based on the fact that the dry chamber is purged with dessicated air.

The permeability constants were determined at three temperatures that were different from the experimental storage temperatures used. The logarithm of the permeability constant was plotted against the inverse of the testing condition temperature in degrees Kelvin. By referring to this plot the permeability constant at other temperatures can be determined. Results of the permeability constants determined from the WVTR's and the extrapolated values at

experimental test temperatures are in Tables 8 and 9. The permeability constants as a function of temperature are in Figures 4, 5, 6, and 7.

Calculation of Slope and Intercept

As discussed previously it was determined the sorption isotherm could be described by a straight line and expressed mathematically as:

$$m = a + bH$$

The slope and intercept was determined between the initial moisture content and estimated maximum allowable moisture content. Results are reported in Table 10.

Determination of Moisture Content by Calculation Based on Simulation

The moisture contents versus time for the same conditions corresponding to those used in the experimental determination were calculated using Equations (3), (10), and (12). A program utilizing these equations was written for a TI59 programmable calculator. Appropriate values were entered into the program and moisture contents calculated. Results are in Tables 5, 6, and 7. These values were plotted on the same graph as the experimentally determined values so a direct visual comparison can be made. These plots are illustrated in Figures 8, 9, and 10.

Table 8.--Permeability constant as determined from water vapor transmission rates.

Permeability constant÷film thickness		
Temp. (°F)		
	$\frac{P}{x'}$	$\frac{g(H_2O)}{(cm^2)(hr)(\Delta ATM)}$
Package A	100	0.000255
	92	0.000170
	83	0.000151
Package B	100	0.000755
	92	0.000525
	83	0.000453
Package C	100	0.000211
	92	0.000142
	83	0.000113

Table 9.--Permeability constant at temperatures used in experimental testing.

Permeability constant÷film thickness		
Temp. (°F)		
	$\frac{P}{x'}$	$\frac{g(H_2O)}{(cm^2)(hr)(\Delta ATM)}$
Package A	90	0.000163
	76	0.000147
	62	0.000140
Package B	90	0.000500
	76	0.000426
	62	0.000400
Package C	90	0.000133
	76	0.000103
	62	0.000092

Table 10.--Slope and intercept from sorption isotherms.

Product	Temperature (°F)	Slope b	Intercept a
A	62	0.00388	0.6707
	76	0.00799	0.5224
	90	0.02143	-0.1581
B	62	0.0492	2.564
	76	0.0424	2.988
	90	0.0405	2.596
C	62	0.0642	2.091
	76	0.0616	1.959
	90	0.0773	1.360

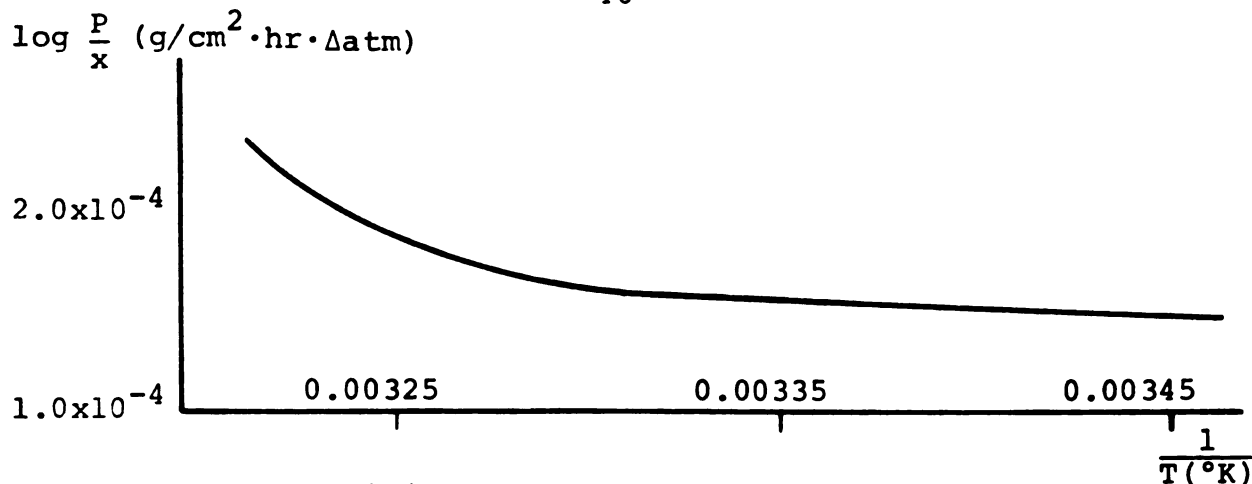


Figure 4. Permeability constant as a function of temperature--Package A.

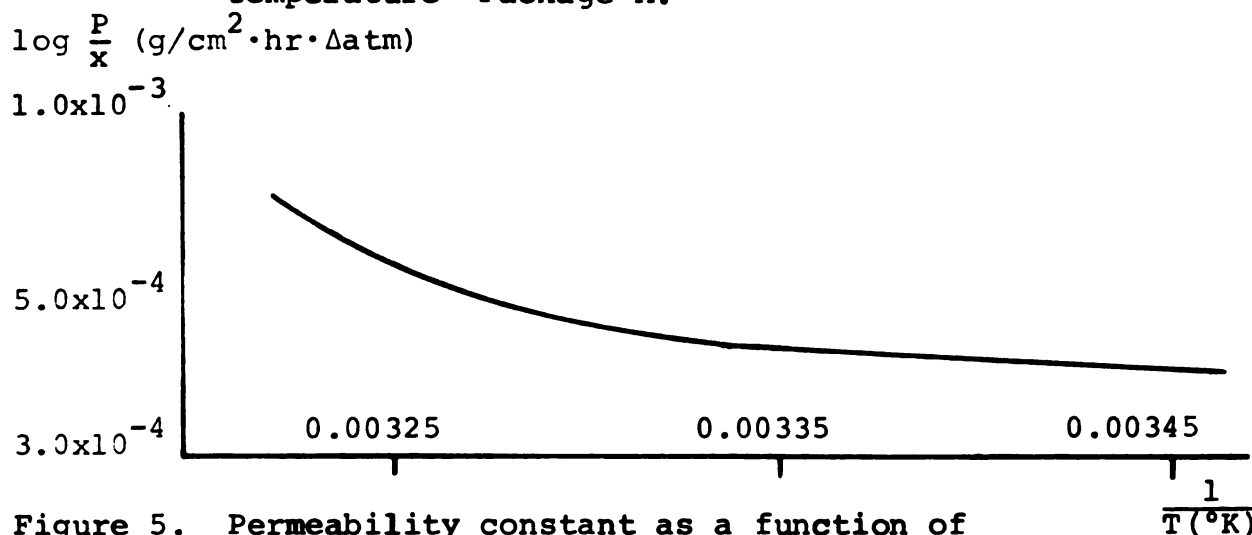


Figure 5. Permeability constant as a function of temperature--Package B.

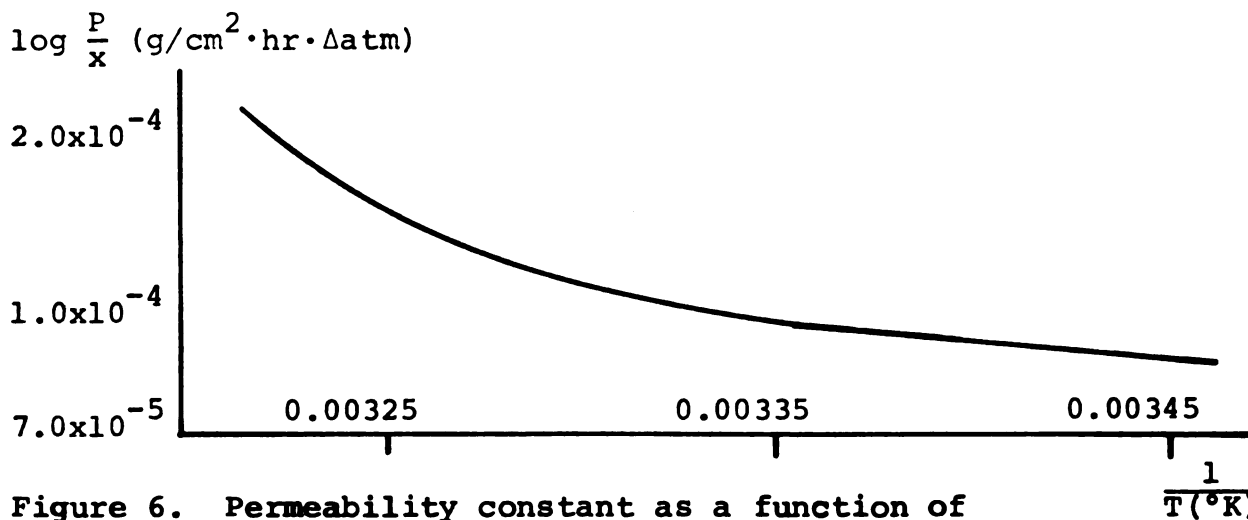


Figure 6. Permeability constant as a function of temperature--Product C.

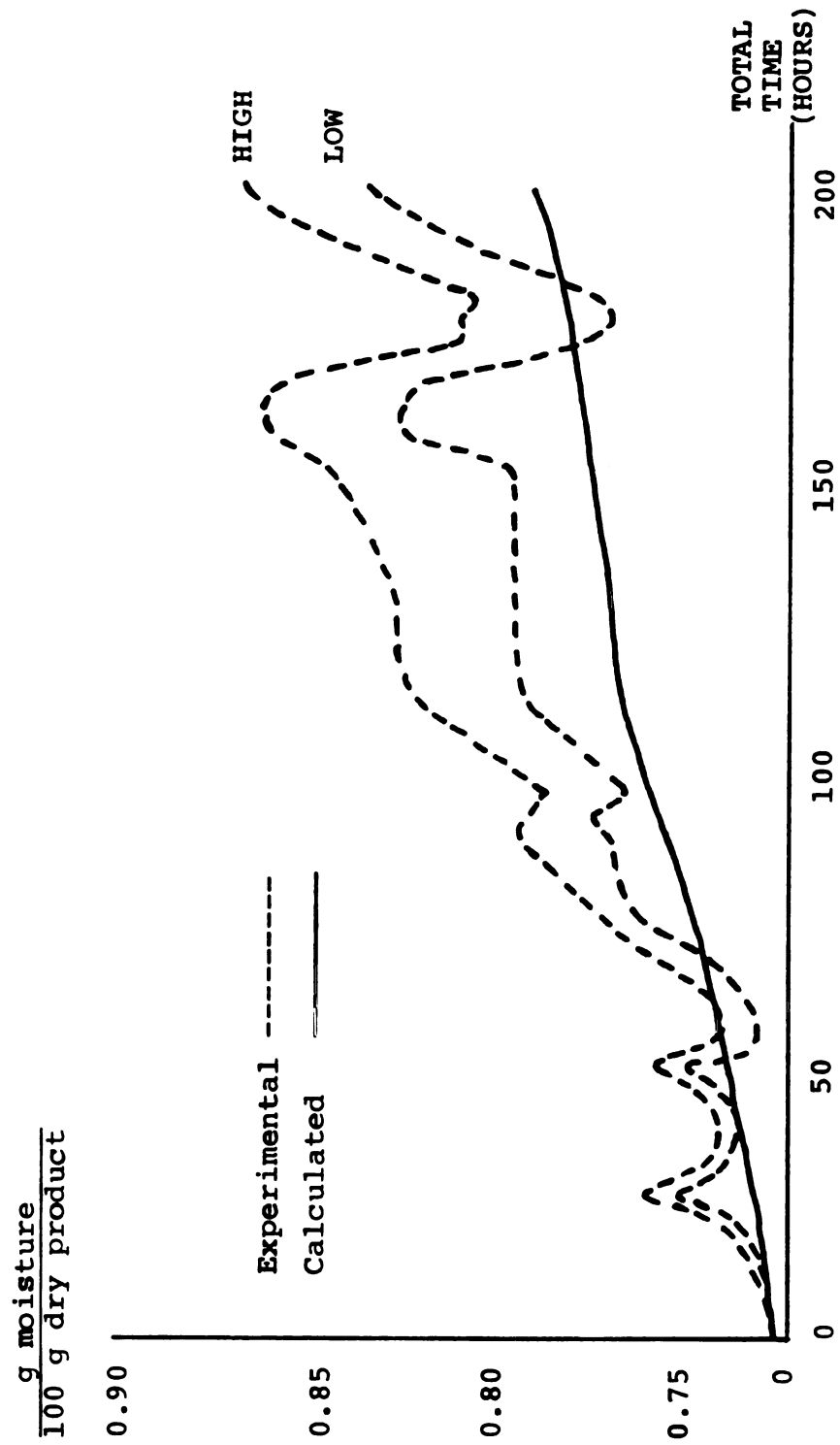


Figure 7. Experimental and calculated results--Product A.

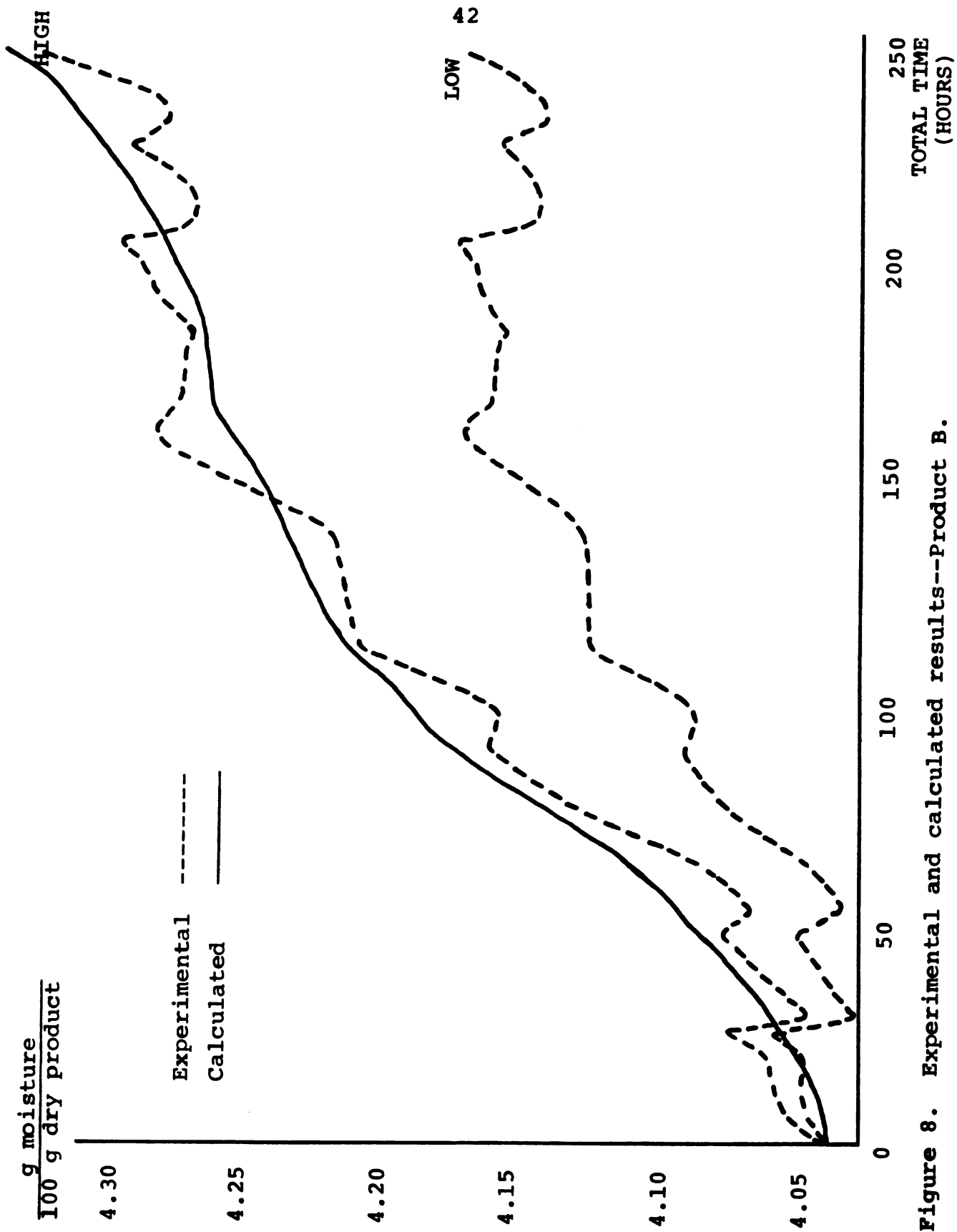


Figure 8. Experimental and calculated results--Product B.

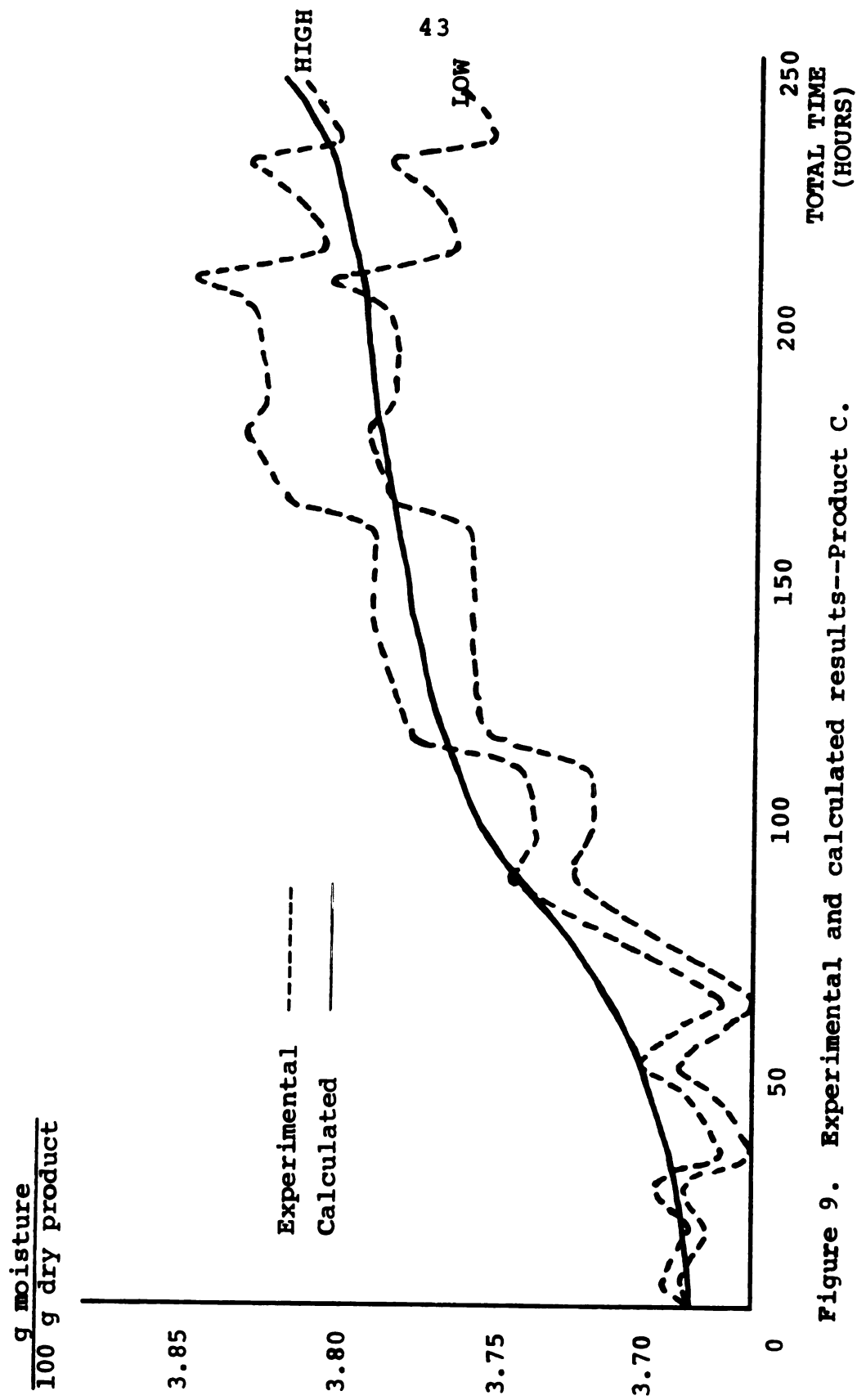


Figure 9. Experimental and calculated results--Product C.

RESULTS AND DISCUSSION

The moisture content results at the end of each interval as determined through time-steps by the calculation method compared very well with the moisture content results determined experimentally. The greatest percentage difference for each product is in Table 11.

Table 11.--Greatest percentage difference between calculated and experimental data.

Product	% Difference
	$(\frac{M_{ex} - M_{calc}}{M_{ex}} \times 100)$
A	10.0
B	4.1
C	1.5

Because the times the packages were subjected to the conditions at each interval were relatively short, the incremental moisture content changes between intervals were relatively small. The overall increases between initial and final moisture contents were most significant. The overall increase results are in Table 12.

Table 12.--Overall moisture content increases.

Product	Overall experimental moisture content increase (%)			Overall calculated moisture content increase (%)
	ave	low	high	
A	17.2	15.6	20.4	8.8
B	4.8	3.2	7.0	7.3
C	2.8	2.1	3.4	3.5

The experimental moisture content results of the five samples of each product varied significantly from low to high values. The overall variations are listed in Table 12 and illustrated graphically in Figures 7, 8, and 9. The overall calculated moisture content results compared very well to the range of the experimental results for Products B and C and not as well for Product A.

The results of the calculation did not follow the graphs of the experimental results exactly as seen in Figures 7, 8, and 9. For instance, when the package was subjected to a lower relative humidity at the same temperature as the previous interval, the moisture content decreased. The calculated results was not able to follow the experimental decrease in moisture content. The reason for this can be attributed to the fact that the calculation is based on the erroneous assumption that the product in the package is in equilibrium with the relative humidity of the internal environment. .

One factor in the calculation is the internal relative humidity of the package at the beginning of any interval. Assuming equilibrium in the package, the change of the weight of the moisture penetrated through the container for a period of time is defined as:

$$\Delta Q = \frac{P}{x} \cdot A \cdot \frac{p_s}{100} (H_e - H_i) \Delta t \quad (8)$$

For any specific interval the temperature and external relative humidity are constant. Therefore, the only variable in the above equation is the internal relative humidity (H_i). The direction of the penetration of water vapor will depend on the value of $(H_e - H_i)$. If this value is positive water vapor will penetrate into the package. But, when this value is negative the internal relative humidity is greater than the external relative humidity and the concentration gradient (partial pressure difference) is such that there will be a net loss of water vapor from the package to the external environment. This results in a lower calculated moisture content than the previous interval.

The relationship between the equilibrium moisture content and relative humidity can be expressed by the sorption isotherm or as: $m = a + bH_i$, where a and b are constant for each product at a temperature.

By rearranging this equation to $H_i = \frac{m - a}{b}$, the internal relative humidity can be determined at any time as

a function of the moisture content and the slope and intercept of the sorption isotherm.

The sorption isotherm of a product is determined at a constant temperature. At various temperatures the sorption isotherm will be different and thus the relationship between the internal relative humidity and moisture content will change as the temperature changes. This can be illustrated for Products A, B, and C in Figures 2, 3, and 4 which show the sorption isotherm at three temperatures.

The calculation is based on the assumption of equilibrium between the product and the internal humidity of the package. When a package was transferred from one temperature to another, the internal conditions changed greatly according to the relationship between the moisture content and relative humidity as a function of the sorption isotherm. The product and internal environment will require a certain time to equilibrate. Since equilibrium is assumed for the calculation, the theoretical internal relative humidity will be lower than the actual internal relative humidity.

Therefore, when the experimental moisture content decreases, the internal relative humidity must be greater than the external relative humidity according to Equation (8). Since the theoretical internal relative humidity is lower than the actual internal relative humidity because of the assumption of equilibrium, the calculation did not give decreased moisture content values corresponding to decreased experimental moisture content values.

Proof that equilibrium had not been reached can be illustrated by the experimental results of intervals 16 and 17 in Table 5. Even though the external relative humidity decreased from 75.4% to 64.5%, the product still adsorbed water vapor and thus the moisture content increased. If the product and internal conditions had reached equilibrium in storage interval 16, the relative humidity inside the package would have been greater than the external relative humidity of storage interval 17. The result would have been a concentration gradient (partial pressure difference) such that the product loses water vapor to the lower external relative humidity. The same situation can be illustrated by intervals 20 and 21.

One reason why equilibrium had not been reached was the short time periods the packages were subjected to the conditions at each interval before transferring to the next storage interval. Secondly, the packaging materials which were used are good water vapor barriers, therefore the moisture transfer during short time-periods is relatively small.

As can be seen in Figures 7, 8, and 9, although the calculated moisture content values never decreased, the trends of the calculated values did follow the trends of the experimental values very well for Products B and C and to a lesser extent for Product A.

This difference in trend for Product A can be seen in Figure 7 in which the trend of the calculated values followed the trend of the experimental values up to a point. Between

intervals 15 and 16 in Table 5 the experimental moisture content increased sharply while the calculated value increased much slower. The test conditions during this interval changed drastically from 90°F/55.5%RH to 76°F/75.4%RH. Because of the drastic condition change and the assumption of equilibrium this calculated value was not able to follow the experimental value. Drastic condition changes such as these are not common in the course of a normal day.

Figures 8 and 9 illustrate that the trends of the calculated values followed the trends of the experimental values very well for Products B and C. This can be attributed to the fact that the change of conditions between intervals for these two products were smoother and less severe than for Product A. The model was designed this way to determine the effect of severity of condition change on the results of the calculation. It was discovered that as the change of conditions became more severe, the deviation of the calculated to experimental results increased.

The experimental conditions the products were subjected to are much more severe than what would be expected in normal distribution. During the course of a normal day the weather conditions change at a gradual and smoother rate. For instance, the temperature does not ordinarily jump from 76 to 90°F in a matter of minutes or the relative humidity change from 60% to 80% in the same time period. The experimental model was designed to subject the products to drastic changes of temperature and humidity. Thus, since the calculation based on

simulation adequately predicted the moisture content for drastic changes over a short time period it can be assumed that this method can be used to predict the moisture content over a longer time period where the conditions change gradually and the product is closer to equilibrium with the environment.

Experimental Error of Actual Testing

In the determination of the initial moisture content evolution of volatile components of the food may have occurred even though the vacuum oven method was used to minimize these effects. If this was the case the loss of weight may have been more than just loss of moisture resulting in a higher moisture content determination than the actual moisture content. This measurement is critical since all subsequent moisture content values are based on the initial moisture content.

Five samples of each product were used in the actual testing at each interval. The results of the determination of moisture content at each interval varied throughout these five samples. This can be explained by material variations of the laminates, either variations in thickness or variations in the construction of the laminate. The quality of the seal may also have affected these results as long as it was not a defective seal resulting in a defective pouch. A leaker would cause extreme variation in the experimental moisture content determination.

During the course of the actual testing, the test condition containers were opened and closed periodically in

order to weigh the samples. Because of this the relative humidity above the saturated salt solutions required time to equilibrate. Therefore for a certain time period the actual relative humidity inside the test container may have been different than the desired relative humidity.

While one package was being weighed the other packages remained in the test container. Because the container had to be opened each time another package was weighed the conditions inside the container varied slightly which may have affected the moisture content of the product. To minimize this effect the order of weighing the samples was rotated.

Experimental Error of Calculation Based on Simulation

✓ (As was the case in the actual testing,) the initial moisture content determination is (also) critical in the calculation method. The calculation depends on the sorption isotherms which are determined by the equilibrium moisture content at different relative humidities. The equilibrium moisture content at each relative humidity is calculated from the total weight gain or loss at equilibrium and the initial moisture content. Any error in the initial moisture content determination would directly affect the sorption isotherms and thus affect the calculated results.

Also, the initial moisture content is used to determine the initial interval relative humidity of the package

at the beginning of the first interval. All subsequent calculated moisture contents depend on this initial internal relative humidity.

✓ In determining the sorption isotherms, samples were placed over a saturated salt solution in a closed container. Periodic weighings were made until equilibrium was established. In order to weigh the samples the containers were periodically opened and closed thus causing the relative humidities inside the test containers to fluctuate. Therefore the equilibrium moisture content may have been determined at a relative humidity other than the stated value.

✓ Saturated salt solutions can provide a stable humidity condition but the condition created is not necessarily the relative humidity cited in the literature. (Also, the various references disagree as to the humidity created.) *Since* No attempt was made to measure the actual relative humidity inside the test container. →

✓ Obtaining stable humidities depends on the purity of the salt, the purity of the water, the preparation of the salt solution, and the temperature.

Chemically pure salts and distilled water were used in the preparation of the solutions. Any excess salt crystals sticking above the surface of the solution or any excess of the aqueous phase will affect the humidity established.

✓ The temperature used for the determination of the sorption isotherms were average values over the course of the experiment. Fluctuations in temperature will affect the sorption isotherm determination. According to the literature a temperature difference of only 2°F between the solution and vapor causes an error of approximately 5% relative humidity. The temperature is also assumed constant for the calculation.

Determination of the water vapor transmission rates is another area of possible error. The MoCon instrument is a fast method for determining water vapor transmissions but introduces some error over the dish method.

Also the water vapor transmission rates were determined for a flat sheet of material. This value may differ somewhat from the water vapor transmission rate of the package itself. During machining of the pouch the laminated structure may be damaged and the moisture barrier properties reduced slightly. This is particularly evident for Products A and B packaged in gusseted pouches. The plow used to form the gusset may damage the laminated structure or a gusset fold may not be properly heat sealed resulting in a small air channel formed in the fold.

✓ Surface area was determined by measuring the flat sheet dimensions of a pouch and averaging the values of three pouches. Possible errors result from the accuracy of the measuring instrument and the measurement itself.

Since the packaging materials are laminates, variations in their construction will result in experimental error. A 5 to 10% variation in thickness is not uncommon in the packaging industry and any defects in the laminating layers will add to this error.

Manufacturers are faced with the question of the degree to which he must protect his product to get it to the consumer in an acceptable condition. One method of determining this is actual field storage which is both time consuming and expensive. An alternative is the accelerated test technique but to assume that "similar" products will have similar shelf-lives is inaccurate. This method has severe limitations and is still relatively expensive and time consuming. The method presented in this paper is not intended to replace actual field storage, accelerated test techniques, or other mathematical methods. It is a practical tool that can be used by a packaging engineer in package development when a new product has to be introduced in a short lead time and the package's performance must be known for a particular set of environmental conditions.

The shelf-life of packaged dry food products depends greatly upon the geographical location and time of year at which the packaged product is placed into that location. Because of this it may be economical to package differently for various geographical areas or even package differently in the same area during various times of the year.

By obtaining weather data for a geographical region the calculation based on simulation can be used to predict a package's performance in that region. Conversely, for a desired shelf-life in any particular region the required package protection can be determined thus reducing the possibility of overpackaging.

✓ Considering all of the possible errors in this study, the calculated results are in good agreement with the experimental results. The change of conditions used in the experiment were much more severe than those expected in the course of a normal day. The trends of the calculated results were able to follow the trends of the experimental results even though the conditions changed drastically between intervals.

✓ This calculation method is a quick, inexpensive, and reasonably accurate method for introduction of a product with short lead times but should be verified by actual storage monitoring during the course of the introduction. Verification is a common practice in food packaging no matter what method is used to predict storage life.

SUMMARY

The determination of a products' shelf-life is important to food packaging. Reactions that determine storage life of packaged dry food products depends mostly on the moisture content of the product.

A calculation based on simulation was proven to adequately predict moisture content, with certain limitations, during short constant condition storage intervals when the conditions change from interval to interval. Condition changes between intervals were more severe than those expected during the course of a normal day.

The calculation can be used as a practical tool by the packaging engineer in package development for introduction of a new product in short lead times. The development time can be reduced along with reducing the possibility of overpackaging or underpackaging. The performance of a package can be predicted in a particular geographical region by obtaining weather data for the region and applying it to the calculation. This type of determination is important because the shelf-life of packaged dry food products depends upon the geographical location and time of year the product is placed into that location. Determining the shelf-life of

a product by actual field storage in all market locations for introduction during all seasons of the year is expensive and time consuming.

Future work in this area should include actual field storage with constant monitoring of the environmental changes and periodic moisture content determinations. The recorded environmental conditions can then be used in the calculation to predict the moisture content with comparison to experimental moisture content. By actual field storage the change of conditions is gradual and smoother and there is a better chance of the product in the package being close to equilibrium with the internal environment.

REFERENCES

- Adamson, A. 1960. Physical Chemistry of Surfaces. J. Wiley and Sons, New York.
- Aguilera, J. M., Chirife, J., Flink, J., and Karel, M. 1975. Computer simulation of nonenzymatic browning during potato dehydration. Lebensm. Wiss. U. Technol. 8:128.
- Boquet, R., Chirife, J., and Iglesias, H. A. 1978. Equations for fitting water sorption isotherms of foods: Part II. J. Food Technol. 13, 319.
- Caurie, M. 1971. A single layer moisture adsorption theory as a basis for the stability and availability of moisture in dehydrated foods. J. Fd. Technol. 6: 193.
- Caurie, M., Tung-Ching Lee, Salomon, M., and Chichester, C. O. 1976. J. Food Sci. 41:448.
- Charie, H. J., East, E., and Van der Veen, W. 1963. Effect of moisture on storage properties of dehydrated food products. Proc. 1st Int'l Congress on Food Sci. and Technol. 1:45.
- Chirife, J. and Iglesias, H. A. 1978. Equations for fitting water sorption isotherms of foods: Part I--A review. J. Fd. Technol. 13, 159.
- Davis, E. G. 1970. Evaluation and selection of flexible films for food packaging. Fd. Technol. in Australia. 22:62.
- Easter, R. A. 1953. Forecasting shelf-life. Mod. Pkg. 2:128.
- Felt, C. E., Buechele, A. C., Borchardt, L. F., and Koehn, R. C. 1945. Determining shelf-life of packaged cereals. Cereal Chem. 22(3):261.
- Gregg, S. J. and Sing, K. S. 1967. Adsorption surface area and porosity. Academic Press, New York.
- Harrington, J. F. 1973. Packaging seed for storage and shipment. Seed Sci. and Technol. 1973:701.

- Heiss, R. 1958. Shelf-life determinations. Mod. Pkg. 8: 119-125, 176.
- Heiss, R. and Eichner, Dr. K. 1971. Moisture content and shelf-life: Part 1. Food Manu. 46(5):53.
- Iglesias, H. A., Boquet, R., and Chirife, J. 1977. On the evaluation of B.E.T. constants from the B.E.T. isotherm equation. J. Food Sci. 42(5):1387.
- Iglesias, H. A. and Chirife, J. 1976a. Prediction of the effect of temperature on water sorption isotherms of food material. J. Fd. Technol. 11, 109.
- Iglesias, H. A. and Chirife, J. 1976b. A model for describing the water sorption behavior of foods. J. Food Sci. 41:984.
- Iglesias, H. A., Chirife, J., and Lombardi, J. L. 1975. An equation for correlating equilibrium moisture content in foods. J. Fd. Technol. 10:289.
- International Critical Tables, Vol. 1, pp. 67-68, 1926.
- Karel, M. 1973. Quantitative analysis of food packaging and storage stability problems. Am. Inst. Chem. Eng. (Symp. Ser.) 69(132):107.
- Karel, M. 1975. Protective packaging of foods. In "Physical Principles of Food Preservation." Ed. Karel, M., Fennema, O. R., and Lund, D. B., p. 399. Marcel Decker, N.Y.
- Karel, M., Mizrahi, S., and Labuza, T. P. 1971. Computer prediction of food storage. Mod. Pkg. 44(8):54.
- King, J. C. 1968. Rates of moisture sorption and desorption in prous, dried foodstuffs. Fd. Technol. 22:165.
- Labuza, T. P. 1968. Sorption phemomena in foods. Food Technol. 22(3):263.
- Labuza, T. P. 1971. Kinetics of lipid oxidation in foods. CRC Crit. Rev. Food Technol. 2(10):355.
- Labuza, T. P. 1972. Nutrient losses during drying and storage of dehydrated foods. CRC Crit. Rev. Food Technol. 3:217.
- Labuza, T. P. 1973. Effects of dehydration and storage. Food Technol. 27:21.

- Labuza, T. P. 1975. Theory, determination and control of physical properties of food materials. D. Reidel Pub. Co. Dordrecht-Holland, 197.
- Labuza, T. P., Mizrahi, S., and Karel, M. 1972. Mathematical models for optimization of flexible film packaging of foods for storage. Trans. of the ASAE., 150.
- Labuza, T. P., Tannenbaum, S. R., and Karel, M. 1970. Water content and stability of low-moisture and intermediate moisture foods. Food Technol. 24(5):35.
- Landrock, H. H. and Proctor, B. E. 1951. A new graphical interpolation method for obtaining humidity equilibria data, with special reference to its role in food packaging studies. Food Technol. Aug. 1951.
- Lockhart, H. E. 1962. A review of the state of the art and summary of permeability theory. Mich. St. U. School of Pkg., Projects 5 and 6.
- Manathunya, V. 1976. Predictions of shelf-life of packaged cereal by an accelerated test technique and a mathematical model. Mich. St. U. School of Pkg. thesis.
- Mizrahi, S. and Karel, M. 1977a. Accelerated stability tests of moisture-sensitive products in permeable packages by programming rate of moisture content increase. J. Fd. Sci 42(4):958.
- Mizrahi, S. and Karel, M. 1977b. Accelerated stability tests of moisture sensitive products in permeable packages at high rates of moisture gain and elevated temperatures. J. Food Sci. 42(6):1575.
- Mizrahi, S., Labuza, T. P., and Karel, M. 1970a. Computer-aided predictions of extent of browning in dehydrated cabbage. J. Food Sci. 35:799.
- Mizrahi, S., Labuza, T. P., and Karel, M. 1970b. Feasibility of accelerated tests for browning in dehydrated cabbage. J. Food Sci. 35:804.
- Oswin, C. P. 1945. The kinetics of package life. Jr. Soc. Chem. Ind. Trans. 64:419.
- Quast, D. G. and Karel, M. 1972a. Computer simulation of storage life of foods undergoing spoilage by two interacting mechanisms. J. Food Sci. 37:679.

- Quast, D. G. and Karel, M. 1972b. Technique for determining oxygen concentration inside packages. J. Food Sci. 37:490.
- Quast, D. G. and Karel, M. 1973. Simulating shelf-life. Mod. Pkg. 3:50.
- Resnik, S. and Chirife, J. 1979. Effect of moisture content and temperature on some aspects of nonenzymatic browning in dehydrated apple. J. Food Sci. 44(2):601
- Rockland, L. B. 1969. Water activity and storage stability. Food Technol. 23(10):1243.
- Salwin, H. 1963. Moisture levels required for stability in dehydrated foods. Food Technol. 17:1114.
- Salwin, H. and Slawson, V. 1959. Moisture transfer in combinations of dehydrated foods. Food Technol. 12:715.
- Simon, I. B., Labuza, T. P., and Karel, M. 1971. Computer-aided prediction of food storage stability: Oxidation deterioration of shrimp product. J. Food Sci. 36:280.
- Strolle, E. O. and Cording, J., Jr. 1965. Moisture equilibria of dehydrated mashed potato flakes. Food Technol. 5:171.
- Vapor Pressures of Pure Substances. CRC Handbook of Chemistry and Physics 1977-78. p. D180.
- Viollaz, P., Chirife, J., and Iglesias, H. A. 1978. Slopes of moisture sorption isotherms of foods as a function of moisture content. J. Food Sci. 43:606.
- Wink, W. A. and Sears, G. R. 1950. Instrumentation studies LVII. Equilibrium relative humidities above saturated salt solutions at various temperatures. TAPPI 33(9): 96A.