

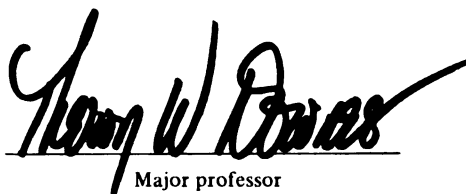
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**RATE OF LIPID OXIDATION AT VARYING INITIAL OXYGEN
 CONCENTRATIONS USING OXYGEN ABSORBING SACHETS IN
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Dena Briggs Thomas

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**RATE OF LIPID OXIDATION
AT VARYING INITIAL OXYGEN CONCENTRATIONS
USING OXYGEN ABSORBING SACHETS IN THE PACKAGES**

By

Dena Briggs Thomas

A THESIS

**Submitted to
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ABSTRACT

RATE OF LIPID OXIDATION AT VARYING INITIAL OXYGEN CONCENTRATIONS USING OXYGEN ABSORBING SACHETS IN THE PACKAGES

By

Dena Briggs Thomas

This study was performed to determine the effect of varying initial oxygen concentrations and the use of oxygen absorbers on the shelf life of potato chips packaged in metallized polyester and metallized polypropylene. Potato chips were stored for 22 weeks. Changes in headspace oxygen concentration, moisture content, and hexanal concentration were measured. The hexanal and sensory data showed that chips in metallized polypropylene at 10% initial oxygen concentration with an absorber were less rancid than chips at lower oxygen concentrations without absorbers and less rancid than chips at 2% initial oxygen concentration with absorbers. The hexanal data also showed that chips in metallized polyester without absorbers produced significantly more hexanal at an initial oxygen concentration of 2% than at an initial oxygen concentration of 0.2%.

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NOMENCLATURE

a_w	water activity
AU	Area Response Units
BET	Brunauer, Emmett, and Teller
C, c	constants
$^{\circ}\text{C}$	degrees Celcius
cc	cubic centimeters
CCs	Calibration Curve slope
EMC	equilibrium moisture content
ERH	equilibrium relative humidity
$^{\circ}\text{F}$	degrees Farenheit
g	gram
GC	gas chromatograph
H_p	concentration of hexanal in product sample($\mu\text{g/g}$)
IMC	initial moisture content
$L\cdot$	a free radical
LH	any unsaturated fatty acid
$\text{LOO}\cdot$	the peroxy radical
LOOH	a lipid hydroperoxide
m	moisture content (dry basis)
m_1	monolayer value
ml	milliliter
mm	millimeter
μl	microliter
$^1\text{O}_2$	singlet oxygen
$\cdot\text{OH}$	hydroxyl radical
P	product weight
p	partial pressure of water above the sample
p_o	vapor pressure of pure water

P_p	water vapor permeability of each package
PET	polyester
PP	polypropylene
ppb	parts per billion
P_f	final product weight
P_i	initial product weight
ppm	parts per million
PV	peroxide value
R_1, R_2	relative humidity of the external and internal package environments
RH	relative humidity
S	saturation vapor pressure
STP	standard temperature and pressure
TBA	thiobarbituric acid
TBARS	thiobarbituric acid reactive substances
V_i	Volume of injection
V_t	Total sample volume
W_c	weight change
W_d	weight of dry product
W_i	initial weight
W_f	dried weight
WVTR	water vapor transmission rate
$WVTR_p$	water vapor transmission rate for each package

INTRODUCTION

Lipid oxidation is a major limiting factor in the shelf-life of potato chips. The major products of lipid oxidation are hydroperoxides, which are colorless, tasteless, and odorless. Hydroperoxides, however, break down to low molecular weight compounds which impart flavors and odors to food products that are associated with rancidity. The secondary products of oxidation are free radicals, peroxides, epoxides, aldehydes, ketones, cyclic monomers, dimers, and polycyclic aromatic hydrocarbons; many of which are toxic (Bidlack et al. 1973).

Several factors can influence the rate of lipid oxidation. Jeon (1983) reports that factors such as light, relative humidity, temperature, type of frying oil used and availability of oxygen affect the production of lipid oxidation products. Quast and Karel (1971) report that lipid oxidation is likely the most common mechanism of oxygen uptake in dried foods such as potato chips. For fried foods, the extent of oxidation of the frying oil also contributes to the product's oxidation.

Over the years, different products of oxidation have been measured as indices of the extent of lipid oxidation. Peroxide value, thiobarbituric acid reactive substances (TBARS), and hexanal are three that are commonly used.

Oxygen absorbers have long been used to reduce oxygen in the headspace of packages to prolong shelf-life without

adding preservatives. Patents for oxygen absorbers have been granted as early as 1938. There are several different types of oxygen absorbers currently being developed. The most common oxygen scavenger on the market today is an iron complex which oxidizes to rust. These absorbers can be adjusted with humidity factors that will make them usable with a variety of products with low to intermediate water activities. That is the type that will be used in this study.

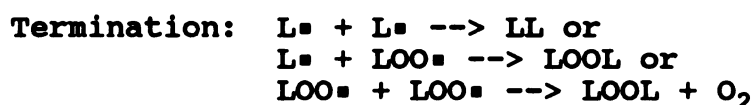
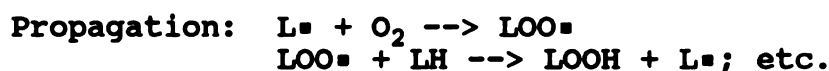
The objectives of this study were to:

1. Determine an initial headspace oxygen concentration above which the rate of lipid oxidation proceeds independent of the oxygen concentration.
2. Determine the extension in shelf-life obtained by using an oxygen absorbing sachet in packages with initial oxygen concentrations of 0%, 2%, and 10%.
3. Determine how using metallized polyester and metallized polypropylene with and without absorbers affects the rate of lipid oxidation.

LITERATURE REVIEW

Mechanism of Lipid Oxidation

The mechanism of oxidation is well documented (Gutteridge and Halliwell, 1990; Nawar, 1985). Lipids oxidize by a free-radical chain process. This process has 3 steps: 1)Initiation, free-radical formation; 2)Propagation, free-radical chain reactions; and 3)Termination, formation of non-radicals.



LH is any unsaturated fatty acid, $\text{L}^\bullet$ is a free radical formed by removing hydrogen from a carbon next to a double bond, $\text{LOO}^\bullet$ is the peroxy radical formed, and LOOH is a lipid hydroperoxide, the major primary lipid oxidation byproduct. In linoleic acid, a hydrogen is removed from the doubly allylic methylene on carbon-11 to create a delocalized pentadienyl radical. Oxygen addition at carbons-9 and -13 produces conjugated 9- and 13-hydroperoxide isomers (Frankel, 1984). Hydroperoxides readily decompose into the many secondary byproducts of oxidation.

Nawar (1985) points out that the initiation reaction in this process has a high activation energy making the

reaction unlikely without a catalyst. Some catalysts of the initiation step are thought to be transition metals, light, decomposing hydroperoxides, and singlet oxygen ($^1\text{O}_2$). Singlet oxygen is also believed responsible for photosensitized oxidation. Singlet oxygen is electrophilic enough to react directly with a double carbon bond, whereas stable (triplet state) oxygen is not (Nawar, 1985). When linoleate is photosensitized by singlet oxygen, four hydroperoxide isomers are formed: conjugated 9- and 13-diene and unconjugated 10- and 12-diene hydroperoxides (Frankel, 1984).

Factors Influencing the Rate of Oxidation

Light, transition metals, temperature, water activity, oxygen availability, antioxidants, and the type and condition of oil all can influence the rate of lipid oxidation in fried foods.

Light

Light has been known to contribute to oxygen uptake and subsequently oxidation for many years. Several have studied the effects of light on lipid oxidation. Quast and Karel (1972) demonstrated that artificial room light or sunlight will increase oxygen uptake by potato chips, and as water activity increases, this effect becomes more significant. A study performed by Columbus Instruments International

Corporation showed that potato chips exposed to a 30 watt lamp light at 43°C consumed oxygen at a rate twenty times greater than potato chips stored in the dark at 22°C (Columbus Instruments International Corporation, 1993).

Jeon and Bassette (1984) monitored n-hexanal production in fluorescent light-exposed potato chips and potato chips stored in the dark. They found that light-exposed potato chips produced moderately higher amounts of n-hexanal than control chips over the first 60 hours of the study, and rapid production of n-hexanal occurred in light-exposed potato chips after 60 hours. Fluorescent lighting is commonly used in grocery stores and emits light in wave lengths between 350 and 500 nanometers. Kail (1984) reports that only blue-printed and metallized structures adequately protect snack foods from acceleration of oxidation by light.

Transition Metals

Transition metals, such as copper and iron, can shorten the induction period and increase oxidation rate. Trace amounts of heavy metals are found in most edible oils. They are picked up from the soil during plant growth or from equipment during processing (Nawar, 1985). Because these transition metals can exist in two or more valency states, they have oxidation-reduction potential and are pro-oxidants. They can act as pro-oxidants in accelerating hydroperoxide decomposition, attacking an unoxidized

substrate to remove a hydrogen and form a free radical, or activating oxygen molecules to the singlet oxygen state.

Temperature

It is well known that increasing temperature increases molecular movement. Thus, logic dictates that a high-temperature storage environment will have oxygen molecules that move quickly and have increased affinity for free radicals in the lipid oxidation process, increasing the oxidation rate. However, Quast and Karel (1972) state that temperature has a weak effect on the rate of oxidation in potato chips, and therefore if accelerated testing is to be done, relatively high temperatures must be used. Berends (1993) found that as oxygen concentration was held constant, greater amounts of hexanal were produced as the temperature increased from 23°C to 40°C to 66°C.

Water Activity

Perishability of foods is strongly related to moisture content. However, different foods with the same moisture content vary greatly in perishability. The difference can be attributed, in part, to how tightly water binds with nonaqueous constituents. Tightly bound water will not support degradative activities, and different foods will allow more or less water to be tightly bound, thereby having different moisture contents at the same level of water

activity (Fennema, 1985). Water activity (a_w) is defined as the partial pressure of water above the sample (p) divided by the vapor pressure of pure water (p_0) at the same defined temperature. This is also equal to the equilibrium relative humidity (ERH) of a food at a given moisture content divided by 100. Water activity is related to moisture content through a sorption isotherm.

The rate of lipid oxidation is greatly affected by a food's water activity. Dried foods and high moisture foods will oxidize at a faster rate than intermediate moisture foods. Oxidation proceeds rapidly at water activities below 0.1, but as water activity increases, oxidation rate decreases. It is believed that this protective effect of water is due to hydrogen bonding of hydroperoxides, deactivation of transition metals, and water interfering with free radicals (Nawar, 1985; Labuza et al., 1970). At water activities between 0.55 and 0.85 the rate of oxidation increases. This is likely due to increased mobilization of catalysts. Several studies of model systems confirmed this and suggested that the high water content also exposed new catalysts sites by swelling the polymeric matrices (Heidelbaugh and Karel, 1970; Heidelbaugh et al., 1971; Labuza et al., 1970; Labuza et al., 1971; Karel and Yong, 1981). At very high water activities catalysts are diluted, thus reducing the oxidation rate. A generalized view of the effect of water activity on the chemical reactions in food

systems is described by the "stability map" given in Figure 1, adopted from Labuza (1971).

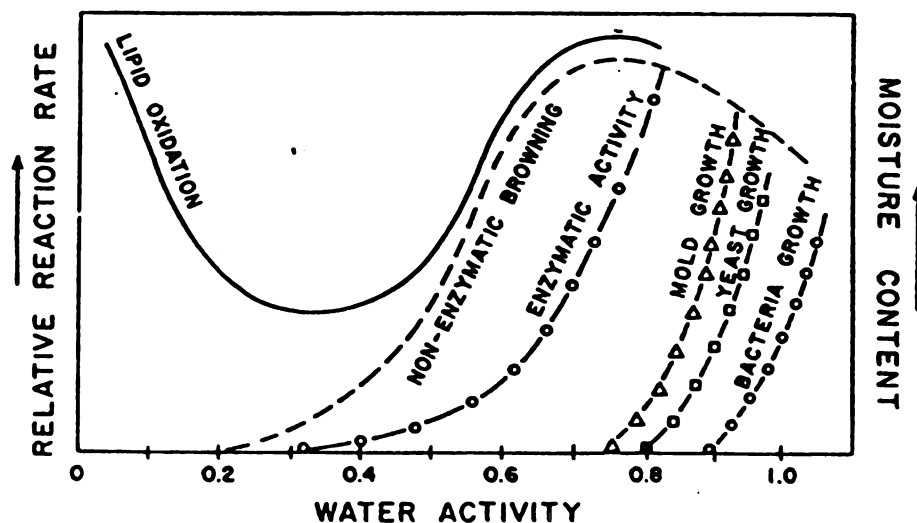


Figure 1: Effect of Water Activity on the Rate of Chemical Reactions in Foods

Monolayer Moisture Content

Minimum reaction rates for degradative processes including oxidation occur at what is called the monolayer water content. Below this monolayer water content the rate of oxidation increases. Therefore, the monolayer water content is the water content which provides the maximum stability of a dried food. The monolayer water content of a food can be computed using the sorption isotherm data and the Brunauer, Emmett, and Teller (BET) equation (Brunauer et al. 1938). The BET equation is

$$a_w/[m(1-a_w)] = 1/m_1c + [(C-1)/(m_1c)](a_w) \quad (1)$$

where a_w equals water activity, m stands for moisture content (dry basis), m_1 is the monolayer value, and C and c are constants. When $a_w/[m(1-a_w)]$ is plotted against a_w for water activities below 0.4, a straight line is generated. From this straight line the monolayer value, m_1 , is found to be $1/(\text{y-intercept} + \text{slope})$. This monolayer water content value will correspond to a specific water activity in the product's sorption isotherm.

Oxygen Availability

The most common mechanism of oxygen uptake in dried foods is probably lipid oxidation as described by Quast and Karel (1971) when studying the rate of oxygen uptake by potato chips fried in sunflower oil with no antioxidants added. They found that in the induction period of oxidation the rate of oxygen uptake is only about one-hundredth of that of chips in the post-induction phase. In 1972, Quast and Karel used 20 g samples of potato chips and found that chips absorb approximately 1200 $\mu\text{l O}_2\text{STP/g}$ during the induction period. The length of the induction period would vary depending on water activity, light, temperature, and frying oil, but the quantity of oxygen absorbed during the induction period appeared independent of these factors. The amount of oxygen absorbed during the induction period was

enough for the chips to become significantly rancid from a flavor standpoint. They also found that oxygen diffusion into potato chips is only likely to be an oxidation rate limiting factor at partial pressures below 0.05 atm except when the relative humidity is also very low.

Antioxidants

Antioxidants are substances that can retard the rate of oxidation or lengthen the induction period. Since food processing oils are exposed to many oxidation catalysts throughout processing, antioxidants are commonly used in these oils specifically for this purpose. The most common action of an antioxidant is accepting free radicals to slow the propagation of oxidation. Cavaletto and Yamamoto (1971) found that adding antioxidants to the frying oil used in roasting macadamia nuts increased the stability of the kernels.

Methods of Quantifying Oxidation

Over the years peroxide value (PV), thiobarbituric acid reactive substances (TBARS), and hexanal have been used to measure the level of oxidative rancidity in food products.

Peroxide Value (PV)

There are numerous analytical procedures for measuring PV. The majority are idometric and they are useful for bulk

lipids and applicable to all normal fats and oils. However, the idometric measurement is highly empirical and any variation in procedure may result in a variation in results (Gray 1991). Other errors that are associated with the idometric measurement are possible adsorption of iodine at unsaturated bonds of fatty acids and liberation of iodine from potassium iodide by oxygen present in the solution to be titrated. Since peroxides are the primary byproduct of lipid oxidation, they are a good measure of oxidation in its initial stage. However, peroxides decompose to secondary oxidation byproducts, making them a poor choice for measurement of oxidation over long periods of time.

Thiobarbituric Acid Reactive Substances (TBARS)

The TBA test was originally meant to measure the level of malonaldehyde (a toxic substance) in a product. However, more substances than just malonaldehyde react with 2-thiobarbituric acid (TBA). Therefore, TBARS is a more accurate description of the measured quantities. Problems associated with TBARS values include the fact that some non-lipids react with TBA and that TBARS can be produced during the testing procedure (Gray, 1991). The assay numbers reported as milligrams of malonaldehyde equivalents per kilogram of sample are also operator dependent and method dependent (Gray and Monahan, 1992).

Hexanal

A third method for measuring rancidity is to measure a volatile product of lipid peroxide decomposition using a gas chromatograph (GC). Melton (1983) concluded that direct quantification of peroxide decomposition products by GC may be more accurate than the TBA test for measuring oxidation. Hexanal is one of the major secondary products of linoleic acid oxidation (Frankel et al., 1981), and the predominant precursor of n-hexanal is free linoleic acid (Matoba et al., 1985). Hexanal was found to comprise the largest proportion of steam-volatile compounds formed by autoxidized potato granules (Buttery, 1961). It was four times more concentrated than any other component and ten times more concentrated than most other compounds. In 1965, it was shown that hexanal was the saturated aldehyde that increased the most during storage of potato chips (Mookherjee et al., 1965) and the flavor produced over the storage period was described as "stale," not rancid.

One concern with measuring hexanal as an indication of oxidation is how well it correlates with sensory data. In 1971, Fuller et al. found sensory evaluation to be more sensitive to oxidation than hexanal measurement. However, in 1981 Tang used a Tenax trapping system with potato chips and found good correlation between diminishing sensory hedonic scores over time and increasing n-hexanal concentrations over time. Fritsch and Gale (1977) found

that rancid odors in low fat foods corresponded to hexanal concentrations of five to ten parts per million (ppm).

Oxygen Scavengers (Absorbers)

For many years, people have been trying to extend shelf-life of food products by removing oxygen from the headspace of packages. As early as 1938 a patent was granted in Finland for keeping food in a closed container with zinc dust, iron powder and some other compounds. Oxygen scavengers allow for extended shelf-life without the use of preservatives. Today oxygen scavengers used in packages can be divided into the five major categories of metal-complex scavengers, non-metal chemical-complex scavengers, photo-sensitive dye scavengers, enzyme scavengers, and synthetic heme-complex scavengers.

Metal-complex Scavengers

Iron is the main active component in most metal-complex oxygen scavengers. Iron is relatively inexpensive, safe, has FDA clearance, has a manipulatable reaction rate with oxygen, and has a much greater affinity for oxygen than most food products (Idol and Wagner). Sulfur produces by products which are difficult to control and impart off-flavors or odors. Aluminum forms a protective skin of oxidized metal. Palladium and platinum are very expensive (Idol and Wagner). The common iron-complexes are generally

contained in sachets which are dropped into the food package. "Ageless" is the name of the oxygen scavenger distributed by Mitsubishi which controls about 70% of the market share (Sacharow, 1991). These sachets are used in Japan and Europe in many products such as bakery goods, precooked pasta, cured or smoked meats, dried foods, nuts, coffee, cheese, and chocolates (Labuza and Breene, 1989).

The amount of iron needed in a sachet is dependent upon the initial oxygen in the headspace, the amount of dissolved oxygen in the food, and the package permeation rate. In general, 1 gram of iron can react with 0.0136 moles of oxygen (STP) which is equal to approximately 300 cc. The chemical reaction is $4 \text{ Fe} + 3 \text{ O}_2 \rightarrow 2 \text{ Fe}_2\text{O}_3$. Headspace oxygen concentrations have been maintained at less than 0.01% using metal-complex oxygen scavengers. The rate of oxygen absorption is dependent on oxygen concentration and humidity.

Many different complexes have been designed to serve specific needs of food products. Multiform Desiccants, Inc. has specifically designed absorbers for use in moist foods ($a_w > 0.65$), dry foods ($0.0 < a_w < 0.7$), and refrigerated foods ($0-5^\circ\text{C}$). Multiform has also introduced a "FreshMax" oxygen absorbing label which adheres to a package inner surface. (Idol and Wagner). Some "Ageless" absorbers are designed to scavenge carbon dioxide as well as oxygen for use in coffee packages.

Studies have shown that these metal-complex oxygen scavengers have been successful in significantly extending the shelf-life of many foods including white bread (Nakamura and Hoshino, 1983), pizza crust, snack foods, fried rice cakes, cheese-filled pasta products (Labuza and Breene, 1989), and some intermediate moisture foods for the space program (Waletzko and Labuza, 1976).

Another metal complex that has received some attention is the mix of PET, MXD6 Nylon, and Cobalt. When combined to create a monolayer container, such as a beverage bottle, the cobalt in the form of a carboxylic acid salt acts as the metal catalyst and MXD6 is the oxidizable component. The system is called "Oxbar" and is processable by the same means as standard PET. Bottles formed, flushed with nitrogen, and stored at room temperature showed no oxygen transmission to the inside in 1.5 years. Other tests with this system on beer, juice, and wine have been successful, but the system is not yet being used commercially although it has FDA approval for use as an additive or blend with PET at levels up to 30% at temperatures below 49°C (Rice, 1990).

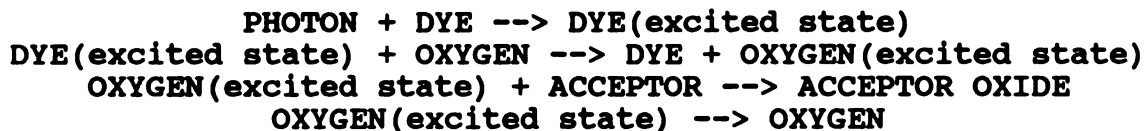
Non-metal Chemical-complex Scavengers

Some non-metal oxygen absorbers have been developed to eliminate the problem of setting off metal detectors on processing lines. They are formed from organometallic molecules that have an affinity for oxygen. Oxygen does not

react with these molecules, but is irreversibly bound to them, so there are no harmful byproducts. They are useful with liquid and semi-liquid products, and can be blended into polymers as a barrier/scavenging layer (Prepared Foods, 1990).

Photosensitive Dye Scavengers

It has been proposed (Rooney, 1981) that the reaction between ground state oxygen and iron complexes is too slow, and that oxygen should be excited to its singlet state for faster scavenging. Photosensitive dyes impregnated on ethyl cellulose film, when illuminated, activate oxygen to its singlet state which then can react with an acceptor to form an oxide. Some tested singlet oxygen acceptors are difuryllidene erythritol (DFE), difurfurylidene-pentaerythritol (PEF), tetraphenylporphine (TPP), dioctyl thallate (DOT), and dimethyl anthracene (DMA). None of these are approved for food contact in the U.S. The reaction scheme follows (Rooney, 1983):



Rubber has been studied, not only as a matrix for holding acceptors, but also as a highly concentrated acceptor in itself. If acceptors approved for food contact can be developed, the advantages of this system would be

that no sachets would need to be added to the packages and the scavenger system would not become active prior to use as long as they are stored in the dark.

Enzyme Scavenger Systems

Glucose oxidase is a known oxidoreductase, transferring two hydrogens from the -CHOH group of glucose to oxygen forming glucono-delta-lactone and hydrogen peroxide. One mole of glucose reacts with one mole of oxygen. However, catalase is a normal contaminant in glucose oxidase and it decreases the effectiveness of glucose oxidase by half. Pure glucose oxidase is very expensive. Glucose oxidase (with catalase) has GRAS (generally regarded as safe) status and can be added to food products such as beer or wine to eliminate dissolved oxygen in the product (Labuza and Breene, 1989). However, the oxidation reaction forms off-flavor by-products which are detectable in beer (Zenner and Salame, 1989). Scott and Hammer (1961) suggested using the glucose oxidase in sachets in dried foods with a humidity factor within the sachet to drive the reaction. They also have a patent for spreading the enzyme in a fine particle matrix throughout food products.

Ethanol oxidase is another enzyme with oxygen scavenging potential. It oxidizes ethanol to acetaldehyde. It has been in use as a breath alcohol analyzer test, but there is no known application for food (Labuza and Breene, 1989).

Synthetic "heme" Scavenger

Aquanautics Corporation has developed oxygen binding "heme" complexes which function well with high water activity food products and CO₂ environments (Zenner and Salame, 1989). The complexes are called LONGLIFE® and their chemical structures mimic that of a heme molecule. The complexes are water soluble and so were necessarily immobilized on silica and other supports. As a fixture of the crown closure, they have been successful in reducing oxygen concentration in a package of aqueous solution from over 2000 parts per billion (ppb) to less than 50 ppb within 24 hours. The absorber need not be in contact with the liquid to be effective.

METHODS

Potato Chip Manufacture

Snowden potatoes were harvested in the fall and stored at 13°C for at least two weeks before processing. Processing potatoes into potato chips consisted of washing the potatoes in cold water, abrasion peeling the potatoes for 30 seconds, slicing the potatoes to 1.5 millimeter-thick slices (± 0.25 mm), rinsing the slices in three batches of fresh, cold water, patting slices dry for over one minute, frying each batch until it stopped bubbling (2 to 3 minutes), and spreading the chips out on paper towel to dry before packaging. Potatoes for an entire day's processing were washed, peeled, and sliced in one batch. Fryer batches were about 210-230 grams each. All potatoes remained submerged in cold water between the stages of initial washing and patting dry for frying. Fully refined soybean oil was used for frying. The soybean oil had added TBHQ and citric acid to preserved stability and methyl silicone to inhibit foaming during frying. Fresh oil was poured for frying at the start of each processing day and oil was added as needed throughout the day. All oil came from the same lot number for all processing days.

Initial Moisture Content

The initial moisture content of freshly fried potato chips was determined using a modified vacuum-oven method. Two to three gram samples (accurately weighed) of fresh, dry chips were placed in aluminum weighing dishes and dried in the vacuum oven under conditions of 30 mm Hg vacuum and 100°C for seven hours. After the vacuum was released, samples were placed in a desiccator until they cooled to room temperature at which time they were weighed. The equation

$$((W_i - W_f) / W_f) \times 100 \quad (2)$$

where W_i = initial weight of chips and W_f = dried weight of chips was used to calculate moisture content on a dry basis. The reported initial moisture content is the average of nine samples.

Sorption Isotherm

A sorption isotherm was developed to determine the equilibrium moisture content (EMC) of the potato chips at different water activities (a_w). Salt solutions were developed using the procedure described in Hygro dynamics Technical Bulletin No. 5 (Creating and Maintaining Humidities by Salt Solutions) and placed in seven tightly sealed, reclosable plastic buckets, creating constant relative humidity environments. The bucket environments equilibrated for two weeks before testing began. Isotherm

data were obtained gravimetrically by measuring product weight change over a two week period in constant temperature and relative humidity. Fresh chips were weighed accurately into tared aluminum weighing dishes on an analytical balance. The samples were then placed into the humidity buckets and weighed at intervals until they reached equilibrium weights for their respective environments. All humidity conditions inside storage containers remained constant as indicated by hygrometer sensors installed in each bucket. Temperature in the storage area was measured at $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Three samples were placed in each humidity bucket and the average EMC is reported.

Packaging

Chips were dried in the open air for 30 to 120 minutes before packaging. Over 30 grams of chips were put into metallized polyester and metallized polypropylene bags. Oxygen absorbers were added to some bags. Bags were vacuumed, flushed with a specialty gas of the desired composition, and sealed on a Smith Super Vac vacuum packager, model GK 165R.

Eight different groups were developed. Four groups had metallized polyester packages. Two of these groups were flushed with nitrogen and two were flushed with a 2% oxygen/98% nitrogen specialty gas from Liquid Carbonic. One group of each atmosphere had an oxygen absorbing label,

capacity 200 cc, dropped into the package. Four groups had metallized polypropylene packages. Two of these groups were flushed with a 2% oxygen/98% nitrogen specialty gas from Liquid Carbonic and two were flushed with a 10% oxygen/90% nitrogen specialty gas from Liquid Carbonic. One group flushed with the 2% oxygen gas had an oxygen absorber of capacity 200 cc dropped into each package and one group flushed with the 10% oxygen gas had an oxygen absorber of capacity 400 cc dropped into each package. The materials, initial oxygen concentrations, and use of absorbers for the eight groups are shown in Table 1.

Table 1
Packaging Characteristics of Different Groups

Group	Material	Initial [O₂]	Absorber
1	metallized polyester	0%	no
2	metallized polyester	0%	yes
3	metallized polyester	2%	no
4	metallized polyester	2%	yes
5	metallized polypropylene	2%	no
6	metallized polypropylene	2%	yes
7	metallized polypropylene	10%	no
8	metallized polypropylene	10%	yes

After sealing, bags were inflated with the appropriate gas supplied by Liquid Carbonic. Gas cylinder regulators were connected to needles by teflon tubing and after flushing the tubing with the appropriate gas, the needles were inserted through septa attached to the bags and the bags were inflated until they were firm. Septa attached to the bags were made of clear, circular, silicone dabs with

radii of approximately 8 millimeters and thickness of approximately 5 millimeters adhered to a section of electrical tape.

Water Vapor Transmission Rate

Water vapor transmission rate was measured gravimetrically. Approximately 100 grams of desiccant was added to three bags of each material. The bags were heat sealed and weighed. Three empty bags of each material were also heat sealed and weighed. Packages were stored at conditions of 72°F and 50% relative humidity. Packages were weighed every two to three days until a constant rate of moisture gain was obtained. The net moisture weight gain was equal to the difference in weight over time of the packages with desiccant minus the difference in weight of the empty, control packages. The net weight gain was plotted as a function of time. The slope of the straight line portion of the graph equals the water vapor transmission rate for each package ($WVTR_p$). The water vapor permeability of each package (P_p) was calculated using the following equation:

$$P_p \text{ (g/package/day/mmHg)} = WVTR_p / [S(R_1 - R_2) / 100] \quad (3)$$

where S equals the saturation vapor pressure at test temperature and R_1 and R_2 equal the relative humidity of the external and internal package environments, respectively.

Chip Weights

The weights of chips prior to packaging and directly after were recorded. The weights of chip packages were taken at one or two week intervals for the first few months. Weights of chip packages were taken just prior to destruction for hexanal measurement. This allows measurement of the moisture content of the chips over time, which would correspond to their water activity level over time.

Volume Measurement

The volume of each inflated bag was measured by submersion in water. Each bag was submerged and the water displaced ran into a graduated cylinder for measurement. The volume of each bag was measured within the first 24 hours after packaging and at the time of destruction.

Headspace Oxygen Concentration

Headspace oxygen concentrations were measured with an Illinois Instruments Inc. model 3500 headspace oxygen analyzer. A sampling needle connected to the instrument by a tube was inserted through the septum into each bag. The bag was squeezed to produce a flow rate of approximately 0.05 liter/minute and the oxygen concentration was displayed by the instrument. The oxygen concentration of the headspace was measured on all bags within the first 24 hours

after packaging and at the time of destruction. Selected bags from each of the groups with oxygen absorbers were sampled for headspace oxygen concentration periodically over the first few weeks and not used for any other purpose.

Storage

All bags were stored hanging by clothespins on large wooden drying racks in an environmentally controlled room. The bags were hung by the material outside of their seams so that the pouch part of the bags did not touch other bags or the rack. The storage environment was monitored using a portable hygrometer. Temperature and relative humidity were constant at $73^{\circ}\text{F} \pm 2^{\circ}\text{F}$ and $50\% \text{ RH} \pm 2\%$. Light exposure was variable in the storage area. The storage arrangement is shown in Figure 2.

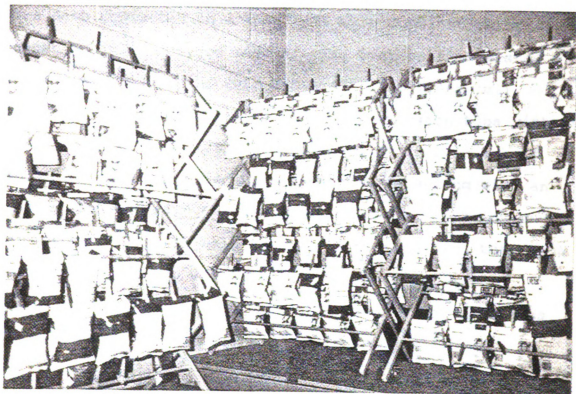


Figure 2: Storage Arrangement of Potato Chip Packages

Hexanal Quantification

Apparatus for Trapping of Volatiles

A gas flushing/volatile trapping system was designed. A cylinder of compressed nitrogen was connected to 3 flow meters through a series of copper tubing. The flow meters were each connected to a test cell by a combination of copper tubing and tygon tubing. The test cell consisted of a modified gas washing tube and a 450 milliliter (ml) Erlenmeyer flask modified to fit the dispersion tube without leaking. The exit tube of the dispersion tube is a ball joint. A pyrex cylinder trap 11 centimeters long with an inside diameter of 0.6 centimeters and a socket joint end was connected to the ball joint of the dispersion tube by a spring-loaded clamp which held the joint tight so no volatiles would be lost to the atmosphere. The apparatus without the trap attached is shown in Figure 3.

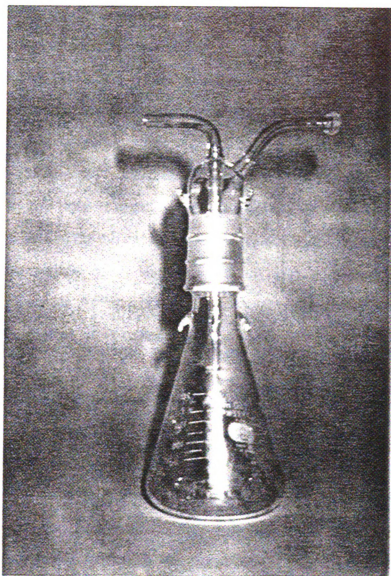


Figure 3: Apparatus for Trapping of Volatiles

Trapping of Volatiles

Approximately 10 grams of chips weighed accurately were placed in the test cell. The test cell was closed tightly and the trap connected. Each trap was packed with four grams $\pm$ 0.1 grams Tenax-GR (80/100 mesh) between wadded pieces of glass wool. The test cell was then placed in a water bath and covered with aluminum foil to block out light. Nitrogen was flushed through the test cell and Tenax trap at an approximate rate of 25 cubic centimeters (cc) per minute for 22 hours. The test cell was flushed at room temperature for the first hour to remove oxygen from the cell before heating to minimize further oxidation. After one hour the water bath was turned on and allowed one hour to equilibrate to 70°C. The water bath remained at 70°C for the next 20 hours of flushing.

Extraction and Concentration Procedure

The extraction and concentration procedure was developed by Berends (1993). One microliter (μ l) of HPLC grade 2-methylbutane (Aldrich Chemical Co., Milwaukee, WI) was injected into the gas chromatograph to ensure its purity before it was used to wash the hexanal from the pyrex traps. The Tenax traps were placed into a single hole cork stopper that was placed into the end of a 25 ml graduated centrifuge tube. Using disposable transfer pipettes, 1 ml of 2-methylbutane (isopentane) was pipetted into the socket end

of the Tenax trap. The centrifuge tube was then placed in a centrifuge to accelerate the extraction of hexanal from the Tenax. The centrifuge (International Equipment Co., Boston, MA) was set at 500 revolutions per minute for 2 minutes, forcing solvent containing hexanal to the bottom of the graduated centrifuge tube. A 1.0 - 1.5 ml aliquot of isopentane was pipetted into the Tenax trap and it was centrifuged again. This was repeated a third time. Three to four ml of extractant were collected in the bottom of the centrifuge tube. Nitrogen was used to concentrate the hexanal by evaporating the extractant to a volume of 1.0 ml. This enabled trace amounts of hexanal to be detected by the gas chromatograph. The one ml in the centrifuge tube was quickly transferred to a 1.8 ml Supelco Screw Cap Vial with a Hole Cap and Septum. The vials were stored in the freezer to prevent evaporation of the isopentane and concentration of the sample. After hexanal was removed, Tenax traps were rinsed with isopentane, centrifuged, and baked in a 100°C oven for more than 12 hours. Tenax was reused four times and then removed. When Tenax was removed the glass traps were washed, rinsed with isopentane, dried in a 100°C oven, cooled, and refilled with fresh Tenax-GR and conditioned.

**Percent Recovery of Hexanal From Tenax
and Concentration Technique**

A recovery study was done to determine the percentage of hexanal that is recoverable from the extraction and concentration procedure. Two solutions of hexanal in isopentane were made and tested in duplicate to determine the percent recoverable.

A 0.9 μ l aliquot of each solution was injected into the programmed GC in triplicate. An average area response was used as a basis for 100% recovery.

One milliliter of each solution was injected into a pyrex trap packed with freshly conditioned Tenax-GR. Then the extraction and concentration procedure was followed. A 0.9 μ l aliquot of each extract was injected into the programmed GC. Injections were done in triplicate. The area responses of the three injections for each extract were averaged. The averages were divided by the area response set as a basis for 100% recovery for that solution to determine the percent recovery using this technique.

Gas Chromatography

A 5 μ l syringe (Hamilton Co., Reno, NV) was placed in a freezer to cool. 0.9 μ l aliquots of sample were injected with the cooled syringe into a Hewlett Packard gas chromatograph (GC), model 5890, equipped with a 60 meter Supelcowax 10 capillary column and a flame ionization

detector (FID). Standard solutions of hexanal in acetonitrile were made prior to injection of samples to ensure consistency of the GC's response. A Hewlett Packard 3392A integrator was interfaced with the GC. The conditions of the GC were programmed at an initial temperature of 40°C for one minute followed by heating at a rate of 5°C per minute to a final temperature of 150°C which was held for 10 minutes. The injection port temperature was 250°C. The range was set at 2 and the attenuation at 0.

Hexanal Calibration Curve Development Procedure

The standards made up for the calibration curve consisted of hexanal and acetonitrile (used due to low boiling point of isopentane). Volumetric flasks used for the procedure were washed, rinsed with distilled water, rinsed again with acetonitrile, and dried in a 100°C air oven. While the flasks were drying, the purity of the acetonitrile was evaluated using the GC. The GC conditions were programmed for an initial temperature of 40°C to be held for 1 minute and then increasing to a final temperature of 150°C at a rate of 5°C per minute. The final temperature was held for 10 minutes. The injection port temperature was 200°C and the detector temperature was 250°C. The range was set at 2 and the attenuation was zero.

Three 0.9 μ l injections of acetonitrile were made into the GC using the same 5 μ l syringe. No peaks near the

retention time of hexanal were observed. After flasks were dry, they were removed and cooled to room temperature and labeled with their appropriate concentrations.

0.01 grams of hexanal were added to a 100 ml volumetric flask providing an initial standard solution with a hexanal concentration of 100 parts per million (ppm).

5 ml of 100 ppm solution were added to 5 ml acetonitrile in a 10 ml volumetric flask for a concentration of 50 ppm. 2 ml of 100 ppm solution were added to 8 ml acetonitrile in a 10 ml volumetric flask for a concentration of 20 ppm. 2.5 ml of 100 ppm solution were added to 22.5 ml acetonitrile in a 25 ml volumetric flask for a concentration of 10 ppm. 2 ml of 100 ppm solution were added to 48 ml acetonitrile in a 50 ml volumetric flask for a concentration of 4 ppm.

0.9 μ l injections into the GC were made in triplicate from each flask using the same 5 μ l Hamilton syringe. After each injection, the syringe was washed with acetone, and placed in a 100°C oven for 10 minutes to evaporate all residual solvent. The results from the three injections at each concentration were averaged and the average values were plotted. During the course of this study, the Supelcowax column was altered. Therefore, different calibration curves were created from the same standard solutions before and after altering the column. Appendix A contains the data and plots for both calibration curves.

RESULTS AND DISCUSSION

Product Model

Potato chips fried in soy bean oil were packaged in metallized polyester and metallized polypropylene bags with and without oxygen absorbers and with varying initial oxygen concentrations. Potato chips are around 30 - 45% fat by weight (Orr and Cash, 1991) and soy bean oil is approximately 54% linoleic acid by weight. As mentioned earlier, linoleic acid is the predominant precursor of n-hexanal (Mantoba et al., 1985). This makes the product model around 16 - 24% linoleic acid which is the approximate oxidizing substrate concentration. The initial moisture content was experimentally determined and an equilibrium sorption isotherm was developed to determine the relationship between a_w and the product model. The water vapor transmission rate, water vapor permeability, and the oxygen permeability of the two package materials were tested and calculated.

Storage Environment

The temperature of the storage environment during the study normally fluctuated between 72 and 74°F. The temperature did reach a high of 78°F and a low of 66°F, but these temperatures were only sustained for brief periods. The relative humidity of the storage environment fluctuated

between 35 and 64%. These lows and highs were not maintained for more than 2 weeks at a time, and for the majority of the time, the relative humidity was near 48%.

Initial Moisture Content

The initial moisture content was determined using the following equation:

$$IMC = (W_c/W_d) * 100 \quad (4)$$

where W_c = weight change (grams)
 W_d = weight of dry product (grams)

Initial moisture content was 1.52g H₂O/100 g dry product weight. Data and calculations are in Appendix B.

Equilibrium Sorption Isotherm

From the constant weight (average of triplicate weighings) that was obtained in each of the relative humidity conditions, the equilibrium moisture content was calculated according to the following equation:

$$EMC = [P_f(1+IMC)/P_i] - 1 * 100 \quad (5)$$

where: P_f = final product weight
 P_i = initial product weight
EMC = equilibrium moisture content
IMC = initial moisture content

The sorption isotherm data are in Appendix C. Equilibrium moisture contents at the seven relative humidity environments are in Table 2.

Table 2
Equilibrium Moisture Contents at Each Water Activity

<u>Water Activity</u>	<u>Equilibrium Moisture Content</u>
0.10	3.16
0.21	3.76
0.32	5.46
0.41	5.98
0.51	7.89
0.71	13.29
0.83	23.26

A graph of the experimental sorption isotherm data is shown in Figure 4.

SORPTION ISOTHERM

Fresh Potato Chips at 72F

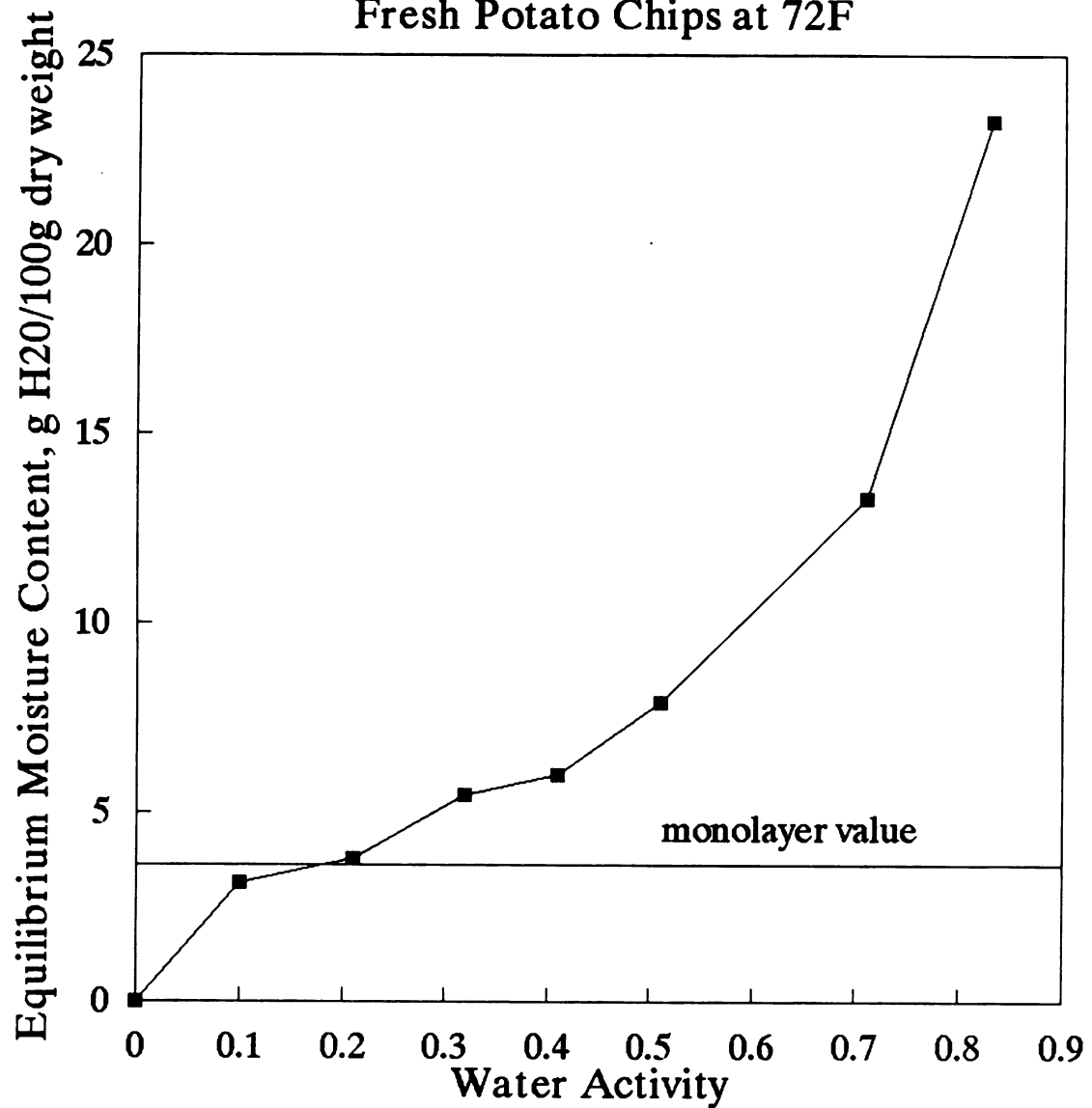


Figure 4: Sorption Isotherm

Using the Brunauer, Emmett, and Teller (BET)
Monolayer Mathematical Model to Predict Product
Moisture Content at Specific Water Activities

The equilibrium sorption isotherm describes the water sorption characteristics of the product. The shape of the curve is a function of the sorption properties of the product. The resultant curve is usually sigmoidal in shape, and can be described by the BET equation among others. From the equilibrium sorption isotherm data, this mathematical model described a linear relationship between water activity and the product equilibrium moisture content up to a water activity of 0.41.

Using Lotus 1-2-3, a statistical analysis was performed for the BET model and a correlation coefficient was calculated. The correlation coefficient estimated the degree of fit between the BET mathematical model and the experimental sorption isotherm. The constant in the BET model was calculated from the linearized form of the equation allowing for determination of the water activity at any product moisture content. The resultant linearized form for the BET model is

$$a_w/(m(1-a_w)) = 1/M_1C + a_w(C-1/m_1C) \quad (1)$$

and the correlation coefficient is 0.9931. The predicted equilibrium sorption isotherm values using the BET model versus the experimental values are given in Table 3.

Table 3
BET Model Expected Values (X_e) versus Actual Equilibrium
Product Moisture Content Values

<u>Water Activity</u>	<u>Experimental EMC</u>	<u>(X_e)</u>	<u>% Difference</u>
0.1	3.16	3.28	3.66
0.21	3.76	4.36	13.76
0.32	5.46	5.35	-2.06
0.41	5.98	6.31	5.23
0.51	7.89	7.73	-2.07
0.71	13.29	13.31	0.15
0.83	23.26	22.88	-1.66

Brunauer, Emmett, and Teller Monolayer Value

Using the data from the equilibrium sorption isotherm and the B.E.T. equation, the monolayer value was determined. The B.E.T. plot of a_w (x-axis) versus $a_w/M_{eq}(1-a_w)$ (y-axis) shown in Figure 5, yielded the linear regression equation:

$$a_w/M_{eq}(1-a_w) = a_w(0.2722) + 0.0050 \quad (6)$$

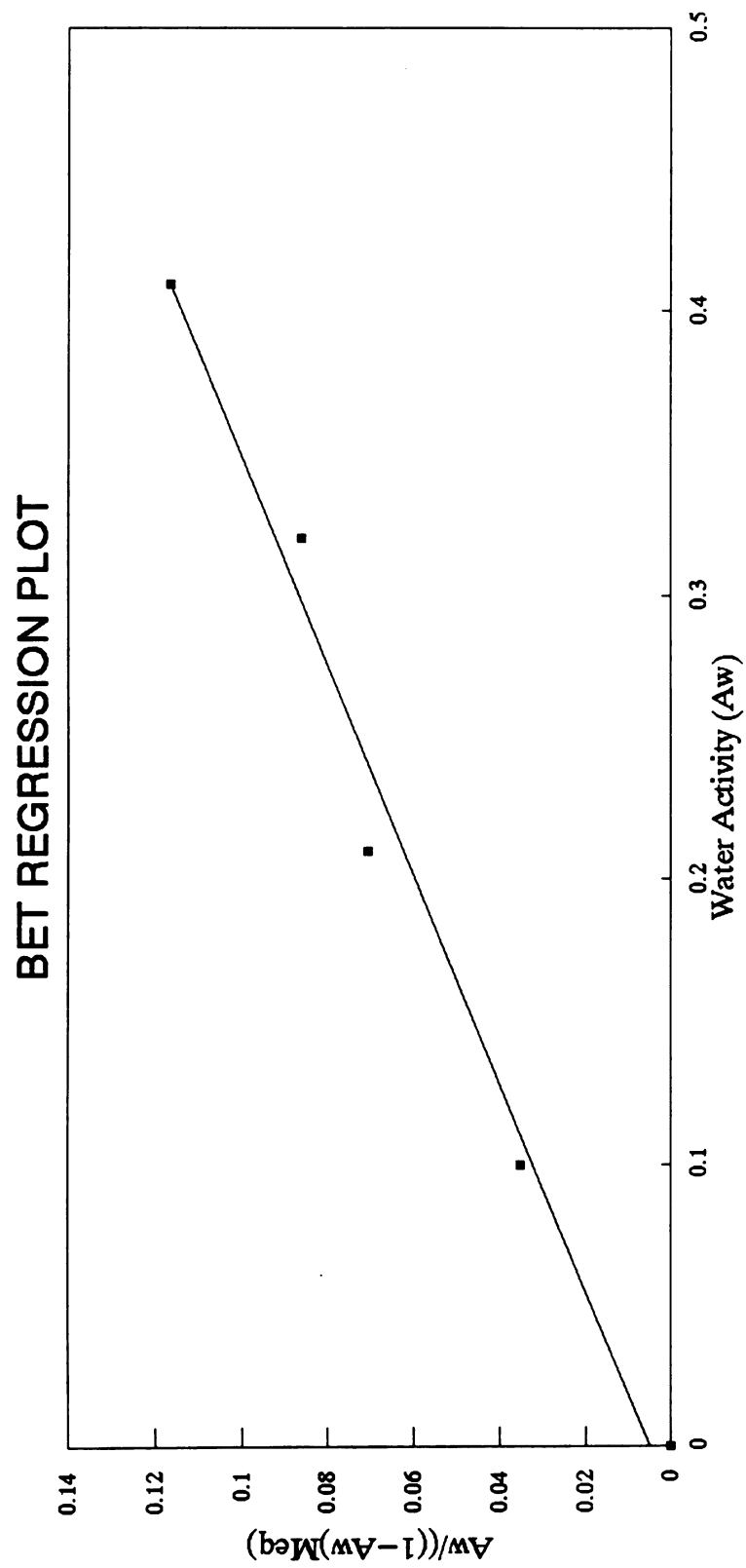


Figure 5: Brunauer, Emmet, and Teller Plot for Determining Monolayer Value

Calculated x- and y-axis values for the B.E.T. plot are given in Table 4.

Table 4 B.E.T. Regression Plot Values for the Sorption Isotherm for Determining Monolayer Value		
Water Activity	x-axis a_w	y-axis $a_w/M_{eq}(1-a_w)$
.41	.41	.1164
.32	.32	.0862
.21	.21	.0706
.10	.10	.0351

Using the slope and y-intercept values from the B.E.T. regression equation, the monolayer moisture content was determined using the following formula:

$$\text{Monolayer Value} = 1/(\text{y-intercept} + \text{slope})$$

The monolayer value was calculated to be 3.608g H₂O/100g dry product. The water activity corresponding to this value found from the sorption isotherm data is 0.197.

Water Vapor Transmission Rate and Permeability

The water vapor transmission rates (WVTR) of the two test packages were found to be 0.002707g/(day*package) for metallized polypropylene and 0.01584g/(day*package) for metallized polyester. Appendix D contains the individual weights of the desiccant and empty packages over time. Appendix E contains the individual weight gains of the desiccant and empty packages over time. The graph of the net weight gain as a function of time is shown in Figure 6.

AVERAGE WEIGHT GAIN OVER TIME OF PACKAGES Storage at 72F and 50%RH

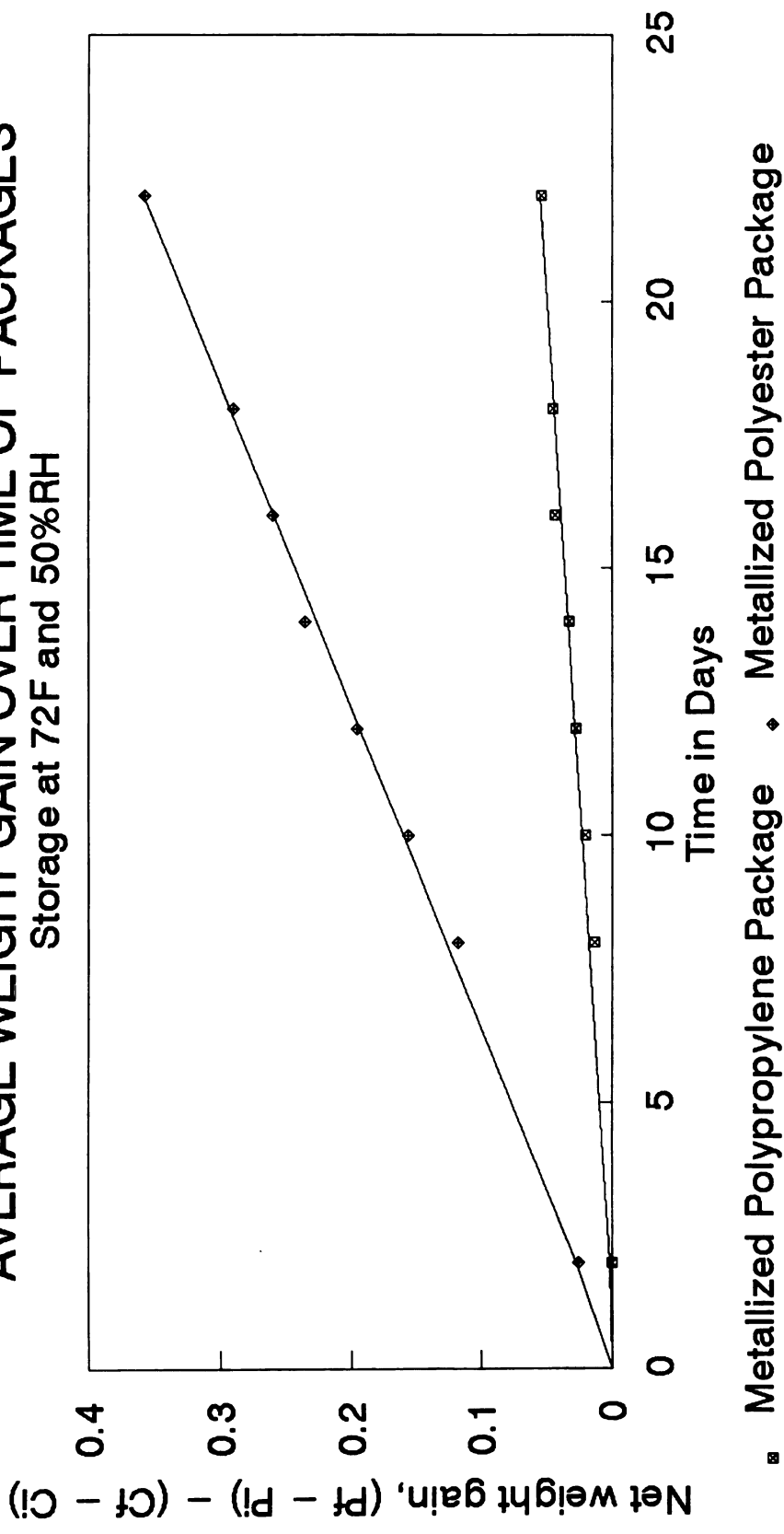


Figure 6: Moisture Weight Gain Over Time of Packaging Materials

A regression analysis was done on the straight line portion of the curves to determine the slopes and consequently the WVTR's. The data used in the regression analyses and the output of the analyses are shown in Table 5. The permeability constant for the metallized polypropylene package was found to be $0.000253\text{g}/(\text{day}\cdot\text{mmHg}\cdot\text{pkg})$. The permeability constant for the metallized polyester package was found to be $0.001572\text{g}/(\text{day}\cdot\text{mmHg}\cdot\text{pkg})$.

Table 5		
Water Vapor Transmission Rate Data		
Time in Days	Moisture Weight Gain of Metallized Polypropylene	Moisture Weight Gain of of Metallized Polyester
0	0	0
2	0.000067	0.025767
8	0.013533	0.117833
10	0.020400	0.156400
12	0.027233	0.195567
14	0.033200	0.235000
16	0.043600	0.260467
18	0.044800	0.290500
22	0.053700	0.358100

Line of Regression Output		
	<u>Polypropylene</u>	<u>Polyester</u>
Slope (WVTR g/(day pkg))	0.002665	0.016556
y-intercept	-0.00392	-0.00545
Correlation Coefficient (R^2)	0.978907	0.98097

Chip Weights

The moisture weight gain of the potato chips in packages was measured gravimetrically. Using the initial moisture content average, the isotherm, and B.E.T. regression data, the moisture content of the chips over time and the corresponding water activities of the chips can be

calculated. A graph of the moisture content over time of chips packaged in metallized polypropylene and metallized polyester is shown in Figure 7. The weights of a representative sample of chip packages are reported in Appendix F. The weight gains of these chips over time are reported in Appendix G.

The actual moisture contents of chips packaged in metallized polyester and metallized polypropylene were compared to computer generated expected values for moisture contents. The expected values were computed from a linear shelf-life model using the following inputted data:

1) temperature of 21°C, 2) IMC of 1.58% dry basis, 3) ERH of 5% for IMC, 4) MC at spoilage of 5.46%, 5) ERH of 32% at spoilage MC, 6) product weight of 31 g, 7) package area of 84 in², 8) storage RH of 48%, and the water vapor permeability coefficients for each material. The computer predicted shelf lives of 179 and 1111 days for chips in metallized polyester and metallized polypropylene respectfully, if moisture content is the limiting factor. Figure 8 shows the actual and predicted values.

The predicted values were low for the first 120 days and then high after that for the metallized polyester package. However, the linear shelf life model was very close to the actual for the metallized polypropylene package and may be useful in predicting the moisture content of unsalted potato chips in metallized polypropylene.

MOISTURE CONTENT OVER TIME

Potato Chips Stored at 73°F; 50% RH

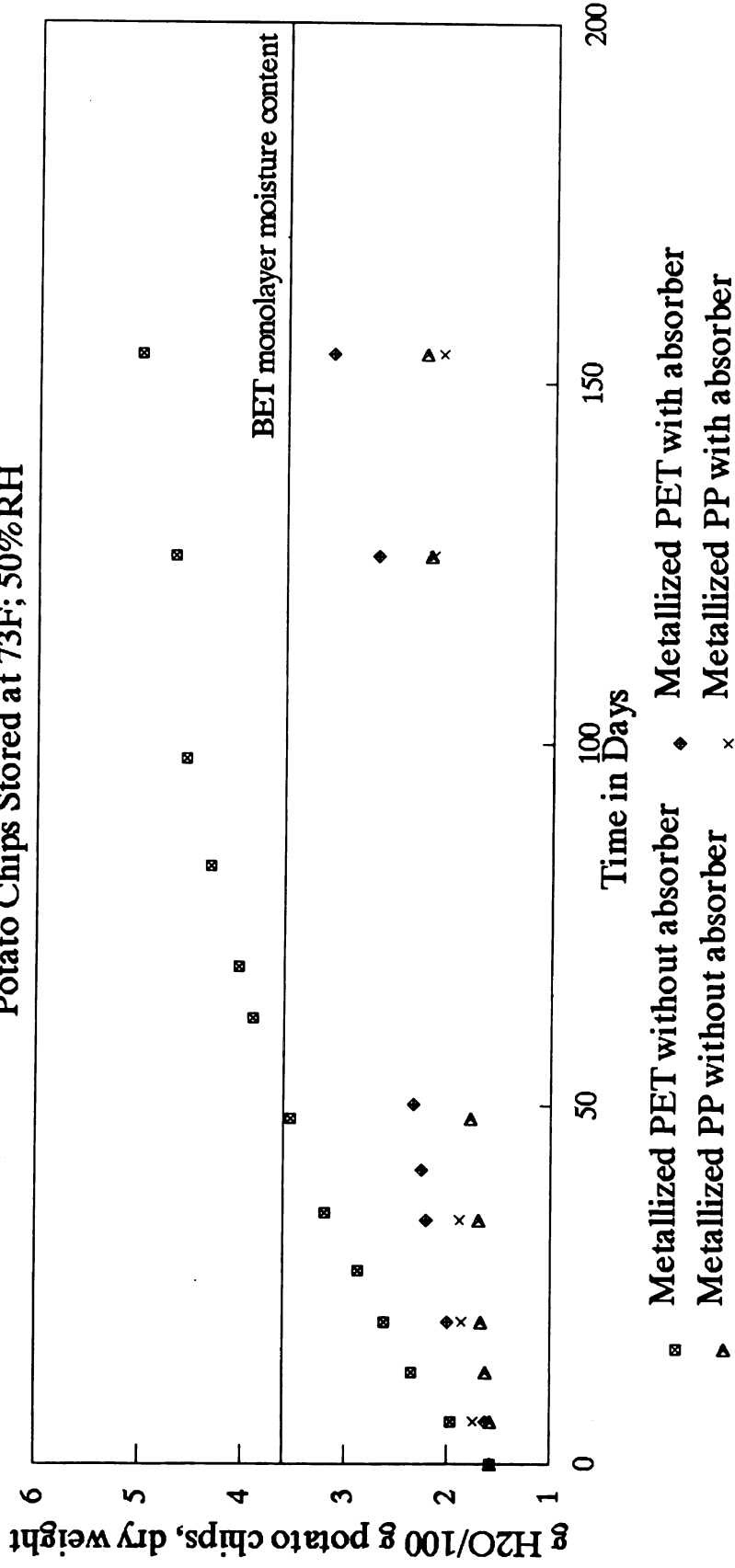


Figure 7: Chip Moisture Content Over Time

MOISTURE CONTENT OVER TIME

Actual Values vs. Linear Shelf Life Model Expected Values

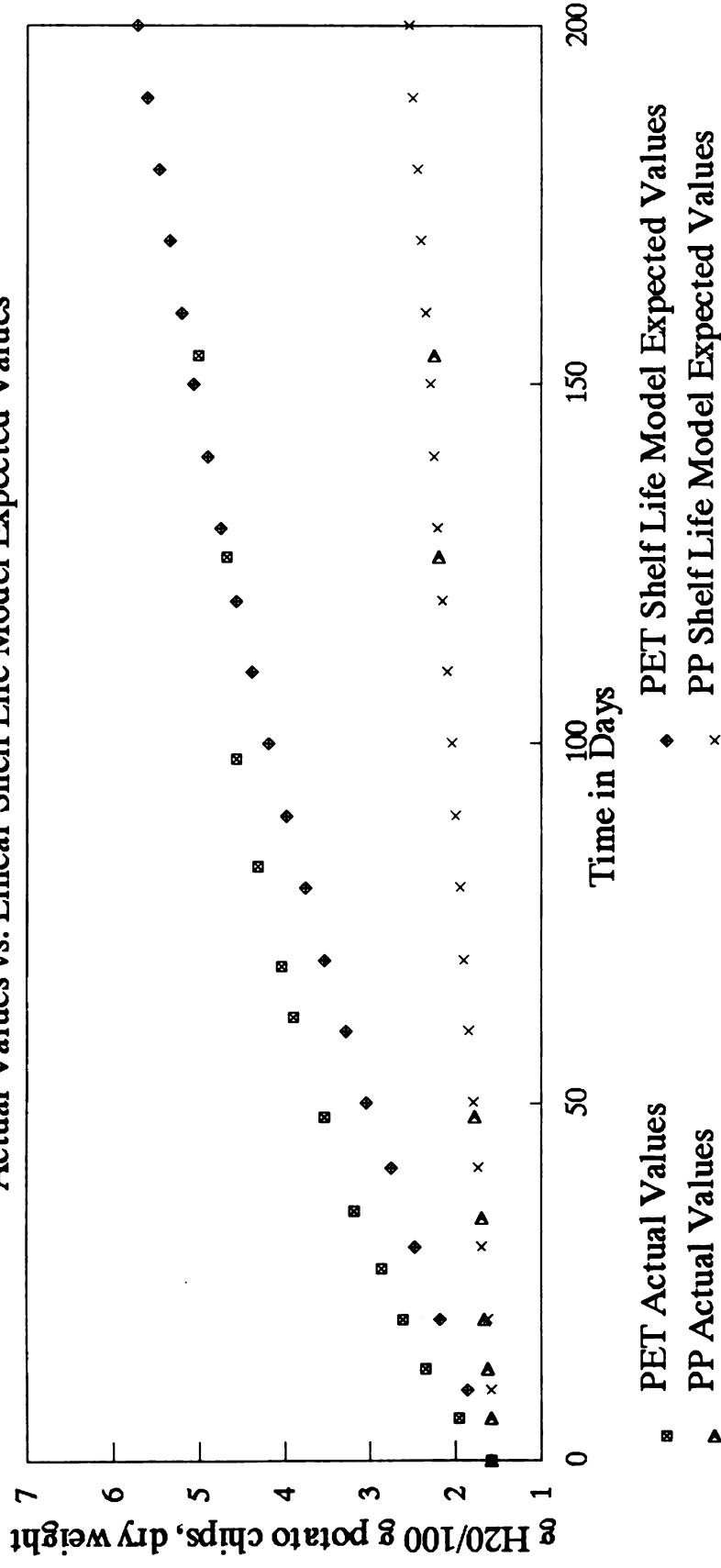


Figure 8: Chip Moisture Content Over Time; Actual Values vs. Predicted Values

Volume Measurement

The volumes measured are only accurate to ± 5 ml. As the initial volume measurement methodology was not tightly controlled, the volumes recorded as the initial volumes are less accurate than the final volumes. The volume data does show that even though the packages were inflated to firmness (pressurized), they did not lose volume over time. Initial and final volume measurements of bags are given in Appendix H.

Oxygen Permeability

The oxygen permeability of the two films, metallized polyester and metallized polypropylene, were measured using the mocon OX-TRAN 200. The oxygen permeability of the metallized polyester was measured at $0.6374 \text{ cc}/100\text{in}^2/\text{day}$ and the oxygen permeability of the metallized polypropylene was measured at $1.568 \text{ cc}/100\text{in}^2/\text{day}$.

Data Collection and Analysis of Headspace Sampling

Headspace Sampling of Oxygen

For the groups with oxygen absorbers, headspace oxygen content was sampled daily until a low point was reached. All groups were sampled for headspace oxygen concentration upon destruction of bags for hexanal quantification. Figures 9, 10, 11 and 12 show the oxygen concentrations of the eight groups over time. The oxygen concentration data

over time for the eight groups is reported in Appendix I. For packages with absorbers, the data shows that as the initial oxygen concentration is raised, the time it takes for a package to reach a headspace oxygen concentration of 0% is shortened (assuming that the absorber capacity is adequate).

HEADSPACE OXYGEN CONCENTRATION OVER TIME

Metallized Polyester Packages Initially Flushed with Nitrogen

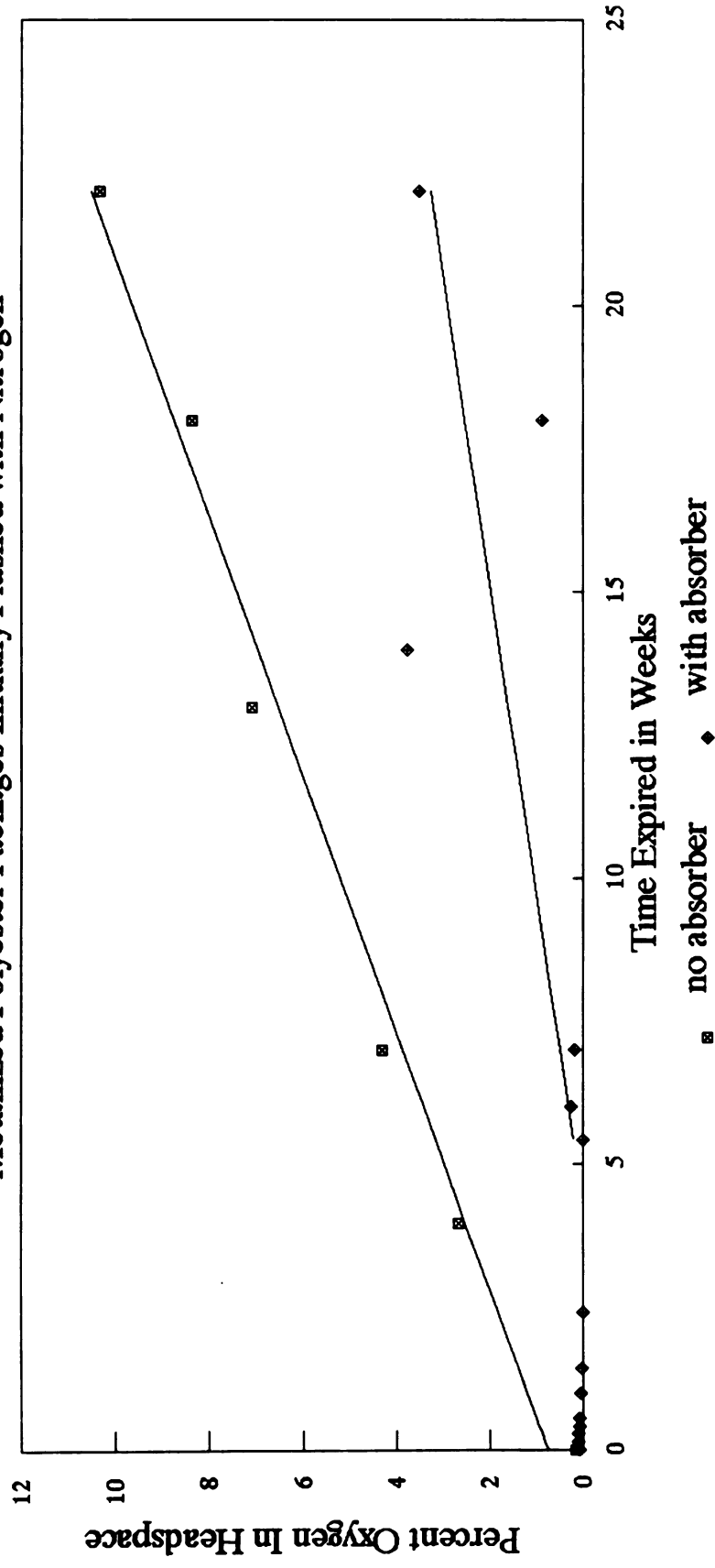


Figure 9: Headspace Oxygen Concentration Over Time
Metallized Polyester Packages Initially Flushed with Nitrogen

Collection of Hexanal from Potato Chips

Packages of potato chips were destroyed for hexanal sampling at times judged appropriate according to the initial headspace oxygen concentration. Three bags of potato chips were be sampled at a time.

Hexanal Data and Quantification

Using gas chromatography, area units of hexanal were obtained from each sampling of chips in duplicate. The averaged area units were then used to quantify hexanal concentration for each potato chip sample. Total grams of hexanal per sample were quantified by substitution into the following equation:

$$H_p = AU / (CCs \times V_i \times P) \times V_t \quad (7)$$

where:

- H_p = concentration of hexanal in product sample ($\mu\text{g/g}$)
- AU = Area Response Units
- CCs = Calibration Curve slope
- V_i = Volume of injection
- P = Product weight
- V_t = Total sample volume

Table 6 shows the average concentrations of hexanal in the potato chips over time in micrograms/gram (ppm). Appendix J contains the hexanal data.

Table 6
Hexanal Concentration in Potato Chips Over Time (ppm)

Time in weeks	Metallized Polyester			
	0% Initial O ₂ w/o absorber	w/absorber	2% Initial O ₂ w/o absorber	w/absorber
4	0.2859			
6		0.3217	0.4167	
7				0.2546
12			0.5006	
13	0.3536			
22	0.4264	0.5344	0.6585	0.5617

Time in weeks	Metallized Polypropylene			
	2% Initial O ₂ w/o absorber	w/absorber	10% Initial O ₂ w/o absorber	w/absorber
2	0.2778		0.2248	
4	0.2920			
6				0.3235
8		0.3628		0.3364
10	0.3568		0.4968	
14			0.3946	
16			0.4783	
22	0.6212	0.4994	0.6277	0.3423

In normal distribution, potato chips have a shelf life of around 40 days (Keener, 1994). This would indicate that the hexanal levels would be above 5ppm at that time (Fritsch and Gale, 1977). The hexanal concentrations measured in this study were low compared to what was expected. The following factors were contributors to these low values. The storage environment was mild, with a low relative humidity. Only the potato chips in metallized polyester packages reached the monolayer moisture content. This means that the rate of oxidation was highest initially and slowed throughout the study for all other potato chip packages. The potato chips were fried in fresh oil with TBHQ

antioxidant added. In industry, oil that has been "seasoned" is used to maintain a consistent product and to extend the usable life of the oil (Paradis, 1993).

Percent Recovery of Hexanal from Tenax-GR

The percent recovery of hexanal from the Tenax-GR was determined to be 91.2%. The data and calculations are given in Appendix K.

Statistical Difference Between Groups

According to Hexanal Data

The hexanal production at the 22 week sampling interval was compared between groups using a t-test. The t-test was performed on a computer software program called Minitab. At the 95% confidence level, chips packaged in metallized polypropylene at an initial oxygen concentration of 10% with an oxygen absorber produce less hexanal than chips packaged in metallized polyester at an initial oxygen concentration of 2% without an oxygen absorber.

At the 90% confidence level, chips packaged in metallized polypropylene at an initial oxygen concentration of 10% with an oxygen absorber produce less hexanal than chips packaged in metallized polyester at an initial oxygen concentration of 0.2% without an absorber. Also at the 90% confidence level, chips packaged in metallized polyester at an initial oxygen concentration of 0.2% without an oxygen

absorber produce less hexanal than chips packaged in metallized polyester at an initial oxygen concentration of 2% without an absorber.

No other significant differences could be found between the groups at the 22 week sampling interval. A possible explanation for this is that the range of data points within each group produced a large standard deviation making it difficult to determine significant differences between groups. A reason for the large range within groups could be attributed to differences in potato chip packages such as pinholes, seal quality, actual absorber capacity, and amount of handling.

The metallized polypropylene packages with absorbers at the initial oxygen concentration of 10% had significantly lower hexanal values than groups packaged in metallized polyester at 0% and 2% initial oxygen concentrations without absorbers. It is possible that this is due to the rapid reduction to and maintenance of 0% oxygen in the headspace of the polypropylene packages with oxygen absorbers. While both of the polyester groups without absorbers started at low oxygen concentrations, the oxygen concentration steadily increased over the 22 week period allowing oxidation to occur. The polypropylene package with the absorber eliminated headspace oxygen quickly, thus terminating any oxidation reactions. Another factor that is relevant to the higher production of hexanal in chips packaged in polyester

compared to chips packaged in polypropylene is the moisture content of the chips. Chips packaged in polyester reached their protective monolayer moisture content after about 7 weeks of storage and were well above this moisture content at 22 weeks allowing increased rates of oxidation. Chips packaged in polypropylene increased in moisture content throughout the study, but remained below their protective monolayer moisture content for the entire 22 weeks, indicating continually decreasing oxidation rates.

The fact that the chips packaged in polyester at 0.2% initial oxygen concentration without absorbers produced significantly less hexanal at 22 weeks than chips packaged in polyester at 2% initial oxygen concentration without absorbers shows that at 2% headspace oxygen, the stoichiometry allows the oxidation reaction to occur. However, at 0.2% oxygen, the oxidation reaction is severely suppressed.

The polypropylene packages at 10% initial oxygen concentration with absorbers are the only packages with absorbers that showed a significant advantage over other packaging methods. This demonstrates the importance of rapidly reducing the headspace oxygen concentration to near 0% and maintaining the oxygen-free atmosphere. It is important to note that all of the packages with absorbers and initial oxygen concentrations of 10% reduced to an oxygen concentration of 0 ppb, and remained there for the

duration of the study. Therefore, the oxygen absorbers of this group had excessive capacity. The oxygen concentrations of packages within other groups with absorbers varied greatly. This indicates that not all absorbers had the same capacity. If all of the absorbers in all of the groups had excessive oxygen sorption capacity, the oxygen concentrations and hexanal production over time within groups may have deviated less providing better statistical data.

Sensory Evaluation

An untrained, inexperienced panel of 3 persons sensory evaluated potato chips from all eight groups after 18 weeks of storage. The panel was in agreement on the flavors and odors of each group of chips. The worst chips were decidedly rancid with a strong, foul odor. The best chips were somewhat stale, but a rancid flavor was not detected. From the best-tasting to the worst-tasting chips, the ranking was as follows:

BEST:

Metallized Polyester; 0% Initial O₂; with absorber
Metallized Polypropylene; 10% Initial O₂; with absorber

MODERATE:

Metallized Polypropylene; 2% Initial O₂; with absorber
Metallized Polypropylene; 2% Initial O₂; without absorber

WORSE:

Metallized Polyester; 2% Initial O₂; without absorber
Metallized Polypropylene; 10% Initial O₂; without absorber

WORST:

Metallized Polyester; 2% Initial O₂; with absorber
Metallized Polyester; 0% Initial O₂; without absorber

The sensory results agreed somewhat with the statistical analysis. The chips packaged in metallized polypropylene with oxygen absorbers at 10% initial headspace oxygen were determined to be less rancid than chips in polyester without oxygen absorbers at 0.2% and 2% initial oxygen concentrations in both analyses. This is consistent with the argument given above. A discussion of the inferences that might be drawn from some similarities and differences between the sensory and analytical results is provided in the following section.

Discussion of Hexanal and Sensory Results

While the sensory evaluation was too limited for reliable statistical conclusions, the consistency of the sensory and hexanal results provides some basis for comparison and discussion.

An important observation made by the sensory panel was that the chips packaged at 0% initial oxygen concentration in polyester with an absorber were the least rancid followed by the chips packaged at 10% initial oxygen concentration in polypropylene with an absorber. The hexanal data showed no statistical difference between these two groups. This may be attributable to the large range of the hexanal values in the 0% initial oxygen concentration group. The hexanal data does show that the 10% initial oxygen concentration group produced less hexanal than the 0% initial oxygen

concentration group for all samples at 22 weeks. This does not necessarily mean that the hexanal and sensory data do not correlate. The sensory analysis was done on packages sampled at 18 weeks and the hexanal data was taken at 22 weeks. This gave the 0% initial oxygen concentration group four weeks to increase the headspace oxygen concentration from less than 1% to 3.5% while the 10% initial oxygen concentration group stayed at 0%. It is possible that the hexanal production was less in the 0% initial oxygen concentration group than in the 10% initial oxygen concentration group at 18 weeks and more at 22 weeks.

Error Analysis

The errors associated with the experiments include both operator and instrumental errors, and are considered normal. Sampling times for hexanal trapping are given in weeks and samples were taken on the 7th day, but not always at the same time of day. Therefore, an 8 hour maximum error is possible.

There is a lack of hexanal data throughout the middle of the storage period. During this time, hexanal measurements were taken and they appeared to be rising at a steady rate. However, it was noticed that hexanal values of chips that were only a couple of weeks old and that had oxygen absorbers in the packages were also high. It was then discovered that the glassware being used to trap

volatiles was not getting clean enough between trappings and oil was building up and oxidizing on the glassware. The glassware was then modified to prevent this, but much of the data already collected had to be discarded.

Error associated with concentrating the volume of extract to a 1 ml sample volume in the centrifuge tube is a result of misreading. Syringe error was a result of misreading the syringe calibrations and manufacturer error. The injection volume into the gas chromatograph could also vary due to leakage through the septa and loss due to evaporation of solvent. Retention time of hexanal could also vary due to carrier gas pressure and any variance between injection time and start time. The error associated with the area response of the integrator was due to the flame ionization detector.

SUMMARY AND CONCLUSIONS

The purpose of this study was to determine the effect of varying initial oxygen concentrations and the use of oxygen absorbers on the shelf life of potato chips. Headspace oxygen concentrations of packages containing oxygen absorbers were monitored daily until they reached a low point. Hexanal was used to quantify the extent of lipid oxidation over time.

BET Monolayer Moisture Content

The BET monolayer value is the moisture content which produces a monolayer coverage of water molecules over the highly reactive sites of the substrate, effectively retarding lipid oxidation. Determining the monolayer value in this study provided a guideline for estimating when the rate of lipid oxidation would be the lowest. The BET monolayer value for these chips was found to be 3.608g H₂O/100g dry product. The corresponding a_w was 0.197. All chips started at moisture contents below the monolayer value, and only chips packaged in metallized polyester without oxygen absorbers ever gained enough moisture to be above the monolayer moisture content value. This would indicate that all other groups were experiencing a retardation in oxidation rate over the course of the study.

Headspace Oxygen Concentration

The headspace oxygen concentration of packages with absorbers had initial average values of 0.0722%, 2.096%, 2.12%, and 9.641%. These initial oxygen concentrations were measured within 6 hours of packaging the potato chips. The metallized polypropylene packages with absorbers and the highest initial oxygen concentration decreased to a headspace oxygen concentration of 0.0000% after only 7 days. The packages of metallized polypropylene with absorbers and an initial average oxygen concentration of 2.096% took 10 days to reach 0.000% O₂. Even though the packages of metallized polyester have a lower oxygen permeability coefficient, they were unable to maintain oxygen concentrations of 0.000% for more than a week and it took the nitrogen flushed packages with absorbers 38 days to reach 0.0000% oxygen while the 2% oxygen flushed packages never reached 0.0000% oxygen.

This data shows that given an absorber of sufficient capacity, oxygen will be depleted from the headspace more quickly with a higher initial oxygen concentration. There is also some indication that a slightly inferior oxygen barrier aids in sustaining the oxygen/absorber reaction.

Applicability of Results

This study indicates the need for more research comparing the effectiveness of nitrogen gas flushing and the

use of absorbers. This study indicates that a combination of gas flushing and using an oxygen absorber would be inefficient since over time lower hexanal values were reported for high initial oxygen concentration packages with oxygen absorbers and for very low initial oxygen concentration packages without absorbers than for intermediate initial oxygen concentration packages with oxygen absorbers.

This study also indicates that a nitrogen gas flushing system must reduce the initial headspace oxygen concentration below 2% in order to be effective. The data supporting this is the hexanal data which shows no significant difference between hexanal production of chips with 2% initial oxygen concentration and 10% initial oxygen concentration.

The economics of the two separate headspace oxygen reducing systems also needs to be compared. It may be found that using absorbers is more economical than nitrogen gas flushing.

APPENDICES

APPENDIX A

Gas Chromatograph Hexanal Calibration Data

Table 7

Hexanal Data for the First Calibration Curve

<u>Sample</u>	<u>Grams of Hexanal Injected</u>	<u>Area Response</u>
1a	3.40E-09	4133
1b	3.40E-09	4014
1c	3.40E-09	4105
Average		4084
2a	8.30E-09	10604
2b	8.30E-09	9594
2c	8.30E-09	12177
Average		10099
3a	1.70E-08	19825
3b	1.70E-08	20220
3c	1.70E-08	22106
Average		20717
4a	2.50E-08	31548
4b	2.50E-08	30331
4c	2.50E-08	34721
Average		32200
5a	4.30E-08	55258
5b	4.30E-08	53823
5c	4.30E-08	56788
Average		55290

FIRST HEXANAL CALIBRATION CURVE

Hexanal in Acetonitrile

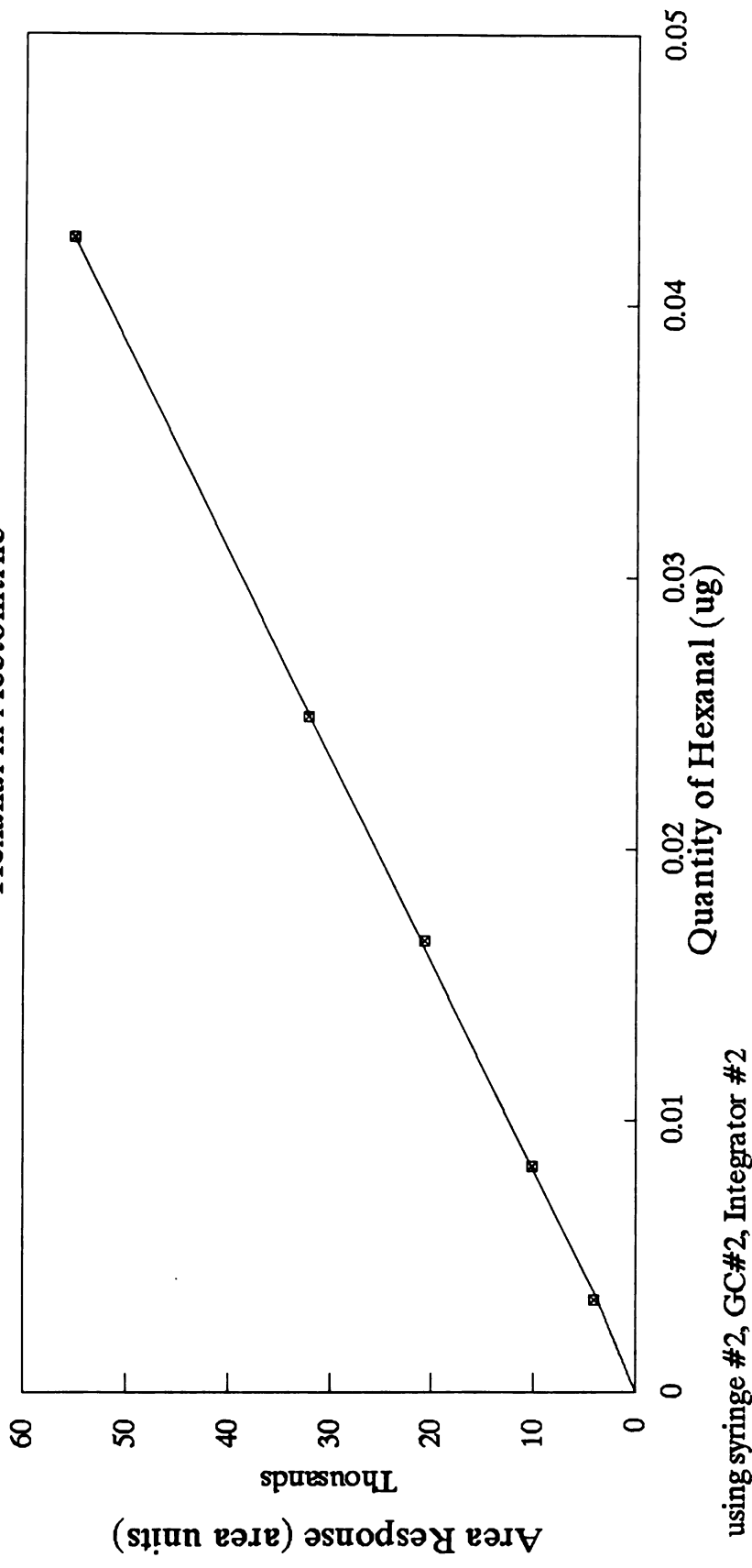


Figure 13: First Hexanal Calibration Curve

APPENDIX A

Gas Chromatograph Hexanal Calibration Data

Table 8

Hexanal Data for the Second Calibration Curve

<u>Sample</u>	<u>Grams of Hexanal Injected</u>	<u>Area Response</u>
1a	3.40E-09	8743
1b	3.40E-09	8556
1c	3.40E-09	8616
Average		8639
2a	8.30E-09	27190
2b	8.30E-09	26197
Average		26694
3a	1.70E-08	56498
3b	1.70E-08	56528
Average		56513
4a	2.50E-08	93025
4b	2.50E-08	88759
Average		90892
5a	4.30E-08	154790
5b	4.30E-08	151940
Average		153365

SECOND HEXANAL CALIBRATION CURVE

Hexanal in Acetonitrile

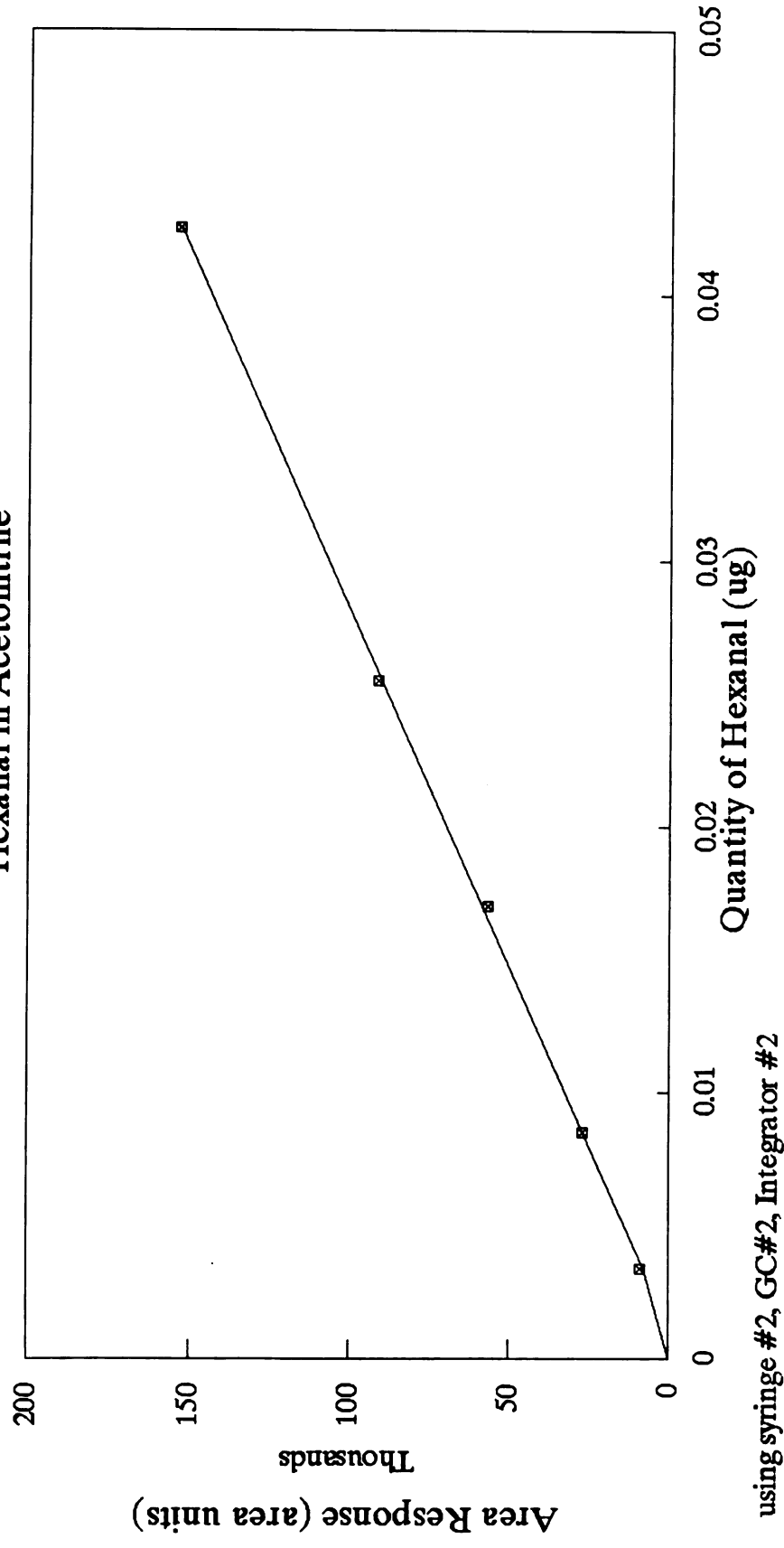


Figure 14: Second Hexanal Calibration Curve

APPENDIX B

Table 9**Chip Initial Moisture Content Data**

<u>Sample Number</u>	<u>Initial Chip Wt.</u>	<u>Dried Chip Wt.</u>	<u>Initial Moisture Content</u>
1	2.5356g	2.4698g	2.66%
2	2.8179g	2.7467g	2.59%
3	2.9587g	2.9449g	0.47%
4	2.3591g	2.3391g	0.86%
5	2.0309g	2.0029g	1.40%
6	2.5151g	2.4767g	1.55%
7	2.2375g	2.2084g	1.32%
8	2.2608g	2.2298g	1.39%
9	2.2261g	2.1944g	1.44%

Range = 0.47% - 2.66%

Average = 1.52%

Sample Standard Deviation = ± 0.715

APPENDIX C

Table 10

Sorption Isotherm Data

<u>Water Activity</u>	<u>Initial Chip Weight(g)</u>	<u>Equilibrium Chip Weight(g)</u>	<u>Equilibrium Moisture Content(EMC)%</u>	<u>Average EMC(%)</u>
0.1	2.1278	2.1701	3.5382	3.1634
0.1	2.1423	2.1748	3.0601	
0.1	2.2127	2.2426	2.8918	
0.21	2.2938	2.3440	3.7418	3.7626
0.21	2.2723	2.3237	3.8164	
0.21	2.0812	2.1265	3.7297	
0.32	2.0182	2.0954	5.4033	5.4567
0.32	2.3088	2.3906	5.1168	
0.32	2.2930	2.3908	5.8500	
0.41	2.1382	2.2314	5.9451	5.9721
0.41	2.2225	2.3184	5.9005	
0.41	2.0858	2.1793	6.0708	
0.51	2.3656	2.5086	7.6569	7.8884
0.51	2.1812	2.3180	7.8871	
0.51	2.0608	2.1948	8.1212	
0.71	2.2210	2.5597	17.0017	13.2903
0.71	2.1837	2.3928	11.2410	
0.71	2.0438	2.2473	11.6283	
0.83	2.0517	2.5016	23.7815	23.2586
0.83	2.2978	2.7315	20.6815	
0.83	2.0916	2.5818	25.3128	

APPENDIX D

WVTR Data, Package Weights

Table 11

Time (Days)	Metallized Polyester Package Weights			Empty Package Weights		
	<u>1</u>	<u>2</u>	<u>3</u>	<u>1</u>	<u>2</u>	<u>3</u>
0	127.51	115.28	112.42	3.6606	3.6395	3.7166
2	127.54	115.30	112.45	3.6618	3.6404	3.7173
8	127.63	115.39	112.54	3.6628	3.6412	3.7175
10	127.67	115.42	112.58	3.6576	3.6422	3.7413
12	127.71	115.46	112.61	3.6580	3.6350	3.7092
14	127.76	115.49	112.65	3.6588	3.6362	3.7105
16	127.79	115.52	112.68	3.6604	3.6386	3.7177
18	127.82	115.55	112.71	3.6615	3.6416	3.7154
22	127.89	115.61	112.77	3.6656	3.6372	3.7171

APPENDIX D

WVTR Data, Package Weights

Table 12						
Metallized Polypropylene Package Weights						
Time (Days)	Desiccant Package Weights			Empty Package Weights		
	<u>1</u>	<u>2</u>	<u>3</u>	<u>1</u>	<u>2</u>	<u>3</u>
0	145.16	120.83	99.70	3.8268	3.8219	3.9526
2	145.16	120.82	99.70	3.8273	3.8221	3.9516
8	145.17	120.84	99.72	3.8273	3.8226	3.9537
10	145.18	120.84	99.74	3.8275	3.8207	3.9531
12	145.18	120.84	99.75	3.8258	3.8224	3.9485
14	145.18	120.85	99.76	3.8297	3.8214	3.9502
16	145.19	120.85	99.77	3.8209	3.8180	3.9468
18	145.19	120.85	99.78	3.8264	3.8155	3.9491
22	145.19	120.86	99.79	3.8258	3.8235	3.9503

APPENDIX E**WVTR Data, Package Weight Gains**

Table 13						
Metallized Polyester Package Weight Gains						
Time (Days)	Desiccant Packages			Empty Packages		
	<u>1</u>	<u>2</u>	<u>3</u>	<u>1</u>	<u>2</u>	<u>3</u>
0	0	0	0	0	0	0
2	0.0267	0.0247	0.0278	0.0012	0.0009	0.0007
8	0.1234	0.1153	0.1196	0.0022	0.0017	0.0009
10	0.1641	0.1463	0.1562	-0.003	0.0027	-0.002
12	0.2043	0.1792	0.1887	-0.003	-0.005	-0.007
14	0.2501	0.2150	0.2287	-0.002	-0.003	-0.006
16	0.2809	0.2441	0.2564	-0.002	-0.001	0.0011
18	0.3158	0.2714	0.2861	0.0009	0.0021	-0.001
22	0.3866	0.3331	0.3487	-0.004	-0.002	0.0005

APPENDIX E

WVTR Data, Package Weight Gains

Table 14

Metallized Polypropylene Package Weight Gains

Time (Days)	Desiccant Packages			Empty Packages		
	<u>1</u>	<u>2</u>	<u>3</u>	<u>1</u>	<u>2</u>	<u>3</u>
0	0	0	0	0	0	0
2	-0.001	-0.002	0.0030	0.0005	0.0002	-0.001
8	0.0080	0.0095	0.0254	0.0005	0.0007	0.0011
10	0.0117	0.0123	0.0372	0.0007	-0.001	0.0005
12	0.0135	0.0169	0.0467	-0.001	0.0005	-0.004
14	0.0195	0.0208	0.0593	0.0029	-0.001	-0.002
16	0.0239	0.0223	0.0690	-0.006	-0.004	-0.006
18	0.0242	0.0239	0.0760	-0.000	-0.006	-0.004
22	0.0302	0.0337	0.0955	-0.001	0.0016	-0.002

APPENDIX E

WVTR Data, Package Weight Gains

Table 15

Average and Net Package Weight Gains

<u>Time (Days)</u>	<u>Metallized Polyester</u>			<u>Metallized Polypropylene</u>		
	<u>Avg Desiccant Gain</u>	<u>Avg Empty Gain</u>	<u>Net Package Gain</u>	<u>Avg Desiccant Gain</u>	<u>Avg Empty Gain</u>	<u>Net Pkg Gain</u>
2	0.0267	0.0009	0.0258	-3.3E-05	-1E-04	.00007
8	0.1194	0.0016	0.1178	0.0143	0.0008	0.0135
10	0.1555	-0.0009	0.1564	0.0204	7.2E-20	0.0204
12	0.1907	-0.0048	0.1956	0.0257	-0.0015	0.0272
14	0.2313	-0.0037	0.2350	0.0332	0.0000	0.0332
16	0.2605	0.0000	0.2605	0.0384	-0.0052	0.0436
18	0.2911	0.0006	0.2905	0.0414	-0.0034	0.0448
22	0.3561	-0.0020	0.3581	0.0531	-0.0006	0.0537

APPENDIX F

Weights of Potato Chips

Table 16

Weights (g) of Potato Chips Packaged in Metallized Polyester at 0% O₂

Bag #	Chip Wt	Package Weights									
		Day 0	Day 13	Day 27	Day 35	Day 48	Day 62	Day 69	Day 83	Day 98	Day 154
1	30.48	35.46	35.27	35.88							
2	32.53	37.80	38.07	38.28	38.33	38.46					
3	31.51	36.55	36.80	36.99	37.10	37.21	37.34	37.37	37.46		
4	30.78	35.95	36.21	36.42	36.51	36.63	36.75	36.79	36.91	36.98	
5	31.52	36.78	37.06	37.22	37.31	37.44	37.54	37.61	37.70	37.78	
6	31.75	36.93	37.15	37.32	37.44	37.53	37.64	37.67	37.78	37.84	38.02
7	31.55	36.65	36.93	37.07	37.19	37.29					
8	30.80	35.97	36.16	36.38	36.41	36.52	36.62	36.67	36.76	36.83	
9	30.84	36.03	36.28	36.46	36.52	36.64	36.75	36.79	36.87	36.93	37.15
10	30.07	35.22	35.46	35.61							
11	31.13	35.97	36.17	36.36	36.45	36.56	36.66	36.69	36.78		
12	31.80	37.58	37.89								
13	31.85	36.81	37.09	37.22							
14	30.79	35.99	36.25	36.39	36.50	36.60					
16	32.72	37.54	38.66	38.83	38.90	39.01	39.15	39.19	39.26	39.33	
17	33.41	38.46	38.72	38.90	38.98	39.11	39.22	39.25	39.35	39.42	
18	30.83	35.81	36.07	36.19	36.34	36.41	36.54	36.57	36.68		
19	30.67	36.26	36.55	36.70	36.77	36.88	36.96	37.01	37.09	37.16	37.38
20	32.42	37.51	37.76	37.88	37.98	38.08	38.19	38.24	38.32	38.4	

APPENDIX F

Weights of Potato Chips

Table 17

Weights (g) of Potato Chips Packaged in Metallized Polyester at 0% O₂ with an Absorber

Bag #	Chip Wt	Package Weights									
		Day 0	Day 6	Day 20	Day 34	Day 41	Day 50	Day 126	Day 154		
1	30.16	39.07	39.08	39.18	39.24	39.23			39.51		
2	30.40	39.52	39.51	39.72	39.80	39.76					
3	30.28	39.36	39.42	39.52	39.60	39.63					
4	30.40	39.61	39.27	39.78	39.88	39.87			40.15		
5	30.54	39.53	39.57	39.68	39.74	39.72					
6	30.57	39.76	39.03	39.90	39.97	39.95	40.04				
7	30.40	39.69	40.03	40.19	40.27	40.27					
8	30.64	39.98	39.98	40.06	40.14	40.15					
9	30.18	39.28	39.28	39.41	39.48	39.48					
10	30.87	40.20	40.22	40.31	40.35	40.39					
11	30.80	39.88	39.92	40.03	40.10	40.11	40.18				
12	30.10	39.22	39.22	39.28	39.31	39.30		39.48			
13	30.66	39.72	39.67	39.77	39.79	39.77					
14	30.39	39.63	39.65	39.69	39.75	39.73		39.91			
15	31.05	39.93	39.93	40.03	40.09	40.09	40.14				
16	30.10	39.42	39.30	39.58	39.64	39.72					
17	30.90	39.76	39.80	39.92	39.98	39.97			40.29		
18	30.37	39.51	39.52	39.62	39.68	39.69	39.75				
19	31.06	40.52	39.55	40.60	40.65	40.61					
20	30.44	39.58		39.64	39.67				40.89		

APPENDIX F

Weights of Potato Chips

Table 18

Weights (g) of Potato Chips Packaged in Metallized Polyester at 24 O₂

Bag #	Chip Wt		Package Weights											
	Day 0	Day 0	Day 13	Day 27	Day 35	Day 48	Day 62	Day 69	Day 83	Day 98	Day 154			
1	32.60	37.81	37.92	38.10	38.20	38.27	38.42	38.46	38.55	38.60	38.83			
2	31.47	36.80	36.95	37.11	37.21	37.31	37.40	37.45	37.53	37.59	37.80			
3	31.86	37.00	37.20	37.37	37.45	37.58	37.69	37.76	37.83	37.91				
4	31.24	36.52	36.71	36.87										
5	32.66	38.23	38.37	38.53	38.60	38.75	38.85	38.88	38.96					
6	31.48	37.97		38.27	38.44									
7	31.80	38.37	38.54	38.70	38.77	38.89	39.00	39.05	39.13					
8	30.97	36.17	36.34	36.50	36.61	36.73	36.82	36.88	36.95	37.01				
9	31.74	36.00	36.15	36.34	36.42	36.52	36.62	36.66	36.74	36.80				
10	30.92	36.27	36.44	36.56	36.69									
11	31.11	36.50	36.70	36.88	36.98	37.09	37.21	37.24	37.33	37.38				
12	30.59	36.27	36.46	36.61										
13	30.12	35.10	35.30	35.46		35.65	35.75	35.77	35.84	35.90				
14	30.22	35.66	35.88	36.05	36.16									
16	30.51	35.85	36.04	36.18	36.31	36.39	36.50	36.55	36.60	36.65				
17	32.30	37.87	38.05	38.20	38.31	38.42	38.53	38.57	38.67					
19	32.15	37.32	37.49	37.62										
20	30.73	35.91	36.05	36.21	36.32	36.39	36.51	36.55	36.64	36.70	36.90			

APPENDIX F

Weights of Potato Chips

Table 19

Weights (g) of Potato Chips Packaged in Metallized Polyester at 0% O₂ with an Absorber

Bag #	Chip Wt		Package Weights		Day 6		Day 20		Day 34		Day 41		Day 50		Day 126		Day 154	
	Day 0	Day 0	Day 0	Day 0	Day 6	Day 6	Day 20	Day 20	Day 34	Day 34	Day 41	Day 41	Day 50	Day 50	Day 126	Day 126	Day 154	Day 154
1	30.89	40.71	40.67	40.78	40.82	40.83	40.82	40.82	40.82	40.83	40.83	40.83						
2	30.42	39.86	39.87	39.88	40.02	40.04	39.88	40.02	40.02	40.04	40.04	40.04						
3	30.35	40.00	40.02	40.10	40.14	40.16	40.10	40.14	40.14	40.16	40.16	40.16	40.2					
5	30.20	39.74	39.71	39.78	39.84	39.86	39.78	39.84	39.84	39.86	39.86	39.86						
6	32.01	41.67	41.74	41.84	41.91	41.92	41.84	41.91	41.91	41.92	41.92	41.92			42.14			
7	30.64	40.48	40.51	40.63	40.71	40.75	40.63	40.71	40.71	40.75	40.75	40.75					41.34	
8	30.94	40.90	40.93	41.04	41.09	41.10	41.04	41.09	41.09	41.10	41.10	41.10	40.13					
9	30.72	40.02	34.38	40.07	40.10	40.10	40.07	40.10	40.10	40.10	40.10	40.10						
10	30.61	40.68	40.69	40.76	40.80	40.82	40.76	40.80	40.80	40.82	40.82	40.82						
11	30.94	40.84	40.84	40.93	40.97	40.97	40.93	40.97	40.97	40.97	40.97	40.97						
12	30.25	39.54	39.35	39.67	39.73	39.77	39.67	39.73	39.73	39.77	39.77	39.77					40.00	
13	30.75	39.53	39.54	39.67	39.74	39.78	39.67	39.74	39.74	39.78	39.78	39.78						
14	30.01	39.37	39.44	39.52	39.58	39.61	39.52	39.58	39.58	39.61	39.61	39.61						
15	30.84	40.06	40.09	40.16	40.21	40.23	40.16	40.21	40.21	40.23	40.23	40.23			40.43			
16	30.29	39.33	39.35	39.44	39.49	39.52	39.44	39.49	39.49	39.52	39.52	39.52						
17	30.26	39.77	39.81	39.93	39.99	40.02	39.93	39.99	39.99	40.02	40.02	40.02						
18	30.99	40.77	40.79	40.90	40.97	40.99	40.90	40.97	40.97	40.99	40.99	40.99	41.05					
19	30.19	40.20	40.22	40.35	40.41	40.43	40.35	40.41	40.41	40.43	40.43	40.43						
20	30.22	40.47	40.44	40.63	40.71	40.74	40.63	40.71	40.71	40.74	40.74	40.74	39.75				41.01	

APPENDIX F

Weights of Potato Chips

Table 20

Weights (g) of Potato Chips Packaged in Metallized Polypropylene at 2% O₂

Chip Package Weights

Bag #	Chip Wt	Day 0	Day 6	Day 13	Day 20	Day 34	Day 48	Day 70	Day 126	Day 154
1	30.45	36.54	36.55	36.52	36.54					
2	30.83	36.70	36.70	36.72	36.75	36.72	36.78		36.87	
3	31.10	37.03	37.05	37.05						
4	30.68	36.93	36.94	36.94	37.00	37.00				
6	30.68	37.50	37.47	37.48						
7	30.79	36.68	36.66	36.68	36.69					
8	30.61	36.80	36.82	36.83	36.85	36.84		36.88		
9	30.98	37.72	37.72	37.74	37.75	37.75		37.80		
10	31.07	37.12	37.09	37.10						36.38
11	30.31	36.17	36.21	36.22	36.21	36.22				
13	30.66	36.60	36.59	36.62	36.63					
14	30.13	36.38	36.40	36.42	36.43	36.45				
15	30.47	36.76	36.78	36.80	36.80	36.80			36.98	
16	30.57	37.09	37.08	37.10	37.10	37.14	37.15			
17	30.90	37.21	37.18	37.20	37.19	37.23	37.25		37.39	
18	30.91	37.11	37.09	37.09	37.11	37.11		37.15		
19	30.89	37.26	37.23	37.25	37.25	37.28	37.29			
20	31.45	37.20	37.23	37.28	37.28	37.28				

APPENDIX F

Weights of Potato Chips

Table 21

Weights (g) of Potato Chips Packaged in Metallized Polypropylene at 2% O₂ with an Absorber

Chip Package Weights

Bag #	Chip Wt	Day 0	Day 6	Day 20	Day 34	Day 41	Day 56	Day 126	Day 154
1	30.48	39.77	39.75	39.77	39.77	39.78			39.84
2	30.19	39.80	39.78	39.82	39.84	39.83			
3	30.15	39.81	40.14	40.18	40.23	40.23			40.28
4	30.40	40.31	40.28	40.33	40.34	40.35		40.41	
5	30.59	37.30	40.13	40.17	40.18	40.18			
6	30.36	40.12	40.09	40.13	40.15	40.15			
7	30.32	39.54	39.54	39.57	39.59	39.57			
8	30.13	40.06		40.09	40.10	40.10			
9	30.62	39.95	39.91	39.97	39.97	39.96			
10	30.96	40.61	40.60	40.64	40.65	40.66	40.67		
11	30.43	40.91		40.93	40.93	40.95			
12	30.85	40.29	40.36	40.51	40.52	40.52			
13	30.07	40.02	40.04	40.08	40.09	40.10			40.13
14	30.40	39.60		39.65	39.67	39.67			39.74
15	30.96	40.58	40.57	40.63	40.63	40.63	40.65		
16	30.43	39.99	39.97	40.02	40.04	40.06	40.07		
17	30.86	40.41	40.39	40.47	40.45				
18	30.22	40.47		40.51	40.51	40.51			
19	30.74	40.70	40.71	40.73	40.73	40.73			
20	30.45	40.50		40.54	40.53	40.55		40.62	

APPENDIX F

Weights of Potato Chips

Table 22

Weights (g) of Potato Chips Packaged in Metallized Polypropylene at 10% O₂

Chip Package Weights

Bag #	Chip Wt	Day 0	Day 6	Day 13	Day 20	Day 34	Day 48
1	30.45	36.54	36.55	36.52	36.54		
2	30.83	36.70	36.70	36.72	36.75	36.72	36.78
3	31.10	37.03	37.05	37.05			
4	30.68	36.93	36.94	36.94	37.00	37.00	
6	30.68	37.50	37.47	37.48			
7	30.79	36.68	36.66	36.68	36.69		
8	30.61	36.80	36.82	36.83	36.85	36.84	
9	30.98	37.72	37.72	37.74	37.75	37.75	
10	31.07	37.12	37.09	37.10			
11	30.31	36.17	36.21	36.22	36.21	36.22	
13	30.66	36.60	36.59	36.62	36.63		
14	30.13	36.38	36.40	36.42	36.43	36.45	
16	30.57	37.09	37.08	37.10	37.10	37.14	37.15
17	30.90	37.21	37.18	37.20	37.19	37.23	37.25
18	30.91	37.11	37.09	37.09	37.11	37.11	
19	30.89	37.26	37.23	37.25	37.25	37.28	37.29
20	31.45	37.20	37.23	37.28	37.28	37.28	

APPENDIX F

Weights of Potato Chips

Table 23

Weights (g) of Potato Chips Packaged in Metallized Polypropylene
at 10% O₂ with an Absorber

Bag #	Chip Wt	Package Weights									
		Day 0	Day 6	Day 20	Day 34	Day 41	Day 126	Day 154			
1	31.07	44.46		44.51	44.51						
2	30.23	43.69	43.78	43.79	43.78			43.85			
3	30.34	43.52	43.61	43.64	43.66	43.69					
4	30.64	44.12		44.21	44.23			44.31			
5	30.03	43.63									
7	30.54	44.26	44.44	44.57	44.67	44.68					
8	30.99	44.57		44.66	44.65						
9	30.68	43.61		43.92	43.93		43.96				
10	30.51	44.66		44.72	44.72			44.70			
11	30.83	44.29	44.39	44.40	44.38						
12	30.19	44.08	44.17	44.20	44.18		44.45				
13	30.59	43.95		44.02	44.03						
14	30.52	44.36	44.39	44.47	44.45						
15	30.56	43.89	43.99	44.00	44.01						
16	30.58	43.55		43.61	43.61			43.59			
17	30.37	44.91	44.94	44.98	44.97						
18	30.43	43.95	44.00	44.03	44.03						
19	30.65	43.92		44.00	44.00						
20	30.49	44.84		44.94	44.93						

APPENDIX G

Weight Gains of Potato Chips

Table 24

		Weight Gains (g) of Potato Chips Packaged in Metallized Polyester at 0% O ₂											
Bag#	Day	6	13	20	27	35	48	62	69	83	98	Day	154
1	0.17	0.26	0.35	0.42	0.48	0.53	0.66						
2	0.14	0.27	0.34	0.48	0.44	0.55	0.66						
3	0.14	0.25	0.33	0.44	0.47	0.55	0.68	0.79	0.82	0.91	1.03		
4	0.15	0.26	0.37	0.47	0.44	0.53	0.66	0.76	0.84	0.96	1.00		
5	0.17	0.28	0.35	0.44	0.39	0.51	0.60	0.71	0.83	0.92	1.00		
6	0.14	0.22	0.32	0.39	0.42	0.54	0.64	0.74	0.85	0.91	1.09		
7	0.15	0.28	0.36	0.42	0.41	0.54	0.64						
8	0.09	0.19	0.30	0.41	0.43	0.44	0.55	0.65	0.70	0.79	0.86		
9	0.15	0.25	0.34	0.43	0.39	0.49	0.61	0.72	0.76	0.84	0.90		
10	0.13	0.24	0.33	0.39	0.39	0.48	0.59						
11		0.20	0.30	0.39	0.41	0.51	0.61	0.69	0.72	0.81			
12	0.18	0.31	0.41	0.40	0.40	0.51	0.61						
13	0.13	0.28	0.34	0.40	0.44	0.52	0.65	1.61	1.65	1.72	1.79		
14	0.14	0.26	1.20	1.29	0.44	0.52	0.65	0.76	0.79	0.89	0.96		
16	1.02	1.12	0.36	0.38	0.38	0.53	0.60	0.73	0.76	0.87			
17	0.17	0.26	0.32	0.44	0.44	0.51	0.62	0.70	0.75	0.83	0.90		
18	0.16	0.26	0.37	0.44	0.37	0.47	0.57	0.68	0.73	0.81	0.89		
19	0.19	0.29	0.32	0.37	0.47	0.57	0.68	0.80	0.84	0.93	1.03		
20	0.15	0.25	0.39	0.47	0.57	0.68	0.80	0.84	0.93	1.03	1.11		
Avg.													
Gain	0.20	0.30	0.39	0.47	0.57	0.68	0.80	0.84	0.93	1.03	1.11		

APPENDIX G

Weight Gains of Potato Chips

Table 25						
Weight Gains (g) of Potato Chips Packaged in Metallized Polyester at 0% O ₂ with an Absorber						
Bag#	Day 6	Day 20	Day 34	Day 41	Day 50	Day 126
1	0.01	0.11	0.17	0.16		0.44
2		0.20	0.28	0.24		
3	0.06	0.16	0.24	0.27		
4		0.17	0.27	0.26		0.54
5	0.04	0.15	0.21	0.19		
6		0.14	0.21	0.19	0.28	
7	0.34	0.50	0.58	0.58		
8	0.00	0.08	0.16	0.17		
9	0.00	0.13	0.20	0.20		
10	0.02	0.11	0.15	0.19		
11	0.04	0.15	0.22	0.23	0.30	
12	0.00	0.06				0.26
13			0.05	0.07	0.05	
14	0.02	0.06	0.12	0.10		0.28
15	0.00	0.10	0.16	0.16	0.21	
16		0.16	0.22	0.30		
17	0.04	0.16	0.22	0.21		0.53
18	0.01	0.11	0.17	0.18	0.24	
19		0.08	0.13	0.09		0.37
20		0.06	0.09			
Av.						
Gain	0.04	0.14	0.20	0.21	0.26	0.27
						0.47

APPENDIX G

Weight Gains of Potato Chips

Table 26

		Weight Gains (g) of Potato Chips Packaged in Metallized Polyester at 24 O ₂											
Bag #		Day 6	Day 13	Day 20	Day 27	Day 35	Day 48	Day 62	Day 69	Day 83	Day 98	Day 154	
1		0.01	0.11	0.24	0.29	0.39	0.46	0.61	0.65	0.74	0.79	1.02	
2		0.03	0.15	0.25	0.31	0.41	0.51	0.60	0.65	0.73	0.79	1.00	
3		0.06	0.20	0.28	0.37	0.45	0.58	0.69	0.76	0.83	0.91		
4		0.07	0.19	0.26	0.35								
5		0.03	0.14	0.23	0.30	0.37	0.52	0.62	0.65	0.73			
6		0.06		0.27	0.30	0.47							
7		0.04	0.17	0.26	0.33	0.40	0.52	0.63	0.68	0.76			
8		0.07	0.17	0.23	0.33	0.44	0.56	0.65	0.71	0.78	0.84		
9		0.08	0.15	0.24	0.34	0.42	0.52	0.62	0.66	0.74	0.80		
10		0.07	0.17	0.26	0.29	0.42							
11		0.07	0.20	0.30	0.38	0.48	0.59	0.71	0.74	0.83	0.88		
12		0.07	0.19	0.23	0.34								
13		0.10	0.20	0.25	0.36								
14		0.07	0.22	0.28	0.39	0.50							
16		0.04	0.19	0.27	0.33	0.46	0.54	0.65	0.70	0.75	0.80		
17		0.05	0.18	0.26	0.33	0.44	0.55	0.66	0.70	0.80			
19		0.05	0.17	0.29	0.30								
20		0.05	0.14	0.23	0.30	0.41	0.48	0.60	0.64	0.73	0.79	0.99	
Avg.													
Gain	0.06	0.17	0.26	0.33	0.43	0.43	0.53	0.64	0.69	0.77	0.83	1.00	

APPENDIX G

Weight Gains of Potato Chips

Table 27

Weight Gains (g) of Potato Chips Packaged in Metallized Polyester
at 0% O₂ with an Absorber

Bag#	Day 6	Day 20	Day 34	Day 41	Day 50	Day 126	Day 154
1		0.07	0.11	0.12			
2	0.01	0.02	0.16	0.18			
3	0.02	0.10	0.14	0.16	0.20		
5		0.04	0.10	0.12			
6	0.07	0.17	0.24	0.25		0.47	
7	0.03	0.15	0.23	0.27			
8	0.03	0.14	0.19	0.20			0.44
9		0.05	0.08	0.08	0.11		
10	0.01	0.08	0.12	0.14			
11	0.00	0.09	0.13	0.13			
12		0.13	0.19	0.23			0.46
13	0.01	0.14	0.21	0.25			
14	0.07	0.15	0.21	0.24			
15	0.03	0.10	0.15	0.17		0.37	
16	0.02	0.11	0.16	0.19			
17	0.04	0.16	0.22	0.25			
18	0.02	0.13	0.20	0.22	0.28		
19	0.02	0.15	0.21	0.23			
20		0.16	0.24	0.27			0.56
Avg. Gain	0.03	0.11	0.17	0.19	0.20	0.42	0.48

APPENDIX G

Weight Gains of Potato Chips

Table 28

Weight Gains (g) of Potato Chips Packaged in Metallized Polypropylene at 2% O₂

Bag #	Day 6	Day 13	Day 20	Day 34	Day 48	Day 70	Day 126	Day 154
1	0.01		0.00					
2	0.00	0.02	0.05	0.02	0.08		0.17	
3	0.02	0.02						
4	0.01	0.01	0.07					
7			0.01					
8	0.02	0.03	0.05	0.04		0.08		
9	0.00	0.02	0.03	0.03		0.08		
11	0.04	0.05	0.04	0.05				0.21
13		0.02	0.03					
14	0.02	0.04	0.05				0.20	
15	0.02	0.04	0.04					0.22
16		0.01	0.01	0.05	0.06			
17				0.02	0.04			0.18
18			0.00			0.04		
20	0.03	0.08	0.08	0.08				
Avg. Gain	0.02	0.03	0.04	0.04	0.06	0.07	0.19	0.20

APPENDIX G

Weight Gains of Potato Chips

Table 29

Weight Gains (g) of Potato Chips Packaged in Metallized Polypropylene
at 2½ O₂ with an Absorber

Bag #	Day 6	Day 20	Day 34	Day 41	Day 56	Day 126	Day 154
1	-0.02	0.00	0.00	0.01			0.07
2	-0.02	0.02	0.04	0.03			
3	0.33	0.37	0.42	0.42			0.47
4	-0.03	0.02	0.03	0.04		0.10	
6	-0.03	0.01	0.03	0.03			
7	0.00	0.03	0.05	0.03			
8		0.03	0.04	0.04			
9	-0.04	0.02	0.02	0.01			
10	-0.01	0.03	0.04	0.05	0.06		
11		0.02	0.02	0.04			
12	0.07	0.22	0.23	0.23			
13	0.02	0.06	0.07	0.08			0.11
14		0.05	0.07	0.07			0.14
15	-0.01	0.05	0.05	0.05	0.07		
16	-0.02	0.03	0.05	0.07	0.08		
17	-0.02	0.06	0.05	0.04			
18		0.04	0.04	0.04			
19	0.01	0.03	0.03	0.03			
20		0.04	0.03	0.05			0.12
Avg.							
Gain	0.02	0.06	0.07	0.07	0.07	0.11	0.20

APPENDIX G

Weight Gains of Potato Chips

Table 30

Weight Gains (g) of Potato Chips Packaged in Metallized Polypropylene at 10% O₂

Bag #	Day 6	Day 13	Day 20	Day 34
1	0.01		0.00	
2	0.00	0.02	0.05	0.02
3	0.02	0.02		
4	0.01	0.01	0.07	
7			0.01	
8	0.02	0.03	0.05	0.04
9	0.00	0.02	0.03	0.03
11	0.04	0.05	0.04	0.05
13		0.02	0.03	
14	0.02	0.04	0.05	
15	0.02	0.04	0.04	
16		0.01	0.01	0.05
17				0.02
18			0.00	0.00
20	0.03	0.08	0.08	0.08
Avg. Gain	0.02	0.03	0.04	0.04

APPENDIX G

Weight Gains of Potato Chips

Table 31
 Weight Gains (g) of Potato Chips Packaged in Metallized Polypropylene
 at 10% O₂ with an Absorber

Bag #	Day 6	Day 20	Day 34	Day 41	Day 126	Day 154
1		0.05	0.05			
2	0.09	0.10	0.09			0.16
3	0.09	0.12	0.14	0.17		
4		0.09	0.11			0.19
7	0.18	0.31	0.41	0.42		
8		0.09	0.08			
9	0.31	0.31			0.35	
10		0.06	0.06			0.04
11	0.10	0.11	0.09		0.16	
12	0.09	0.12	0.10			
13		0.07	0.08			
14	0.03	0.11	0.09			
15	0.10	0.11	0.12			
16		0.06	0.06			0.04
17	0.03	0.07	0.06			
18	0.05	0.08	0.08			
19		0.08	0.08			
20		0.10	0.09			
Avg. Gain	0.08	0.11	0.12	0.30	0.26	0.11

APPENDIX H

Table 32

**Initial and Final Volumes of Potato Chip Packages
of Metallized Polyester Inflated with Nitrogen**

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
2	940	910
3	815	890
4	840	880
7	920	900
8	835	910
9	800	970
11	830	1000
14	890	940
18	920	870
19	915	920
20	820	910

Table 33

**Initial and Final Volumes of Potato Chip Packages
of Metallized Polyester Inflated with Nitrogen
and Packaged with Oxygen Absorbers**

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
1	860	950
2	730	760
4	965	970
9	860	940
10	800	850
11	910	855
12	925	930
14	910	970
15	810	810
16	760	910
17	855	900
18	790	880
19	820	820

APPENDIX H

Table 34

Initial and Final Volumes of Potato Chip Packages of
Metallized Polyester Inflated with 2% Oxygen/98% Nitrogen

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
1	860	940
2	905	910
3	975	940
5	885	890
6	920	910
7	940	950
9	900	890
10	840	830
14	960	960
17	880	890
20	920	930

Table 35

Initial and Final Volumes of Potato Chip Packages of
Metallized Polyester Inflated with 2% Oxygen/98% Nitrogen
and Packaged with Oxygen Absorbers

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
2	790	790
3	875	950
6	830	920
7	890	830
8	885	940
9	810	905
12	835	940
15	850	960
16	850	805
17	835	920
18	840	1000
20	930	930

APPENDIX H

Table 36

Initial and Final Volumes of Potato Chip Packages
of Metallized Polypropylene
Inflated with 2% Oxygen/98% Nitrogen

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
1	670	790
2	860	940
7	880	850
8	845	870
9	845	890
11	760	820
13	810	830
14	875	860
15	840	810
17	860	910
18	945	960

Table 37

Initial and Final Volumes of Potato Chip Packages
of Metallized Polypropylene Inflated with
2% Oxygen/98% Nitrogen and Packaged with Oxygen Absorbers

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
1	620	820
2	585	850
3	485	770
4	640	850
5	530	820
6	590	810
10	585	750
11	595	850
12	555	780
13	555	750
14	670	780
15	595	850
16	540	730
20	660	820

APPENDIX H

Table 38

Initial and Final Volumes of Potato Chip Packages
of Metallized Polypropylene Inflated with
10% Oxygen/90% Nitrogen

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
1	840	830
3	760	870
4	820	830
5	630	760
6	850	825
7	705	805
8	730	710
9	850	820
10	760	890
11	550	590
12	680	780
13	680	730
14	830	975
15	730	730
16	775	900
17	870	855
18	810	890
19	690	680
20	830	890

APPENDIX H

Table 39

**Initial and Final Volumes of Potato Chip Packages
of Metallized Polypropylene Inflated with
10% Oxygen/98% Nitrogen and Packaged with Oxygen Absorbers**

<u>Package Number</u>	<u>Initial Volume (cc)</u>	<u>Final Volume (cc)</u>
2	750	840
3	680	720
4	770	870
9	710	850
10	755	840
11	710	890
12	640	750
13	660	910
14	705	720
16	615	750
17	760	810
18	685	850
20	675	800

APPENDIX I

Headspace Oxygen Concentration Over Time

Table 40

Average Headspace Oxygen Concentrations (%) of
Metallized Polyester Packages Over Time

Days	0% O ₂ Flushed		2% O ₂ Flushed	
	w/o Absorber	w/Absorber	w/o Absorber	w/Absorber
0	0.1655	0.0722	2.262	2.12
1		0.0989		0.8238
2		0.1059		0.2267
3		0.092		0.0582
4		0.08295		0.0444
7		0.0503		0.03128
8		0.0402		0.01796
17		0.0134		0.00046
24				0.0085
28	2.667		4.103	
31				0.0296
38		0.0000		0.08482
42		0.2633	5.663	0.3263
49	4.31	0.1805		0.1323
84			7.99	
91	7.1			
98		3.77		1.264
126	8.355	0.8827	10.93	2.284
154	10.32	3.52	10.47	6.26

APPENDIX I

Headspace Oxygen Concentration Over Time

Table 41

Average Headspace Oxygen Concentrations (%) of
Metallized Polypropylene Packages Over Time

Days	2% O ₂ Flushed		10% O ₂ Flushed	
	w/o Absorber	w/Absorber	w/o Absorber	w/Absorber
0	2.298	2.096	9.998	9.641
1		0.971		0.7204
2		0.2403		0.03902
3		0.374		0.01404
4		0.0188		0.00092
7		0.00278		0.00000
8		0.00000		0.00000
14	3.493		11.083	
17		0.00000		0.00000
24		0.00000		0.00000
28	4.1		11.873	
31		0.0723		0.00000
38		0.3167		0.00000
42		0.0000	11.593	0.00000
56		0.2775		0.00000
70	6.56		12.24	
98		0.166	15.9	0.00000
112			13.96	
126	10.9	0.0000	17.36	0.00000
154	12.14	8.13	14.14	0.00000

APPENDIX J

Potato Chip Hexanal Data

Table 42

Hexanal Data for Fresh Chips

<u>Sampling Time (weeks)</u>	<u>Chip Weight (g)</u>	<u>Area Resp #1</u>	<u>Area Resp #2</u>	<u>Average Area Response</u>
0	10.0212	2460	2460	2460
0	10.1119	1692	1427	1559.5

<u>Sampling Time (weeks)</u>	<u>Inj. Size(ml)</u>	<u>Q Hex. Inj.(g)</u>	<u>Q Hex. Total(g)</u>	<u>Q Hex. (µg/g)</u>	<u>Average Q Hex (µg/g)</u>
0	0.00095	2.35e-09	0.000002	0.246371	0.211122
0	0.00093	1.65e-09	0.000002	0.175874	

APPENDIX J

Potato Chip Hexanal Data

Table 43

Hexanal Data for Chips in Metallized Polyester;
0% Initial O₂; Without an Absorber

<u>Sampling Time (weeks)</u>	<u>Chip Weight (g)</u>	<u>Area Resp #1</u>	<u>Area Resp #2</u>	<u>Average Area Response</u>
4	10.0457	1777	1561	1669
4	10.0817	4023	4274	4148.5
4	10.0217	2798	2803	2800.5
13	10.3495	4911	4345	4628
13	10.292	3044	3207	3125.5
13	10.1425	3444	3368	3406
22	9.9719	4311	4363	4337
22	10.0495	5010	4934	4972
22	10.0877	4244	3788	4016

<u>Sampling Time (weeks)</u>	<u>Inj. Size(ml)</u>	<u>Q Hex. Inj.(g)</u>	<u>Q Hex. Total(g)</u>	<u>Q Hex. (µg/g)</u>	<u>Average Q Hex (µg/g)</u>
4	0.000925	1.74e-09	0.000002	0.187713	0.285925
4	0.000925	3.63e-09	0.000004	0.389127	
4	0.000925	2.60e-09	0.000003	0.280934	
13	0.000925	3.99e-09	0.000004	0.417127	0.353555
13	0.0009	2.85e-09	0.000003	0.307823	
13	0.0009	3.06e-09	0.000003	0.335715	
22	0.0009	3.77e-09	0.000004	0.420304	0.426443
22	0.0009	4.25e-09	0.000005	0.47042	
22	0.0009	3.53e-09	0.000004	0.388606	

APPENDIX J

Potato Chip Hexanal Data

Table 44

Hexanal Data for Chips in Metallized Polyester;
0% Initial O₂; With a 100cc Capacity Absorber

Sampling Time (weeks)	Chip Weight (g)	Area Resp #1	Area Resp #2	Average Area Response
6	10.1012	3353	3304	3328.5
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE				
22	10.0096	10585	9874	10229.5
22	10.0024	21496	20471	20983.5
22	10.061	8094	7798	7946

Sampling Time (weeks)	Inj. Size(ml)	Q Hex. Inj.(g)	Q Hex. Total(g)	Q Hex. (µg/g)	Average Q Hex (µg/g)
6	0.000925	3.01e-09	0.000003	0.321673	0.321673
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE					
22	0.0009	4.06e-09	0.000005	0.450621	0.534409
22	0.0009	6.95e-09	0.000008	0.772082	
22	0.0009	3.45e-09	0.000004	0.380526	

APPENDIX J

Potato Chip Hexanal Data

Table 45

Hexanal Data for Chips in Metallized Polyester;
2% Initial O₂; Without an Absorber

<u>Sampling Time (weeks)</u>	<u>Chip Weight (g)</u>	<u>Area Resp #1</u>	<u>Area Resp #2</u>	<u>Average Area Response</u>
6	10.0206	3938	3912	3925
6	10.0824	5044	5000	5022
12	10.0792	4076	4127	4101.5
12	10.02755	6545	6572	6558.5
22	10.0299	8713	8332	8522.5
22	10.1864	7499	7541	7520
22	10.0816	5875	5522	5698.5

<u>Sampling Time (weeks)</u>	<u>Inj. Size(ml)</u>	<u>Q Hex. Inj.(g)</u>	<u>Q Hex. Total(g)</u>	<u>Q Hex. (µg/g)</u>	<u>Average Q Hex (µg/g)</u>
6	0.000925	3.46e-09	0.000004	0.373173	0.41673
6	0.000925	4.29e-09	0.000005	0.460288	
12	0.0009	3.59e-09	0.000004	0.396098	0.50058
12	0.0009	5.46e-09	0.000006	0.605063	
22	0.0009	6.95e-09	0.000008	0.770287	0.658469
22	0.0009	6.19e-09	0.000007	0.675341	
22	0.0009	4.81e-09	0.000005	0.529779	

APPENDIX J

Potato Chip Hexanal Data

Table 46

Hexanal Data for Chips in Metallized Polyester;
2% Initial O₂; With a 200cc Capacity Absorber

Sampling Time (weeks)	Chip Weight (g)	Area Resp #1	Area Resp #2	Average Area Response
7	10.0366	2402	2354	2378
7	10.1513	4486	4427	4456.5
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE				
22	10.0221	10222	10506	10364
22	10.1873	23526.5	24700	24113.25
22	10.1318	8354	7768	8061

Sampling Time (weeks)	Inj. Size(ml)	Q Hex. Inj.(g)	Q Hex. Total(g)	Q Hex. (μg/g)	Average Q Hex (μg/g)
7	0.0009	2.28e-09	0.000003	0.252759	0.254596
7	0.0009	2.34e-09	0.000003	0.256434	
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE					
22	0.0009	4.10e-09	0.000005	0.454067	0.561719
22	0.0009	7.79e-09	0.000009	0.849834	
22	0.0009	3.48e-09	0.000004	0.381257	

APPENDIX J

Potato Chip Hexanal Data

Table 47

**Hexanal Data for Chips in Metallized Polypropylene;
2% Initial O₂; Without an Absorber**

<u>Sampling Time (weeks)</u>	<u>Chip Weight (g)</u>	<u>Area Resp #1</u>	<u>Area Resp #2</u>	<u>Average Area Response</u>
2	10.0018	2570	2692	2631
2	10.0027	2689	2729	2709
4	10.068	2953	2952	2952.5
10	10.0886	4782	4695	4738.5
10	10.0874	2642	2788	2715
10	10.2044	3497	3508	3502.5
22	10.0405	9487	10148	9817.5
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE				
22	10.2478	13334	13654	13494
22	10.0926	9930	10731	10330.5

<u>Sampling Time (weeks)</u>	<u>Inj. Size(ml)</u>	<u>Q Hex. Inj.(g)</u>	<u>Q Hex. Total(g)</u>	<u>Q Hex. (µg/g)</u>	<u>Average Q Hex (µg/g)</u>
2	0.000925	2.48e-09	0.000003	0.267568	0.27076
2	0.000925	2.53e-09	0.000003	0.273951	
4	0.000925	2.72e-09	0.000003	0.292047	0.292047
10	0.0009	4.08e-09	0.000005	0.449051	0.356805
10	0.0009	2.54e-09	0.000003	0.279699	
10	0.0009	3.14e-09	0.000003	0.341665	
22	0.0009	7.94e-09	0.000009	0.878397	0.621199
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE					
22	0.0009	4.94e-09	0.000005	0.535297	
22	0.0009	4.09e-09	0.000005	0.449904	

APPENDIX J

Potato Chip Hexanal Data

Table 48

Hexanal Data for Chips in Metallized Polypropylene;
2% Initial O₂; With a 200cc Capacity Absorber

<u>Sampling Time (weeks)</u>	<u>Chip Weight (g)</u>	<u>Area Resp #1</u>	<u>Area Resp #2</u>	<u>Average Area Response</u>
8	10.0410	3868	3748	3808
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE				
22	11.1121	12881	12792	12836.5
22	10.2038	18565	18079	18322
22	10.3235	7236	6742	6989

<u>Sampling Time (weeks)</u>	<u>Inj. Size(ml)</u>	<u>Q Hex. Inj.(g)</u>	<u>Q Hex. Total(g)</u>	<u>Q Hex. (μg/g)</u>	<u>Average Q Hex (μg/g)</u>
8	0.000925	3.37e-09	0.000004	0.362841	0.362841
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE					
22	0.0009	4.76e-09	0.000005	0.475988	0.499361
22	0.0009	6.23e-09	0.000007	0.678934	
22	0.0009	3.19e-09	0.000004	0.343161	

APPENDIX J

Potato Chip Hexanal Data

Table 49

Hexanal Data for Chips in Metallized Polypropylene;
10% Initial O₂; Without an Absorber

Sampling Time (weeks)	Chip Weight (g)	Area Resp #1	Area Resp #2	Average Area Response
2	10.0954	2506	2168	2337
2	10.0575	1917	1933	1925
10	10.0798	5709	5727	5718
10	10.0506	5916	6107	6011.5
10	10.0024	4123	4127	4125
14	10.0366	3941	4187	4064
16	10.0184	4922	5174	5048
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE				
22	10.0794	15628	14565	15096.5
22	10.0350	11054	10644	10849
22	10.2494	23459	23315	23387

Sampling Time (weeks)	Inj. Size(ml)	Q Hex. Inj.(g)	Q Hex. Total(g)	Q Hex. (µg/g)	Average Q Hex (µg/g)
2	0.000925	2.25e-09	0.000002	0.241158	0.224783
2	0.000925	1.94e-09	0.000002	0.208407	
10	0.0009	4.82e-09	0.000005	0.531508	0.496781
10	0.0009	5.04e-09	0.000006	0.557713	
10	0.0009	3.61e-09	0.000004	0.401123	
14	0.0009	3.56e-09	0.000004	0.394623	0.394623
16	0.0009	4.31e-09	0.000005	0.478287	0.478287
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE					
22	0.0009	5.37e-09	0.000006	0.591729	0.627723
22	0.0009	4.23e-09	0.000005	0.46792	
22	0.0009	7.60e-09	0.000008	0.82352	

APPENDIX J

Potato Chip Hexanal Data

Table 50

Hexanal Data for Chips in Metallized Polypropylene;
10% Initial O₂; With a 400cc Capacity Absorber

Sampling Time (weeks)	Chip Weight (g)	Area Resp #1	Area Resp #2	Average Area Response
6	10.0963	3004	3695	3349.5
8	10.0415	3764	3205	3484.5
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE				
22	10.0941	6911	6651	6781
22	10.4600	7090	7180	7135
22	10.2446	6959	6559	6759

Sampling Time (weeks)	Inj. Size(ml)	Q Hex. Inj.(g)	Q Hex. Total(g)	Q Hex. (µg/g)	Average Q Hex (µg/g)
6	0.000925	3.02e-09	0.000003	0.323538	0.323538
8	0.000925	3.12e-09	0.000003	0.336351	0.336351
DATA BELOW THIS LINE IS FOR THE SECOND CALIBRATION CURVE					
22	0.0009	3.13e-09	0.000003	0.344804	0.342251
22	0.0009	3.23e-09	0.000004	0.342852	
22	0.0009	3.13e-09	0.000003	0.339098	

APPENDIX K

Table 51

Percent Recovery Data & Calculations

Solution 1:

<u>Injection</u>	<u>Solution for Basis Area Response</u>	<u>Extract 1 Area Response</u>	<u>Extract 2 Area Response</u>
1	24442	21576	23340
2	23260	18555	20608
3	22960	17404	21497
Average	23554	19178	21815
Recovery		81.4%	92.6%
Average Recovery			87%

Solution 2:

<u>Injection</u>	<u>Solution for Basis Area Response</u>	<u>Extract 1 Area Response</u>	<u>Extract 2 Area Response</u>
1	9585	8617	8869
2	9121	8141	8400
3	9702	9385	8827
Average	9469	8714	8699
Recovery		92.0%	91.9%
Average Recovery			91.9%

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