TECHNICAL REPORT
68-62-FL

# INTERRELATIONSHIPS BETWEEN STORAGE STABILITY AND MOISTURE SORPTION PROPERTIES OF DEHYDRATED FOODS (Phase II)

by

Norman Laine

Melpar, Inc.,
Falls Church, Virginia

Contract No. DA19-129-AMC-252 (N)

May 1968

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760


Food Laboratory
FL-75

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## FOREWORD

Previous investigations have indicated that the storage stability of dehydrated foods is greatly affected by the moisture content - water vapor equilibrium. Different foods or food classes have different requirements with respect to moisture and other in-package conditions. This investigation was undertaken for the purpose of obtaining information on the best storage conditions of three selected freeze-dried food products.

The specific objectives were to investigate the storage stability of the foods under varying conditions of moisture, headspace atmosphere and temperature, and to interpret the specific interactions occurring between water vapor, oxygen and freeze-dried foods.

The work was performed by Melpar, Inc., Falls Church, Virginia under Contract Number DA 19-129-AMC-252(N). Dr. Norman Laine was the Official Investigator. The U. S. Army Natick Laboratories Project Officer was Dr. Karl R. Johnson. The Alternate Project Officer was Dr. John G. Kapsalis.

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## ABSTRACT

Investigations were made on the moisture sorption behavior and the chemical stability upon storage of freeze-dried beef, sweet potatoes and spinach. Moisture sorption isotherms were determined at 22.0°and 37.8°C using a constant temperature spring balance. On the basis of these isotherms the B.E.T. monomolecular layer of water, heat of adsorption and constants of the Fugassi-Mitchell equation were determined. The foods were stored at three moisture levels, representing values below, at and above the B.E.T. monolayer, under air and under nitrogen, at 25° and 37.8°C for 3, 7 and 11 weeks. The foods were evaluated by a total of ten chemical tests and by limited sensory evaluation. Results showed that deterioration was more pronounced for foods stored under air than under nitrogen. In addition, foods stored at high moisture contents showed more deterioration than foods stored at lower moisture levels. All foods were more stable at the monolayer moisture level.

# INTERRELATIONSHIPS BETWEEN STORAGE STABILITY AND MOISTURE SORPTION PROPERTIES OF DEHYDRATED FOODS (Phase II)

## 1. INTRODUCTION

### 1.1 Objective

The final report on the study of "Interrelationships Between Storage Stability and Moisture Sorption Properties of Dehydrated Foods" is submitted in compliance with contract DA 19-129-AMC-252(N). The purpose of this work was to examine the storage stability of dehydrated foods under varying moisture conditions, different atmospheres, and different temperatures. The moisture levels for storage were determined from the moisture sorption isotherms of freshly dehydrated foods. The results of chemical evaluation of the stored foods were correlated with the results of the moisture sorption isotherms of these foods to interpret the specific interactions occurring between water vapor, oxygen and dehydrated foods.

### 1.2 Summary of Method

Moisture sorption isotherms of freshly dehydrated foods (sweet potato, beef, and spinach) were measured at 22.0° and 37.8° with the use of a constant-temperature spring balance. Monolayer sorption values and heats of sorption were determined from these sorption isotherms. Three moisture levels were chosen (below, at, and above the moisture monolayer value). Chemical evaluations were made on the stored foods to determine the amount of oxidation of fats, production of amino acids, and browning reactions. Moisture sorption isotherms were measured on foods stored at 37.8°C for periods of 3, 7 and 11 weeks. Chemical evaluations were made on foods stored at 25°C and 37.8°C.

## 1.3 Summary of Results

In this study, sweet potatoes, beef and spinach were put into storage. Chemical evaluation and moisture sorption isotherms of the stored food samples were measured. Due to the large number of food samples placed in storage, all of the food samples could not be evaluated after the completion of the storage periods at 25°C and 37.8°C. These foods were stored at -20°C until they could be evaluated. Changes occurring in the foods at -20°C were felt to be negligible for the few weeks that the foods were stored at this temperature.

Moisture sorption values at 22°C and 37.8°C are given for beef, sweet potatoes, and spinach stored at 37.8°C for 3, 7, and 11 weeks. Also included in this report are the chemical evaluations made on the foods stored at 25°C and 37.8°C for 3, 7 and 11 weeks.

The results of this final report indicate that deterioration has occurred in the stored foods. In general, the deterioration is more marked for foods stored under air than for foods stored under nitrogen. In addition, foods stored at high moisture levels exhibited more deterioration than foods stored at low moisture levels. One interesting exception to these general observations was that a loss in pigmentation occurred in sweet potatoes stored at low moisture levels. One conclusion drawn from this report is that the three foods were more stable at the monolayer moisture level.

## 2. EXPERIMENTAL PROGRAM

### 2.1 Constant-Temperature Sorption Apparatus

The constant-temperature sorption apparatus used in this second phase of the program was the same as that used in the first phase of the program under Contract No. DA 19-129-AMC-252(N), Project No. 1K01251A034. This apparatus (Figure 1) was fully described in the First Triannual[1] and Final Reports[2] of that contract. One modification of that apparatus was made to permit sorption studies to be made below room temperature. The box was lined with 20 feet of 3/8-inch diameter copper tubing which was connected to a standard refreigerator compressor unit (Westinghouse, 1/6th horsepower motor). Freon-12 was used as the refrigerant. No insulation was added to the box, the walls of which were 3/4-inch-thick plywood except for the front wall which was 1/4-inch-thick plexiglass. A temperature of 15° C has been maintained in the box with an outside room temperature of 24° C. The lower limit of temperature control is not known yet, but with this refrigeration additional constant temperatures can be maintained between 15° and 45° C with the outside laboratory temperature of 24° C.

### 2.2 Isotherm Measurements

The moisture sorption isotherms were measured by the static equilibrium method. The method was as follows. Powdered food samples were put into quartz sample buckets which were then suspended on springs

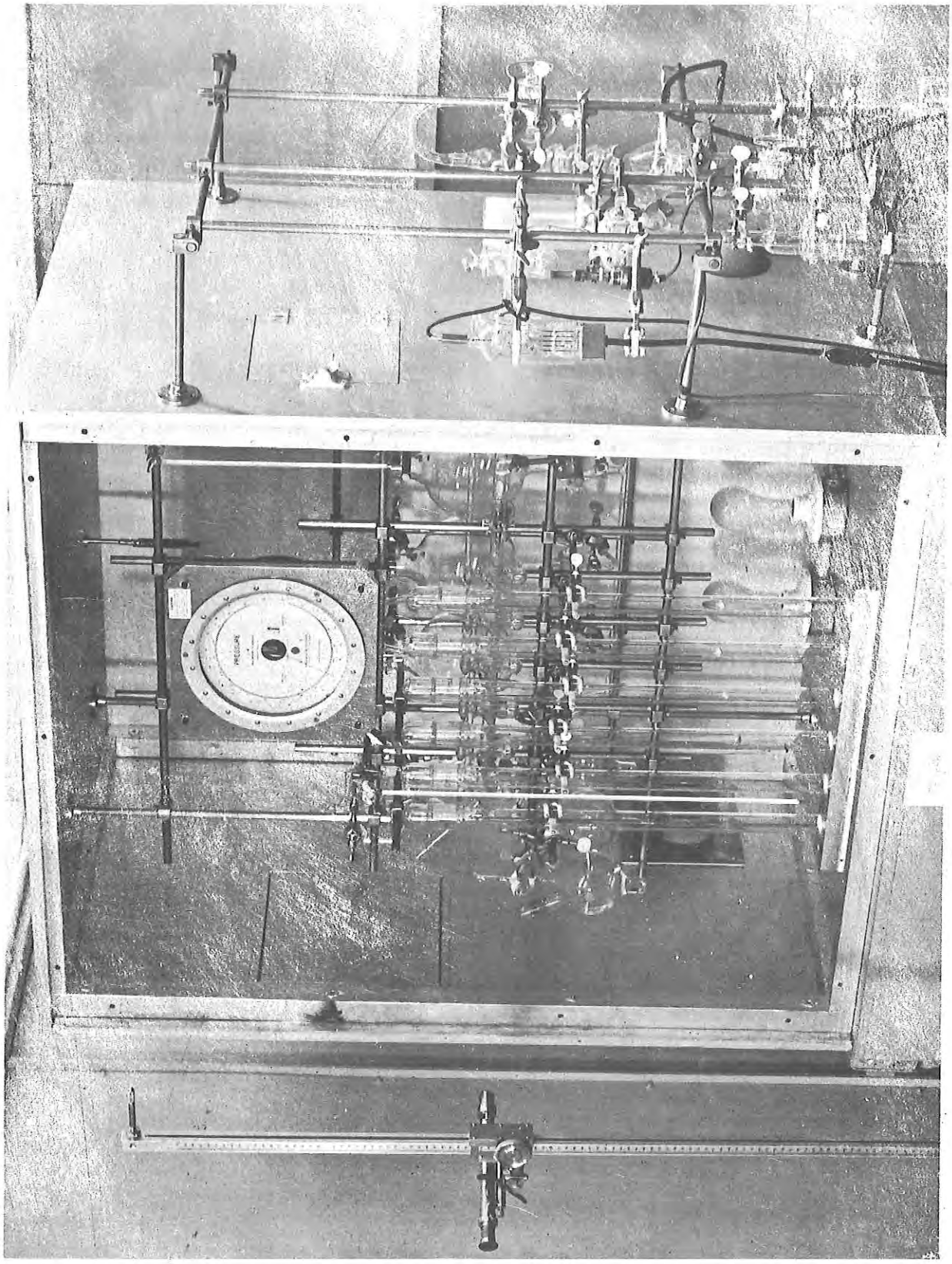

Figure 1. View of Constant Temperature Box, Sorption Apparatus and Cathetometer

in the sorption tubes (McBain-Bakr balance).[3] The entire apparatus was evacuated until the food samples ceased evolving moisture, which usually took about 30 hours. This was determined by following the movement of the spring with the aid of the cathetometer. It was assumed that equilibrium was reached when the spring did not expand or contract for four straight hourly readings. The sorption tubes were closed, water was let into the system to the desired pressure, and the sorption tubes were opened. The weight gain of the food samples due to moisture sorption was determined by measuring the amount of the spring extension. In all cases, equilibrium was assumed after four hourly cathetometer readings were the same. In the measurements of the isotherms, nine points were taken. An effort was made to spread these points uniformly over the vapor pressure range at the temperature used. The moisture sorption isotherms were measured at temperatures of 22.0° and 37.77°C. Temperature was controlled within ±0.05°C. Equilibration of the foods with moisture usually took 8 to 24 hours; a complete moisture sorption isotherm at any temperature, including desorption measurements, took about 2 weeks. For each sample, four or five desorption points, including complete desorption, were measured. For desorption, the sample tubes were then opened and the equilibrium point determined. For complete evaluation, the tubes were opened during evacuation.

The water used in these experiments was triply distilled water which was degassed before use.

Moisture sorption isotherms were measured at 22° and 37.8°C on freshly prepared, freeze-dried beef, spinach, and sweet potatoes. The foods were ground to -40 mesh in a Wiley mill before the measurements. The proximate analysis of these foods is given in Table 1. Two samples of each food were used at each temperature. Different samples of food were used at the different temperatures of measurement because of irreversible interactions occurring between water and the foods. Sample weights were approximately 0.2 grams. This weight was determined by the volume of the quartz brickets, the length of the sorption tubes (approximately 60 cm), and the linearity of the spring constants (0 to 0.5g). At high relative pressures, the upper limit could easily be reached. The extended spring would occupy the entire length of the tubes.

## 2.3 Choice and Analysis of Foods

Care was exercised in choosing the specific foods for this study. Foods were desired which were simple enough in chemical composition so that the results could be interpreted and possibly extrapolated to similar food stuffs. Another criterion for the foods was that they undergo an easily measureable degradation under some storage conditions. From work under previous contracts [2, 4] it was determined that, beef, spinach, and sweet potatoes met these conditions; therefore, these three foods were

chosen for study under the present contract. Proximate analysis of these three foods is given in Table 1. These analysis are on a moisture-free basis.

Table 1

PROXIMATE ANALYSIS OF FOODS (Moisture-free Bases)

| Food | % Protein | % Fat | % Carbohydrate | % Ash |
|---|---|---|---|---|
| Beef | 49.2 | 48.7 | 0 | 0.2 |
| Spinach | 23.5 | 45.9 | 29.1 | 1.5 |
| Sweet Potato | 5.7 | 2.2 | 88.6 | 3.5 |

2.4 Storage Conditions Employed

Each food was stored at three different moisture levels corresponding to monolayer sorption and one level each above and below the monolayer value. These moisture levels are shown in Table 2. At each moisture level the foods will be stored under an atmosphere of air and an atmosphere of nitrogen at 25° C and 37.8° C. In addition, dry samples of each food were stored at -20° C under air and under nitrogen for use as references in the evaluations. The foods were stored for periods of 3, 7 and 11 weeks. In previous studies, these storage intervals were found to be suitable for the detection of chemical changes occurring in the foods.

The moisture level corresponding to the monolayer value was derived experimentally (See section 3). The lower moisture level was set at the point attained by freeze-drying the foods in a commercial freeze-dryer (American Sterilizer Co., Model L-3 Laboratory Freeze-Dryer).

Table 2

RELATIVE HUMIDITIES AND MOISTURE

CONTENTS OF THE FOOD SAMPLES IN STORAGE

| | Beef | | Spinach | | Sweet Potato | |
|---|---|---|---|---|---|---|
| **25°C Storage** | | | | | | |
| | R.H. (%) | Moisture Content (%) | R.H. (%) | Moisture Content (%) | R.H. (%) | Moisture Content (%) |
| Low Moisture level | 8.5 | 1.4 | 6.0 | 1.4 | 4.5 | 1.2 |
| Monolayer Level | 28.0 | 3.5 | 21.5 | 4.8 | 21.5 | 3.8 |
| High Moisture Level | 61.0 | 7.9 | 54.5 | 11.1 | 58.2 | 11.0 |
| **37.8°C Storage** | | | | | | |
| Low Moisture Level | 8.5 | 1.4 | 6.0 | 1.4 | 4.5 | 1.2 |
| Monolayer Level | 28.0 | 3.3 | 21.5 | 4.8 | 21.5 | 4.5 |
| High Moisture Level | 61.0 | 7.9 | 54.5 | 11.1 | 58.2 | 11.0 |

The upper limit was chosen to be a moisture value larger than that necessary for the formation of the second monolayer

## 2.5 Preparation of Foods for Study

### 2.5.1 Freeze-Dried Beef

#### 2.5.1.1 Procedure for Freeze Drying the Beef

Extra lean beef was purchased from the local supermarket. The meat was made into large patties one-half inch in thickness and frozen. The patties were cut into strips 1/2-inch thick and placed in the freeze dryer. The lower plate was set at 250°F for the first 4 hours, after which the heat was discontinued. The freeze-drying cycle was completed in 2 days. After breaking the vacuum of the freeze-dryer with nitrogen, the beef was removed and placed in glass jars under nitrogen.

#### 2.5.1.2 Method for Adjusting the Ground Beef to a Moisture Content of 1.4%, 3.5% and 7.9%.

Using a vegetable slicer, the freeze-dried beef was reduced to a uniform particle size resembling coarse sawdust. It was then placed in a polyethylene bag and thoroughly blended.

One third of the ground beef from the above blend was packaged in metallized aluminum foil pouches in air and under nitrogen. This represented the food stored below the monolayer moisture level.

One portion of the dried beef was placed in a humidity chamber overnight which increased its moisture content to 10.8%. The color of the beef changed from a red to a reddish-brown. Aliquots of this high-moisture-content beef were blended with beef having 1.4% moisture content to produce blends with a moisture content of 3.5% and 7.9%. These blends were equilibrated overnight in a polyethylene bag and packaged in air and under nitrogen in metallized aluminum pouches.

2.5.1.3 Procedure for Packaging Under Nitrogen

The metallized aluminum pouches containing the ground beef and a heat sealer were placed in a dry box. The box was evacuated and flushed with nitrogen 5 times. The pouches were then heat sealed and placed in storage.

2.5.2 Freeze-Dried Spinach

2.5.2.1 Procedure for Freeze-Drying the Spinach

Twelve-ounce packages of frozen Belair chopped spinach were purchased from a local supermarket. The frozen spinach was cut in strips 1/2-inch in width and freeze-dried in an American Sterilizer Laboratory Model Freeze Dryer. For the first 4 hours the lower plate of the freeze-dryer was maintained at 250°F. The temperature was then lowered to 100°F and maintained for 2 days. After breaking the vacuum of the freeze with nitrogen, the spinach was removed and placed in glass jars under nitrogen and held at -20°C until used in the study.

2.5.2.2 Procedure for Adjusting and Storing the Spinach at a Moisture Content of 1.4%, 4.8% and 11.1%

The spinach was ground with a mortar and pestle and a uniform blend obtained by mixing in a polyethylene bag.

An aliquot of the blend was held in the freeze-dryer overnight with the lower plate at 80°F. The spinach was removed from the freeze-dryer and immediately placed in 125 ml. Erlenmeyer flasks containing rubber stoppers. Spinach stored at a moisture content of 1.4% was prepared by placing the flasks in a plastic box which was evacuated and flushed 5 times with nitrogen. The flasks were then sealed with rubber stoppers. This sample represented spinach stored at a moisture content of 1.4%.

A portion of the spinach was left overnight in a humidity cabinet saturated with water vapor. Blends of this sample were made with the spinach with a moisture content of 1.4% to produce blends of 4.8% and 11.1%. These blends were packaged under nitrogen and stored in Erlenmeyer flasks in the same manner as explained for the spinach with a moisture content of 1.4%.

2.5.3 Freeze-Dried Sweet Potatoes

2.5.3.1 Procedure for Freeze Dehydration

The sweet potatoes were cooked for 30 minutes in boiling water, peeled and sliced into liquid nitrogen. The frozen slices were trayed and

placed in the freeze-dryer. The lower plate of the freeze-dryer was held at 80°F and the run completed in 3 days. After breaking the vacuum of the freeze-dryer with nitrogen, the food was ground to a powder by means of a mortar and pestle and stored in glass jars under nitrogen at -20°C.

2.5.3.2 Method for Adjusting the Moisture Content to the Three Storage Levels

The freeze-dried sweet potatoes contained 1.2% water and were used for one storage condition. An aliquot was placed in a humidity cabinet saturated with water and frequently mixed because this food absorbed water very rapidly. The moisture content of the food increased to 11% in 2 hours, and this sample was used for a second storage condition. Blends of these two samples were used for the monolayer moisture levels.

2.5.3.3 Procedure for Packaging Under Nitrogen

The food was placed in test tubes equipped with rubber stoppers. The tubes were placed in a plastic box which was evacuated and flushed 5 times with nitrogen. The tubes were then stoppered, taped shut and placed in storage.

The sweet potatoes with a moisture content of 1.2% increased to 1.6% during the above procedure. The water originated chiefly from the sweet potatoes with a moisture content of 11.0%. When the spinach was

packaged under nitrogen, the food with a lower level of water was packaged separately and this increase in moisture did not occur.

Metallized aluminum pouches were not used to package the sweet potatoes as in the case of beef due to the discovery of an occasional leaking pouch. The leak presumably occurred when two seals cross each other. Packaging the food in glass tubes with a rubber stopper produced a better seal.

## 2.6 Evaluation of Stored Foods

### 2.6.1 Evaluation of Ground Beef

#### 2.6.1.1 Moisture Content

Approximately 2 grams of the ground beef were placed in an aluminum dish. After recording the color and odor of the sample, the meat was dried in a vacuum oven at 95-100°C for 5 hours under a vacuum of 29 inches. The loss in weight was reported as moisture.

#### 2.6.1.2 Extraction of the Fat with Ether

Sufficient beef was weighed into a 250 ml. beaker to yield 20 grams of anhydrous food. The moisture was removed by placing the food in the freeze-dryer overnight with the lower plate set at 80°F.

The beef was extracted 5 times with ether. The first extraction required 150 ml. of ether and the latter 4 extractions combined required 75 ml. of ether. The meat and ether were mixed for 15 minutes and allowed to settle 10 minutes. After the supernatant was decanted on to a 15 cm

Whatman #1 filter paper, the filtrate was collected in a 250 ml. volumetric flask.

A 20 ml. aliquot of the ether extract was taken for a peroxide number determination and the remainder of the extract was placed in a tared 250 ml. beaker. The defatted residue and the ether extract were allowed to stand in the hood overnight to remove the ether. The excess ether in the fat was removed by adding a few SiC chips and placing the fat in a vacuum oven at 70° C and 27 inches of vacuum. In 2 hours the fat appeared free of ether. The weight of the fat was then recorded.

2.6.1.3 Digestion of the Defatted Residue with Lipase

Five grams of the defatted beef was weighed into a 250 ml. beaker and 39 ml. of water was added. After soaking for 30 minutes, the pH was recorded and raised to 8.5. When 5 drops of 0.1N NaOH held the pH at or above 8.5 for 5 minutes, the end point was attained. This value was recorded as the titratable acidity.

The enzyme solution consisted of 0.9 gm sodium glycocholate, 0.9 gm pancreatic lipase and 9 ml. of water. One ml. of this solution was added to the defatted beef along with one ml. of toluene as a preservative. The samples were placed in the incubator at 37.8° C and in approximately 1 1/2 hours, the pH was adjusted to 8.5. The beakers were covered with aluminum foil and held in the incubator overnight. The next morning the odor of each sample was recorded. The pH was raised to

8.5 with NaOH. The amount of NaOH required was reported as per cent free fatty acids as oleic acid. Ten ml. of formalin was added and after it had reacted for 5 minutes, the pH was returned to 8.5. This result was expressed as grams of amino nitrogen.

2.6.1.4 Peroxide Number

A 20 ml. aliquot from the 250 ml. of ether extract was placed in a 250 ml. Erlenmeyer flask. Next, 50 ml. of a solution of freshly prepared 60% acetic acid plus 40% chloroform followed by 1 ml. of a saturated KI solution. After shaking for exactly 1 minute, 20 ml. of stable starch plus 80 ml. of water was added. Sufficient freshly prepared .01N sodium thiosulfate was added to cause the blue color to disappear. The results were expressed as millimoles peroxide per 100 grams of fat.

2.6.1.5 Precipitation of Phospholipids

After adding twenty seven ml. of acetone to the beef fat, the beaker was covered with aluminum foil and allowed to stand for approximately 2 hours. The phospholipids were removed by filtration through a Buchner funnel containing 5.5 cm. sheet of Whatman #1 filter paper into a tared 100 ml. beaker and residual acetone was removed by placing them in a vacuum oven for 2 hours at 70°C and 27" of vacuum. The weights were expressed as grams phospholipids per 100 grams of anhydrous beef.

2.6.1.6 Digestion of the Phospholipids with Lipase

One ml of an enzyme solution composed of 0.9 gm sodium glycocholate, 0.9 gm pancreatic lipase and 9 ml. of water was added to the phospholipids in the 100 ml. beaker. Fifteen ml. of water was added and the suspension thoroughly mixed. Twice during the day the pH was adjusted to 7 with 0.1N NaOH. The beakers were covered with aluminum foil and held overnight at 37.8° C. The next morning the pH was adjusted to 8.5 and the amount of alkali required was expressed as per cent free fatty acids as oleic per 100 grams of fat. Two ml. of formalin was added and the pH again returned to 8.5. This result was expressed as milligrams amino nitrogen per 100 grams lipid.

2.6.1.7 Analysis of Lipids Soluble in Acetone

Most of the acetone was removed from the lipids extracted by acetone by allowing them to stand in the hood overnight. The last traces of acetone were removed by placing them in a vacuum oven for 2 hours at 70° C and 27" of vacuum.

The lipids extracted with acetone were digested with lipase according to the procedure outlined in section 2.6.1.6. A formol titration was also carried out.

The free fatty acids present in the fat as oleic acid were determined according to the following procedure. To 1 gram of fat in a 100 ml. beaker

was added 50 ml. of alcohol and 2 ml. of a 1% alcohol solution of phenophthalein. The solution was heated to boiling and then titrated with 0.1N NaOH to a pink color. The results were expressed as percent free fatty acids as oleic acid per 100 grams of fat.

2.6.2 Evaluation of Spinach

2.6.2.1 Moisture Content

The moisture content of spinach was determined according to the procedure shown for ground beef in section 2.6.1.1.

2.6.2.2 Extraction of the Spinach with Water

An adequate amount of spinach was weighed into a 400 ml. beaker to yield 10 grams of anhydrous food. Two hundred ml. of water was added and the food was allowed to soak for 30 minutes. Using a magnetic stirrer, the spinach was mixed for 15 minutes and then filtered by suction through a Buchner funnel containing a 12.5 cm sheet of Whatman No. 4 filter paper. The residue was washed copiously with water. The spinach was extracted three times in this manner.

2.6.2.3 Formol Titration of the Residue Insoluble in Water

The volume of the aqueous slurry the food was adjusted to 400 ml. with water and the pH was recorded. The pH was raised to 8.5 with 0.1N NaOH. When 5 drops of alkali held the pH at or above 8.5 for 5 minutes the end point was attained. This value represented the titratable acidity.

Eighty ml. of a 5% solution of Versene (dipotassium salt of ethylenediaminetetracetic acid) plus 15 ml. of 2N NaOH was added. The chelating agent was allowed to react for 5 minutes and the pH was lowered below a pH of 8.5 with 10 ml. of IN NaOH. The pH was again adjusted to 8.5 with 0.1N NaOH, 25 ml. formalin added and the pH returned to 8.5.

2.6.2.4 Formol Titration of the Aqueous Extract

The volume of the aqueous extract was adjusted to 1 liter with distilled water. A 200 ml. aliquot was placed in a 400 ml. beaker and a formol titration was performed as shown in section 2.6.2.3. The formol titration was carried out using 40 ml. of a 5% Versene solution.

2.6.2.5 Titration of the Aqueous Extract with Iodine

A 200 ml. aliquot of the aqueous extract was placed in a liter Erlenmeyer flask along with 20 ml. of stable starch, 200 ml. of water and a lump of solid $CO_2$. After flushing the Erlenmeyer with $CO_2$ for 2 minutes, the iodine solution was added as rapidly as possible to give a reproducible blue end point. The rate for the addition of the iodine was kept as constant as possible for each of the samples. The time required to reach the blue end point was about 20 seconds. Again after 2, 3 and 5 minutes, the blue end point was reformed with the iodine solution.

When the titration was performed in the presence of 10 ml. formalin, 20 ml. 5% versene or 10 ml. formalin plus 20 ml. versene, the reagents were allowed to react for 5 minutes. Then 200 ml. of water was added

along with a piece of solid $CO_2$ and the titration was performed as above. In each case the results were expressed as milligrams ascorbic acid per 100 grams anhydrous spinach.

2.6.2.6 Changes in the Spinach During the Time Required for Its Analysis

For best results, only that amount of spinach which could be analyzed continuously was used. Spinach is quite fragile and its aqueous extract contains many readily oxidizable compounds. The iodine titration values for the aqueous extract were 30 to 40 units higher if performed on the same day as the extraction than if the aqueous extract were allowed to stand overnight in the refrigerator. Further, in the latter case, the tan or brown color of the extract became more intense. If the residue, which is insoluble in water, is allowed to stand in the refrigerator in excess of one day, the spinach begins to break up into fine particles. This is accompanied by an increase in the titratable acidity and amine nitrogen, presumably due to the hydrolysis and/or unwinding of the hemicelluloses and proteins.

When the spinach was taken out of storage, it was immediately analyzed. Previous work on spinach indicated that if it were frozen at moisture levels at and above the monolayer moisture level, changes occurred in the hemicelluloses as well as in the pectins. Freezing caused an enhancement of the titratable acidity.

### 2.6.3 Evaluation of Sweet Potatoes

#### 2.6.3.1 Extraction of the Sweet Potatoes with Water

Sufficient food was weighed out to yield 10 grams of anhydrous material. The food was ground with a mortar and pestle in the presence of water. The final volume was 200 ml. After allowing the food to soak 30 minutes in water, it was agitated for 15 minutes and filtered. The food was extracted four more times with 200 ml. of water and the filtrate was adjusted to a final volume of 1 liter.

#### 2.6.3.2 Formol Titration of the Residue Insoluble in Water

A formol titration was carried out on the insoluble residue of spinach suspended in 200 ml. of water. The procedure for the formol titration is the same as that used for the residue of spinach insoluble in water. The formol titration was performed in the presence of 40 ml. of a 5% solution of Versene. The amount of alkali required to return the pH to 8.5 with alkali represented the concentration of cations combined with the carboxyl groups of the hemicelluloses as pectins. Twenty ml. of formalin were added and after a reaction period of 5 minutes, the pH was returned to 8.5.

#### 2.6.3.3 Formol Titration of the Aqueous Extract

A formol titration was performed in a 200 ml. aliquot of the aqueous extract in the same manner as on the insoluble residue.

2.6.3.4 Titration of the Aqueous Extract with a 0.01N Iodine Solution

The procedure used for the titration of the aqueous extract of sweet potatoes by a 0.01N iodine solution is given in section 2.6.2. The iodine titration was also performed in the presence of 10 ml. of formalin, 20 ml. of 5% Versene, 10 ml. of formalin plus 20 ml. of Versene.

# 3. EXPERIMENTAL RESULTS

## 3.1 Moisture Sorption Measurements

### 3.1.1 Moisture Sorption Isotherms

Moisture sorption isotherms for freeze-dried beef, spinach, and sweet potato powder were measured at 22°C and 37.77°C. The equilibrium moisture sorption values for these isotherms are given in Tables 3 through 20. In addition, moisture desorption values for the original and stored foods are reported in Tables 21 through 27. The sorption isotherms for the original foods are given in Figures 2 through 4.

### 3.1.2 BET Monolayer Values

The equilibrium moisture sorption values were used to calculate the BET[5] monolayer values for the freeze-dried food powders.

TABLE 3

Moisture Sorption on Beef at 22°C
(Stored 3 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | N 1.4%$H_2O$ | N 3.5%$H_2O$ | N 7.6%$H_2O$ | Air 1.4%$H_2O$ | Air 3.5%$H_2O$ | Air 7.6%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 8.1 | 15.7 | 14.5 | 13.8 | 15.3 | 13.2 | 14.8 |
| 0.1 | 15.2 | 20.5 | 18.5 | 18.0 | 20.1 | 17.5 | 19.6 |
| 0.2 | 26.2 | 30.2 | 25.7 | 26.0 | 29.3 | 25.2 | 28.9 |
| 0.3 | 35.0 | 43.2 | 35.7 | 38.9 | 41.7 | 34.7 | 41.1 |
| 0.4 | 45.8 | 55.0 | 46.9 | 48.8 | 52.0 | 46.5 | 51.8 |
| 0.5 | 58.3 | 71.0 | 65.0 | 60.8 | 67.2 | 63.8 | 64.1 |
| 0.6 | 79.1 | 90.2 | 84.4 | 73.2 | 85.9 | 83.2 | 76.8 |
| 0.7 | 108.7 | 120.0 | 114.1 | 101.8 | 114.2 | 110.7 | 103.0 |
| 0.8 | 151.1 | 171.3 | 163.3 | 156.2 | 160.8 | 158.0 | 157.5 |
| 0.85 | 196.3 | 223.0 | 210.0 | 214.5 | 199.5 | 205.2 | 210.0 |

TABLE 4

Moisture Sorption on Beef at 22.0°C
(Stored 7 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 3.5%$H_2O$ | $N_2$ 7.6%$H_2O$ | Air 1.4%$H_2O$ | Air 3.5%$H_2O$ | Air 7.6%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 8.1 | 7.2 | 9.4 | 10.1 | 7.8 | 8.3 | 7.7 |
| 0.10 | 15.2 | 14.0 | 15.3 | 17.4 | 14.2 | 13.9 | 13.4 |
| 0.20 | 26.2 | 24.6 | 21.5 | 28.1 | 25.2 | 20.9 | 21.8 |
| 0.30 | 35.0 | 33.4 | 26.5 | 36.3 | 35.6 | 35.0 | 38.9 |
| 0.40 | 45.8 | 50.1 | 37.2 | 48.9 | 52.1 | 43.1 | 41.6 |
| 0.50 | 58.3 | 59.7 | 57.3 | 55.8 | 56.5 | 52.8 | 47.4 |
| 0.60 | 79.1 | 77.8 | 75.5 | 70.7 | 78.8 | 69.5 | 58.0 |
| 0.70 | 108.7 | 106.6 | 100.7 | 99.5 | 104.4 | 93.2 | 82.1 |
| 0.80 | 151.1 | 156.0 | 152.1 | 152.0 | 152.5 | 124.0 | 124.3 |
| 0.85 | 196.3 | 200.3 | 202.6 | 202.0 | 192.8 | 173.3 | 164.5 |

TABLE 5

Moisture Sorption on Beef at 22.0°C
(11 Weeks storage at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | N 1.4%$H_2O$ | N 3.5%$H_2O$ | N 7.6%$H_2O$ | Air 1.4%$H_2O$ | Air 3.5%$H_2O$ | Air 7.6%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 8.1 | 8.1 | 9.3 | 8.7 | 6.9 | 14.5 | 12.3 |
| 0.10 | 15.2 | 13.4 | 15.0 | 15.4 | 12.0 | 20.5 | 19.8 |
| 0.20 | 26.2 | 20.0 | 23.6 | 30.3 | 22.3 | 23.8 | 30.0 |
| 0.30 | 35.0 | 27.2 | 40.2 | 47.0 | 36.8 | 28.6 | 38.0 |
| 0.40 | 45.8 | 36.0 | 52.9 | 50.9 | 48.6 | 37.7 | 40.4 |
| 0.50 | 58.3 | 50.1 | 61.2 | 52.8 | 57.8 | 58.0 | 48.7 |
| 0.60 | 79.1 | 69.6 | 76.4 | 69.0 | 75.4 | 76.3 | 63.9 |
| 0.70 | 108.7 | 95.0 | 109.0 | 99.6 | 99.5 | 97.0 | 86.6 |
| 0.80 | 151.1 | 128.6 | 146.9 | 152.1 | 132.1 | 134.8 | 120.0 |
| 0.85 | 196.3 | 158.8 | 175.0 | 184.0 | 159.7 | 166.1 | 156.2 |

TABLE 6

Moisture Sorption on Beef at 37.8°C
(Stored 3 weeks at 37.8°C)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 3.5%$H_2O$ | $N_2$ 7.6%$H_2O$ | Air 1.4%$H_2O$ | Air 3.5%$H_2O$ | Air 7.6%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 9.4 | 8.1 | 6.9 | 7.1 | 8.7 | 9.1 | 8.2 |
| 0.1 | 16.3 | 13.2 | 12.3 | 12.5 | 14.7 | 13.7 | 13.1 |
| 0.2 | 26.8 | 21.4 | 20.9 | 21.2 | 23.7 | 20.2 | 21.2 |
| 0.3 | 36.2 | 31.1 | 30.1 | 30.9 | 32.7 | 28.8 | 30.6 |
| 0.4 | 46.0 | 42.8 | 41.1 | 41.8 | 43.1 | 38.3 | 41.1 |
| 0.5 | 58.3 | 55.5 | 54.2 | 53.2 | 55.7 | 50.5 | 53.2 |
| 0.6 | 77.5 | 72.2 | 72.2 | 69.8 | 73.3 | 67.6 | 68.1 |
| 0.7 | 102.8 | 93.0 | 94.3 | 93.2 | 95.0 | 89.2 | 89.8 |
| 0.8 | 142.1 | 129.9 | 132.2 | 133.8 | 131.9 | 125.8 | 129.2 |
| 0.85 | 185.4 | 158.7 | 163.3 | 165.8 | 161.1 | 153.9 | 159.8 |

TABLE 7

Moisture Sorption on Beef at 37.8°C
(Stored 7 weeks at 37.8°C
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 3.5%$H_2O$ | $N_2$ 7.6%$H_2O$ | Air 1.4%$H_2O$ | Air 3.5%$H_2O$ | Air 7.6%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 9.4 | 9.0 | 9.2 | 8.3 | 9.3 | 9.3 | 7.1 |
| 0.1 | 16.3 | 16.2 | 15.4 | 15.2 | 17.8 | 15.6 | 13.1 |
| 0.2 | 26.8 | 28.7 | 23.3 | 25.4 | 31.8 | 24.1 | 22.4 |
| 0.3 | 36.2 | 38.4 | 28.5 | 34.4 | 42.1 | 31.0 | 30.5 |
| 0.4 | 46.0 | 47.4 | 37.3 | 44.5 | 52.3 | 41.7 | 39.2 |
| 0.5 | 58.3 | 57.6 | 49.4 | 56.0 | 64.2 | 55.8 | 50.0 |
| 0.6 | 77.5 | 73.2 | 64.4 | 70.7 | 79.2 | 72.8 | 64.9 |
| 0.7 | 102.8 | 94.0 | 86.4 | 94.7 | 100.5 | 95.0 | 185.0 |
| 0.8 | 142.1 | 124.3 | 119.0 | 129.5 | 133.0 | 127.4 | 117.2 |
| 0.85 | 185.4 | 150.5 | 147.2 | 158.7 | 161.0 | 156.0 | 146.3 |

TABLE 8

Moisture Sorption on Beef at 37.8°C
(Stored 11 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 3.5%$H_2O$ | $N_2$ 7.6%$H_2O$ | Air 1.4%$H_2O$ | Air 3.5%$H_2O$ | Air 7.6%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 9.4 | 11.1 | 10.2 | 8.3 | 8.2 | 5.4 | 6.7 |
| 0.10 | 16.3 | 17.8 | 17.2 | 14.8 | 14.7 | 10.4 | 11.7 |
| 0.20 | 26.8 | 24.3 | 26.4 | 24.2 | 24.0 | 18.2 | 18.7 |
| 0.30 | 36.2 | 28.8 | 34.3 | 32.7 | 31.9 | 25.0 | 28.5 |
| 0.40 | 46.0 | 40.4 | 46.4 | 42.0 | 47.3 | 39.0 | 42.1 |
| 0.50 | 58.3 | 58.2 | 62.7 | 52.5 | 59.2 | 52.2 | 51.3 |
| 0.60 | 77.5 | 73.7 | 81.6 | 67.8 | 72.1 | 68.1 | 63.2 |
| 0.70 | 102.8 | 93.9 | 106.0 | 91.0 | 100.0 | 91.4 | 85.8 |
| 0.80 | 142.1 | 132.5 | 148.1 | 133.4 | 139.5 | 129.1 | 125.0 |
| 0.85 | 185.4 | 168.0 | 186.0 | 171.3 | 171.0 | 163.4 | 159.2 |

TABLE 9

Moisture Sorption on Sweet Potatoes at 22°C
(Stored 3 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.2%$H_2O$ | $N_2$ 4.4%$H_2O$ | $N_2$ 11%$H_2O$ | Air 1.2%$H_2O$ | Air 4.4%$H_2O$ | Air 11%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 10.3 | 11.5 | 10.3 | 7.4 | 11.6 | 10.3 | 7.5 |
| 0.10 | 18.1 | 21.4 | 19.7 | 13.6 | 21.4 | 19.4 | 12.6 |
| 0.20 | 30.0 | 37.2 | 33.0 | 22.1 | 37.4 | 32.1 | 20.2 |
| 0.30 | 41.5 | 50.3 | 42.8 | 33.0 | 49.8 | 42.6 | 28.0 |
| 0.40 | 65.9 | 70.2 | 64.2 | 51.6 | 70.3 | 63.4 | 48.5 |
| 0.50 | 95.9 | 97.0 | 93.4 | 80.2 | 98.3 | 91.6 | 77.8 |
| 0.60 | 140.0 | 142.1 | 132.3 | 120.7 | 134.8 | 132.0 | 118.3 |
| 0.70 | 208.8 | 226.6 | 215.7 | 194.3 | 216.2 | 205.0 | 192.4 |
| 0.80 | 329.0 | 373.0 | 366.0 | 352.0 | 358.0 | 346.7 | 340.0 |
| 0.85 | 433.0 | 447.5 | 446.0 | 446.0 | 430.0 | 426.5 | 421.6 |

TABLE 10

Moisture Sorption on Sweet Potatoes at 22°C
(Stored 7 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.2%$H_2O$ | $N_2$ 4.4%$H_2O$ | $N_2$ 11%$H_2O$ | Air 1.2%$H_2O$ | Air 4.4%$H_2O$ | Air 11%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 10.3 | 8.2 | 10.6 | 6.4 | 10.4 | 11.3 | 6.3 |
| 0.10 | 18.1 | 13.8 | 19.3 | 11.0 | 16.4 | 19.7 | 10.9 |
| 0.20 | 30.0 | 24.4 | 31.5 | 17.9 | 24.7 | 32.0 | 20.0 |
| 0.30 | 41.5 | 44.7 | 40.1 | 27.3 | 44.0 | 42.2 | 29.6 |
| 0.40 | 65.9 | 77.5 | 67.4 | 50.0 | 76.1 | 76.1 | 50.4 |
| 0.50 | 95.9 | 108.9 | 97.4 | 82.4 | 106.3 | 108.0 | 78.7 |
| 0.60 | 140.0 | 157.5 | 143.3 | 126.0 | 151.5 | 151.9 | 120.5 |
| 0.70 | 208.8 | 230.3 | 211.0 | 192.2 | 222.0 | 218.3 | 185.2 |
| 0.80 | 329.0 | 367.0 | 342.0 | 324.6 | 342.2 | 332.4 | 304.6 |
| 0.85 | 433.0 | 456.0 | 435.0 | 425.0 | 424.0 | 411.0 | 385.0 |

TABLE 11

Moisture Sorption on Sweet Potatoes at 22°C
(Storage 11 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.2%$H_2O$ | $N_2$ 4.4%$H_2O$ | $N_2$ 11%$H_2O$ | Air 1.2%$H_2O$ | Air 4.4%$H_2O$ | Air 11%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 10.3 | 9.7 | 10.9 | 5.5 | 7.8 | 17.0 | 12.3 |
| 0.10 | 18.1 | 16.0 | 18.5 | 9.5 | 14.6 | 25.0 | 17.7 |
| 0.20 | 30.0 | 22.5 | 27.4 | 14.3 | 24.4 | 29.1 | 19.8 |
| 0.30 | 41.5 | 34.6 | 36.5 | 18.0 | 38.5 | 41.3 | 28.4 |
| 0.40 | 65.9 | 72.3 | 60.3 | 41.0 | 64.7 | 63.9 | 48.2 |
| 0.50 | 95.9 | 98.0 | 86.0 | 65.6 | 101.3 | 90.0 | 75.0 |
| 0.60 | 140.0 | 136.4 | 124.0 | 105.0 | 137.8 | 125.7 | 112.0 |
| 0.70 | 208.8 | 197.1 | 181.8 | 161.2 | 197.0 | 180.8 | 163.0 |
| 0.80 | 329.0 | 310.0 | 284.6 | 277.9 | 297.0 | 286.0 | 253.6 |
| 0.85 | 433.0 | 396.0 | 379.0 | 333.5 | 367.5 | 364.0 | 328.0 |

TABLE 12

Moisture Sorption on Sweet Potatoes at 37.8°C
(Stored 3 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.2%$H_2O$ | $N_2$ 4.4%$H_2O$ | $N_2$ 11%$H_2O$ | Air 1.2%$H_2O$ | Air 4.4%$H_2O$ | Air 11%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 15.4 | 10.8 | 9.5 | 6.5 | 11.8 | 10.2 | 5.9 |
| 0.10 | 25.5 | 20.5 | 16.8 | 11.6 | 21.0 | 18.0 | 10.3 |
| 0.20 | 38.2 | 36.3 | 27.9 | 20.3 | 36.4 | 29.3 | 17.8 |
| 0.30 | 52.6 | 51.6 | 44.9 | 35.7 | 50.3 | 44.8 | 34.9 |
| 0.40 | 72.3 | 73.3 | 71.0 | 60.0 | 74.5 | 69.5 | 59.7 |
| 0.50 | 98.8 | 102.0 | 100.5 | 85.9 | 102.6 | 98.8 | 85.5 |
| 0.60 | 134.4 | 143.4 | 150.0 | 116.0 | 143.0 | 119.6 | 127.0 |
| 0.70 | 194.8 | 207.7 | 204.1 | 176.4 | 204.5 | 201.5 | 191.0 |
| 0.80 | 296.1 | 344.5 | 341.0 | 310.0 | 341.9 | 333.4 | 316.2 |
| 0.85 | 389.0 | 438.1 | 430.5 | 414.6 | 437.2 | 428.0 | 413.0 |

TABLE 13

Moisture Sorption on Sweet Potatoes at 37.8°C
(Stored 7 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.2%$H_2O$ | $N_2$ 4.4%$H_2O$ | $N_2$ 11%$H_2O$ | Air 1.2%$H_2O$ | Air 4.4%$H_2O$ | Air 11%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 15.4 | 10.7 | 9.8 | 7.0 | 10.4 | 10.4 | 8.6 |
| 0.10 | 25.5 | 20.1 | 16.7 | 13.1 | 18.6 | 19.8 | 15.7 |
| 0.20 | 38.2 | 31.6 | 26.6 | 22.2 | 30.1 | 33.4 | 25.3 |
| 0.30 | 52.6 | 50.5 | 37.9 | 36.7 | 45.7 | 45.6 | 35.0 |
| 0.40 | 72.3 | 72.0 | 60.2 | 59.5 | 70.2 | 68.3 | 63.0 |
| 0.50 | 98.0 | 100.5 | 88.0 | 88.0 | 96.0 | 97.0 | 88.2 |
| 0.60 | 134.4 | 137.7 | 126.3 | 125.8 | 132.3 | 134.0 | 125.0 |
| 0.70 | 194.8 | 190.0 | 177.5 | 175.0 | 186.4 | 186.0 | 178.0 |
| 0.80 | 296.1 | 275.0 | 259.4 | 260.3 | 274.3 | 270.3 | 259.0 |
| 0.85 | 389.0 | 353.0 | 337.0 | 339.0 | 353.0 | 345.0 | 348.0 |

TABLE 14

Moisture Sorption on Sweet Potatoes at 37.8°C
(Stored 11 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | N 1.2%$H_2O$ | N 4.4%$H_2O$ | N 11%$H_2O$ | Air 1.2%$H_2O$ | Air 4.4%$H_2O$ | Air 11%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 15.4 | 8.5 | 11.8 | 6.5 | 14.0 | 10.0 | 8.6 |
| 0.10 | 25.5 | 16.8 | 20.2 | 12.0 | 22.4 | 17.3 | 14.4 |
| 0.20 | 38.2 | 28.6 | 31.3 | 20.2 | 30.0 | 26.7 | 19.3 |
| 0.30 | 52.6 | 44.2 | 44.8 | 36.0 | 44.3 | 43.8 | 38.2 |
| 0.40 | 72.3 | 66.8 | 66.7 | 54.0 | 68.2 | 62.5 | 55.5 |
| 0.50 | 98.0 | 99.5 | 97.5 | 81.8 | 99.9 | 95.0 | 87.5 |
| 0.60 | 134.4 | 145.0 | 137.0 | 124.0 | 138.0 | 132.0 | 125.0 |
| 0.70 | 194.8 | 205.0 | 200.0 | 183.5 | 193.6 | 195.0 | 184.0 |
| 0.80 | 296.1 | 313.0 | 296.5 | 276.0 | 296.4 | 295.0 | 272.5 |
| 0.85 | 389.0 | 393.5 | 381.0 | 359.0 | 383.0 | 368.5 | 354.0 |

TABLE 15

Moisture Sorption on Spinach at 22.0°C
(Stored 3 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 4.8%$H_2O$ | $N_2$ 11.1%$H_2O$ | Air 1.4%$H_2O$ | Air 4.8%$H_2O$ | Air 11.1%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 27.9 | 13.4 | 16.3 | 19.6 | 16.3 | 14.5 | 17.0 |
| 0.10 | 36.3 | 24.5 | 29.5 | 33.8 | 29.4 | 26.4 | 30.2 |
| 0.20 | 48.8 | 42.2 | 49.6 | 53.1 | 49.2 | 43.8 | 50.4 |
| 0.30 | 60.0 | 60.0 | 65.0 | 66.4 | 63.4 | 57.7 | 64.5 |
| 0.40 | 76.5 | 75.7 | 80.0 | 78.3 | 79.5 | 73.5 | 75.1 |
| 0.50 | 100.0 | 98.2 | 104.2 | 96.5 | 101.5 | 95.3 | 93.7 |
| 0.60 | 134.3 | 134.3 | 136.9 | 121.8 | 132.0 | 132.0 | 121.5 |
| 0.70 | 185.7 | 180.0 | 179.8 | 168.7 | 174.0 | 177.5 | 157.0 |
| 0.80 | 308.0 | 288.0 | 278.3 | 275.0 | 270.8 | 280.0 | 247.0 |
| 0.85 | 441.0 | 411.0 | 400.0 | 380.0 | 376.0 | 374.0 | 344.6 |

TABLE 16

Moisture Sorption on Spinach at 22.0°C
(Stored 7 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 4.8%$H_2O$ | $N_2$ 11.1%$H_2O$ | Air 1.4%$H_2O$ | Air 4.8%$H_2O$ | Air 11.1%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 27.9 | 20.2 | 20.2 | 16.3 | 20.0 | 20.3 | 19.6 |
| 0.10 | 36.3 | 33.8 | 34.9 | 37.8 | 32.2 | 33.4 | 32.8 |
| 0.20 | 48.8 | 49.1 | 49.7 | 43.5 | 44.5 | 47.0 | 48.2 |
| 0.30 | 60.0 | 59.5 | 52.2 | 56.1 | 50.4 | 54.1 | 56.5 |
| 0.40 | 76.5 | 79.4 | 73.1 | 70.2 | 76.5 | 68.5 | 68.5 |
| 0.50 | 100.0 | 104.7 | 97.0 | 83.6 | 97.3 | 102.2 | 90.3 |
| 0.60 | 134.3 | 144.5 | 128.8 | 112.8 | 131.4 | 128.4 | 114.1 |
| 0.70 | 185.7 | 176.0 | 183.6 | 158.3 | 172.0 | 177.0 | 153.8 |
| 0.80 | 308.0 | 295.0 | 290.0 | 256.5 | 273.0 | 268.0 | 244.5 |
| 0.85 | 441.0 | 420.0 | 406.5 | 372.0 | 385.0 | 374.0 | 350.5 |

TABLE 17

Moisture Sorption on Spinach at 22.0°C
(Stored 11 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 4.8%$H_2O$ | $N_2$ 11.1%$H_2O$ | Air 1.4%$H_2O$ | Air 4.8%$H_2O$ | Air 11.1%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 27.9 | 21.5 | 27.8 | 23.6 | 17.5 | 27.2 | 20.5 |
| 0.10 | 36.3 | 34.9 | 43.5 | 36.2 | 29.1 | 29.5 | 33.0 |
| 0.20 | 48.8 | 51.6 | 54.4 | 48.7 | 45.6 | 47.2 | 48.6 |
| 0.30 | 60.0 | 61.9 | 57.1 | 56.0 | 58.4 | 54.3 | 58.0 |
| 0.40 | 76.5 | 80.0 | 72.5 | 68.0 | 76.0 | 68.4 | 70.4 |
| 0.50 | 100.0 | 103.7 | 103.3 | 87.6 | 96.3 | 97.5 | 87.3 |
| 0.60 | 134.3 | 134.6 | 122.0 | 111.1 | 130.0 | 144.2 | 114.4 |
| 0.70 | 185.7 | 181.2 | 169.0 | 149.3 | 169.4 | 170.0 | 145.8 |
| 0.80 | 308.0 | 315.0 | 294.5 | 274.5 | 289.0 | 287.0 | 258.5 |
| 0.85 | 441.0 | 440.0 | 408.0 | 381.0 | 386.0 | 382.0 | 352.0 |

TABLE 18

Moisture Sorption on Spinach at 37.8°C
(Stored 3 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 4.8%$H_2O$ | $N_2$ 11.1%$H_2O$ | Air 1.4%$H_2O$ | Air 4.8%$H_2O$ | Air 11.1%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 15.5 | 13.5 | 22.9 | 20 | 16.0 | 19.5 | 18.5 |
| 0.10 | 24.0 | 25.0 | 32.0 | 30 | 28.0 | 31.7 | 30.5 |
| 0.20 | 32.7 | 44.0 | 42.0 | 34.8 | 43.8 | 44.0 | 35.5 |
| 0.30 | 46.6 | 60.0 | 54.0 | 48.0 | 53.8 | 52.5 | 50.5 |
| 0.40 | 63.3 | 74.5 | 72.0 | 73.7 | 72.5 | 73.0 | 64.5 |
| 0.50 | 84.4 | 97.5 | 88.2 | 90.5 | 88.0 | 90.5 | 85.0 |
| 0.60 | 115.1 | 126.5 | 120.0 | 124.0 | 112.5 | 119.0 | 109.5 |
| 0.70 | 166.4 | 172.0 | 169.9 | 165.5 | 156.5 | 168.0 | 149.5 |
| 0.80 | 250.9 | 237.7 | 279.0 | 249.5 | 241.0 | 249.0 | 237.0 |
| 0.85 | 342.0 | 339.0 | 349.0 | 233.0 | 318.0 | 329.7 | 336.0 |

TABLE 19

Moisture Sorption on Spinach at 37.8°C
(Stored 7 weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 4.8%$H_2O$ | $N_2$ 11.1%$H_2O$ | Air 1.4%$H_2O$ | Air 4.8%$H_2O$ | Air 11.1%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 15.5 | 17.8 | 18.7 | 24.3 | 18.0 | 19.7 | 22.2 |
| 0.10 | 24.0 | 29.6 | 31.2 | 34.4 | 30.0 | 29.8 | 33.4 |
| 0.20 | 32.7 | 42.2 | 46.1 | 44.2 | 41.7 | 39.5 | 43.5 |
| 0.30 | 46.6 | 63.0 | 58.5 | 59.3 | 55.2 | 54.2 | 57.8 |
| 0.40 | 63.3 | 76.5 | 81.3 | 70.0 | 73.8 | 71.3 | 67.6 |
| 0.50 | 84.4 | 101.0 | 107.5 | 92.1 | 97.5 | 96.2 | 88.4 |
| 0.60 | 115.1 | 133.4 | 129.4 | 117.4 | 124.6 | 118.7 | 113.1 |
| 0.70 | 166.4 | 184.3 | 187.0 | 164.7 | 178.3 | 176.0 | 166.0 |
| 0.80 | 250.9 | 289.0 | 294.0 | 267.0 | 277.0 | 267.0 | 265.0 |
| 0.85 | 342.0 | 446.0 | 420.0 | 388.0 | 384.4 | 378.0 | 373.0 |

TABLE 20

Moisture Sorption on Spinach at 37.8°C
(Stored 11 Weeks at 37.8°C)
Equilibrium Weight (mg/g)

| P/Po | Original | $N_2$ 1.4%$H_2O$ | $N_2$ 4.8%$H_2O$ | $N_2$ 11.1%$H_2O$ | Air 1.4%$H_2O$ | Air 4.8%$H_2O$ | Air 11.1%$H_2O$ |
|---|---|---|---|---|---|---|---|
| 0.05 | 15.5 | 18.9 | 13.0 | 22.8 | 30.0 | 23.2 | 31.7 |
| 0.10 | 24.0 | 31.0 | 22.1 | 32.3 | 44.3 | 33.3 | 37.0 |
| 0.20 | 32.7 | 43.5 | 33.7 | 40.0 | 54.9 | 41.8 | 40.2 |
| 0.30 | 46.6 | 58.4 | 43.0 | 55.6 | 66.4 | 57.8 | 52.3 |
| 0.40 | 63.3 | 75.7 | 63.1 | 65.0 | 78.7 | 73.5 | 75.8 |
| 0.50 | 84.4 | 104.1 | 90.2 | 89.7 | 94.0 | 98.7 | 93.5 |
| 0.60 | 115.1 | 132.0 | 114.0 | 114.6 | 129.3 | 124.4 | 118.0 |
| 0.70 | 166.4 | 188.7 | 175.3 | 164.0 | 184.0 | 176.1 | 168.4 |
| 0.80 | 250.9 | 280.0 | 269.8 | 263.0 | 288.3 | 271.0 | 269.0 |
| 0.85 | 342.0 | 416.0 | 393.7 | 393.0 | 436.0 | 400.0 | 405.0 |

TABLE 21

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR FREEZE-DRIED FOOD POWDERS AT 37.77°C AND 22°C

| Rel. Pressure | Equilibrium Wt. (mg/g) at 37.77°C, Po=49.04 mm Hg | | |
|---|---|---|---|
| Pe/Po | Beef | Spinach | Sweet Potato |
| 0.826 | 156.3 | 288.0 | 329.3 |
| 0.623 | 88.3 | 127.6 | 150.7 |
| 0.326 | 46.2 | 52.3 | 56.3 |
| 0.061 | 17.6 | 20.9 | 25.9 |
| 0. | 6.4 | 0.0 | 10.1 |
| Rel Pressure | Equilibrium Wt. (mg/g) at 22°C, Po = 19.83 mm Hg. | | |
| Pe/Po | Beef | Spinach | Sweet Potato |
| 0.786 | 143.3 | 270.4 | 287.8 |
| 0.579 | 83.7 | 132.0 | 130.4 |
| 0.182 | 35.0 | 48.0 | 39.3 |
| 0. | 4.9 | 1.2 | 1.6 |

TABLE 22

Average Equilibrium Moisture Desorption

Values* for Beef at 22.0°C

| P/Po | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 3.5%$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|
| | | | Stored for 3 weeks at 37.8°C | | | |
| 0.869 | 271.2 | 259.2 | 252.0 | 250.9 | 245.3 | 249.7 |
| 0.789 | 174.1 | 167.2 | 158.5 | 163.7 | 160.1 | 160.5 |
| 0.500 | 80.1 | 74.3 | 70.1 | 74.0 | 71.7 | 70.7 |
| 0.0 | 4.2 | 1.4 | 1.7 | 3.6 | 2.1 | 2.8 |
| | | | Stored for 7 weeks at 37.8°C | | | |
| 0.662 | 111.0 | 95.9 | 96.1 | 97.9 | 88.1 | 80.8 |
| 0.419 | 67.8 | 53.3 | 53.1 | 56.2 | 49.1 | 43.8 |
| 0.146 | 50.1 | 25.8 | 24.1 | 28.1 | 23.5 | 19.9 |
| 0.0 | 13.6 | 0.0 | 1.5 | 0.5 | 0.0 | 0.0 |
| | | | Stored for 11 weeks at 37.8°C | | | |
| 0.677 | 114.5 | 101.2 | 178.4 | 108.5 | 110.0 | 101.4 |
| 0.460 | 66.4 | 58.3 | 69.0 | 62.2 | 62.7 | 59.5 |
| 0.177 | 34.5 | 25.7 | 23.0 | 30.7 | 31.0 | 23.3 |
| 0.0 | 6.5 | 5.1 | 0.0 | 7.4 | 7.5 | 0.5 |

* Note: Milligrams of moisture per gram of dry food.

TABLE 23

Average Equilibrium Moisture Desorption

Values* for Beef at 37.8°C

| P/Po | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ Under $N_2$ | 1.5%$H_2O$ under Air | 3.5%$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|
| | | | Stored for 3 weeks at 37.8°C | | | |
| 0.773 | 121.3 | 123.5 | 124.1 | 122.8 | 116.4 | 119.4 |
| 0.520 | 63.5 | 61.5 | 60.8 | 63.3 | 57.5 | 57.1 |
| 0.222 | 32.2 | 28.9 | 26.4 | 31.4 | 27.0 | 25.4 |
| 0.0 | 1.5 | 0.5 | 0.2 | 0.8 | 0.5 | 0.4 |
| | | | Stored for 7 weeks at 37.8°C | | | |
| 0.716 | 101.0 | 95.6 | 103.3 | 111.0 | 101.0 | 95.3 |
| 0.408 | 51.8 | 43.7 | 48.0 | 57.0 | 46.1 | 41.1 |
| 0.139 | 28.2 | 22.0 | 22.9 | 35.3 | 24.6 | 22.2 |
| 0.031 | 20.5 | 10.8 | 11.4 | 22.7 | 12.0 | 10.3 |
| 0.0 | 12.0 | 2.4 | 1.6 | 16.1 | 2.0 | 3.3 |
| | | | Stored for 11 weeks at 37.8°C | | | |
| 0.551 | 66.4 | 75.1 | 63.6 | 69.4 | 60.7 | 63.8 |
| 0.184 | 24.4 | 32.6 | 24.7 | 30.9 | 17.2 | 22.3 |
| 0.033 | 8.3 | 14.0 | 5.4 | 12.6 | 10.0 | 1.8 |
| 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |

* Note: Milligrams of moisture per gram of dry food.

TABLE 24

Average Equilibrium Moisture Desorption

Values* for Sweet Potatoes at 22.0°C

| P/Po | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|
| | | | Stored for 3 weeks at 37.8°C | | | |
| 0.742 | 234.6 | 224.1 | 210.2 | 225.6 | 218.9 | 205.1 |
| 0.606 | 142.9 | 135.5 | 123.7 | 138.9 | 133.8 | 120.9 |
| 0.162 | 51.5 | 47.4 | 36.7 | 49.1 | 36.8 | 36.2 |
| 0.0 | 20.6 | 16.4 | 7.7 | 19.3 | 13.7 | 6.4 |
| | | | Stored for 7 weeks at 37.8°C | | | |
| 0.651 | 188.7 | 171.7 | 154.5 | 182.1 | 180.1 | 149.3 |
| 0.515 | 119.5 | 107.6 | 89.7 | 113.6 | 115.0 | 86.3 |
| 0.237 | 63.7 | 55.7 | 40.2 | 62.5 | 65.5 | 39.1 |
| 0.0 | 16.5 | 7.4 | 1.1 | 14.2 | 15.9 | 1.3 |
| | | | Stored for 11 weeks at 37.8°C | | | |
| 0.551 | 128.8 | 119.0 | 98.9 | 131.1 | 119.4 | 104.8 |
| 0.278 | 60.5 | 52.1 | 33.9 | 60.3 | 58.0 | 42.9 |
| 0.0 | 17.4 | 8.5 | 0.0 | 20.1 | 7.0 | 0.0 |

* Note: Milligrams of moisture per gram of dry food.

TABLE 25

Average Equilibrium Moisture Desorption

Values* for Sweet Potatoes at 37.8°C

| P/Po | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|
| 0.864 | 348.3 | 341.3 | 317.6 | 346.6 | 338.8 | 327.6 |
| 0.734 | 221.3 | 217.8 | 191.9 | 219.6 | 214.0 | 228.9 |
| 0.400 | 74.1 | 70.0 | 51.5 | 72.4 | 70.6 | 58.7 |
| 0.0 | 16.6 | 13.8 | 4.1 | 15.6 | 13.7 | 10.4 |
| | | | Stored for 7 weeks at 37.8°C | | | |
| 0.701 | 201.4 | 187.6 | 187.5 | 198.7 | 192.8 | 185.7 |
| 0.467 | 88.4 | 78.5 | 80.8 | 88.8 | 86.3 | 80.3 |
| 0.133 | 27.7 | 21.1 | 26.3 | 29.4 | 28.3 | 23.8 |
| 0.0 | 13.5 | 3.8 | 10.4 | 12.8 | 10.0 | 6.7 |
| | | | Stored for 11 weeks at 37.8°C | | | |
| 0.581 | 86.8 | 129.4 | 115.2 | 129.7 | 126.2 | 116.4 |
| 0.257 | 46.4 | 44.5 | 36.9 | 42.3 | 44.4 | 35.7 |
| 0.053 | 24.8 | 22.2 | 14.0 | 22.1 | 21.7 | 16.3 |
| 0.0 | 21.7 | 20.3 | 10.4 | 14.9 | 16.5 | 10.4 |

* Note: Milligrams of moisture per gram of dry food.

Table 26

Average Equilibrium Moisture Desorption

Values* for Spinach at 22.0°C

| P/Po | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.1%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.1%$H_2O$ under Air |
|---|---|---|---|---|---|---|
| | | | Stored for 3 Weeks at 37.8°C | | | |
| 0.763 | 242.2 | 238.2 | 233.5 | 233.3 | 235.7 | 213.8 |
| 0.505 | 103.4 | 110.5 | 103.8 | 103.0 | 106.0 | 102.4 |
| 0.0 | 0.0 | 13.2 | 8.3 | 6.9 | 9.1 | 5.9 |
| | | | Stored for 7 Weeks at 37.8°C | | | |
| 0.702 | 192.0 | 183.6 | 171.4 | 183.0 | 184.4 | 166.7 |
| 0.288 | 67.4 | 69.5 | 56.1 | 60.7 | 61.0 | 65.3 |
| 0.0 | 5.7 | 5.5 | 0.0 | 2.1 | 3.3 | 0.0 |
| | | | Stored for 11 Weeks at 37.8°C | | | |
| 0.717 | 214.3 | 191.5 | 187.6 | 195.2 | 196.7 | 180.7 |
| 0.318 | 70.1 | 68.6 | 62.1 | 65.7 | 65.7 | 68.5 |
| 0.0 | 0.0 | 0.6 | 0.0 | 6.0 | 1.0 | 5.3 |

* Note: Milligrams of Moisture per gram of dry food.

TABLE 27

Average Equilibrium Moisture Desorption Values* for Spinach 37.8°C

| P/Po | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.1%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.1%$H_2O$ under Air |
|---|---|---|---|---|---|---|
| | | | Stored for 3 Weeks at 37.8°C | | | |
| 0.420 | 76.7 | 86.3 | 90.0 | 77.6 | 77.2 | 71.3 |
| 0.133 | 20.9 | 41.7 | 62.3 | 40.0 | 38.4 | 37.9 |
| 0.0 | 1.9 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | | | Stored for 7 Weeks at 37.8°C | | | |
| 0.540 | 119.2 | 107.6 | 104.6 | 104.4 | 100.9 | 95.5 |
| 0.086 | 38.7 | 36.4 | 34.6 | 35.3 | 44.6 | 40.0 |
| 0.0 | 9.1 | 0.0 | 0.0 | 1.5 | 0.0 | 0.0 |
| | | | Stored for 11 Weeks at 37.8°C | | | |
| 0.555 | 113.1 | 104.8 | 108.2 | 124.2 | 111.9 | 108.1 |
| 0.147 | 43.5 | 37.6 | 43.2 | 54.8 | 45.4 | 45.6 |
| 0.0 | 1.8 | 0.0 | 2.0 | 6.7 | 0.0 | 1.4 |

* Note: Milligrams of moisture per gram of dry food.

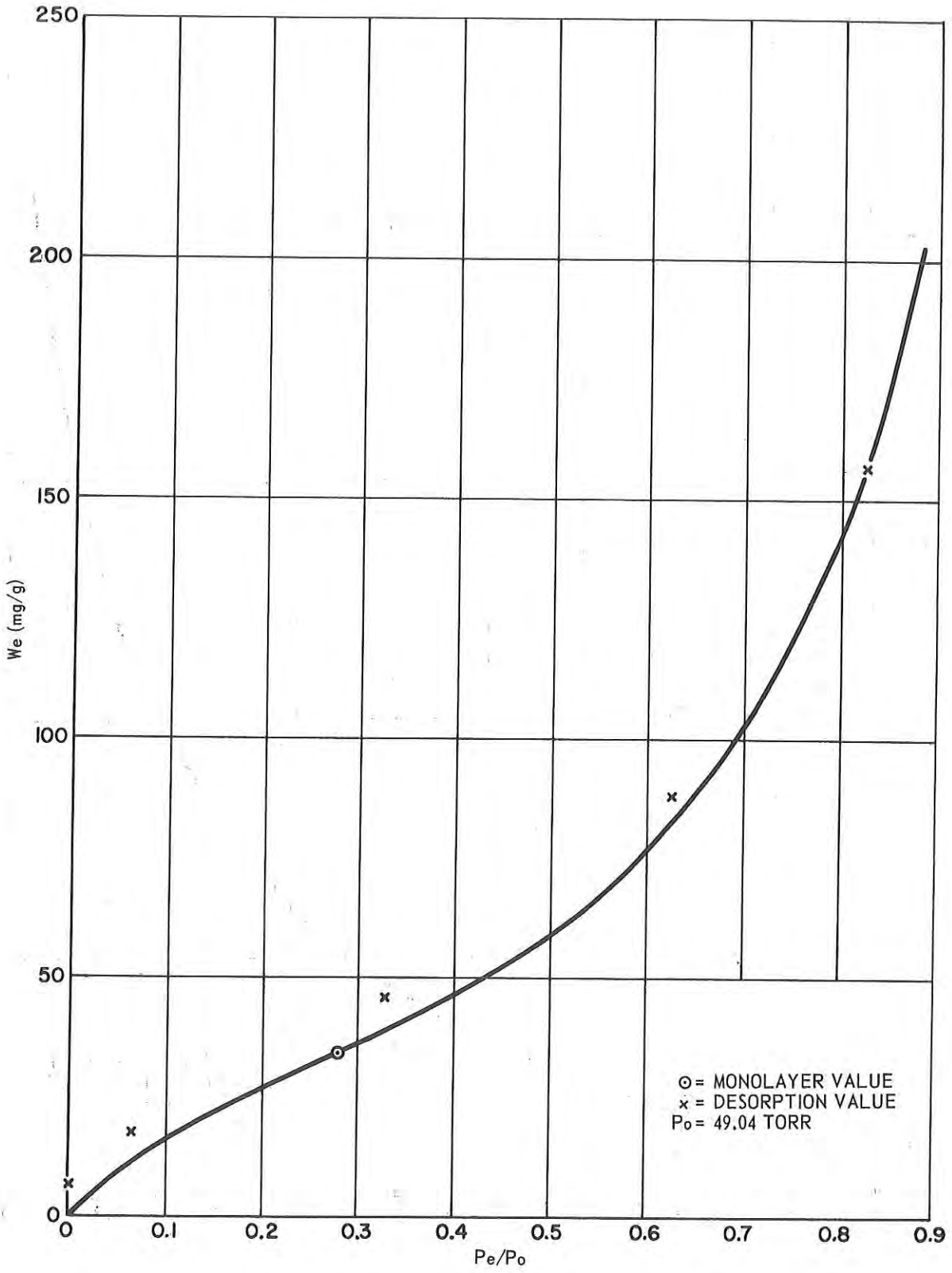


Figure 2. Moisture Sorption Isotherm for Freeze-Dried Beef Powder at 37.77°C

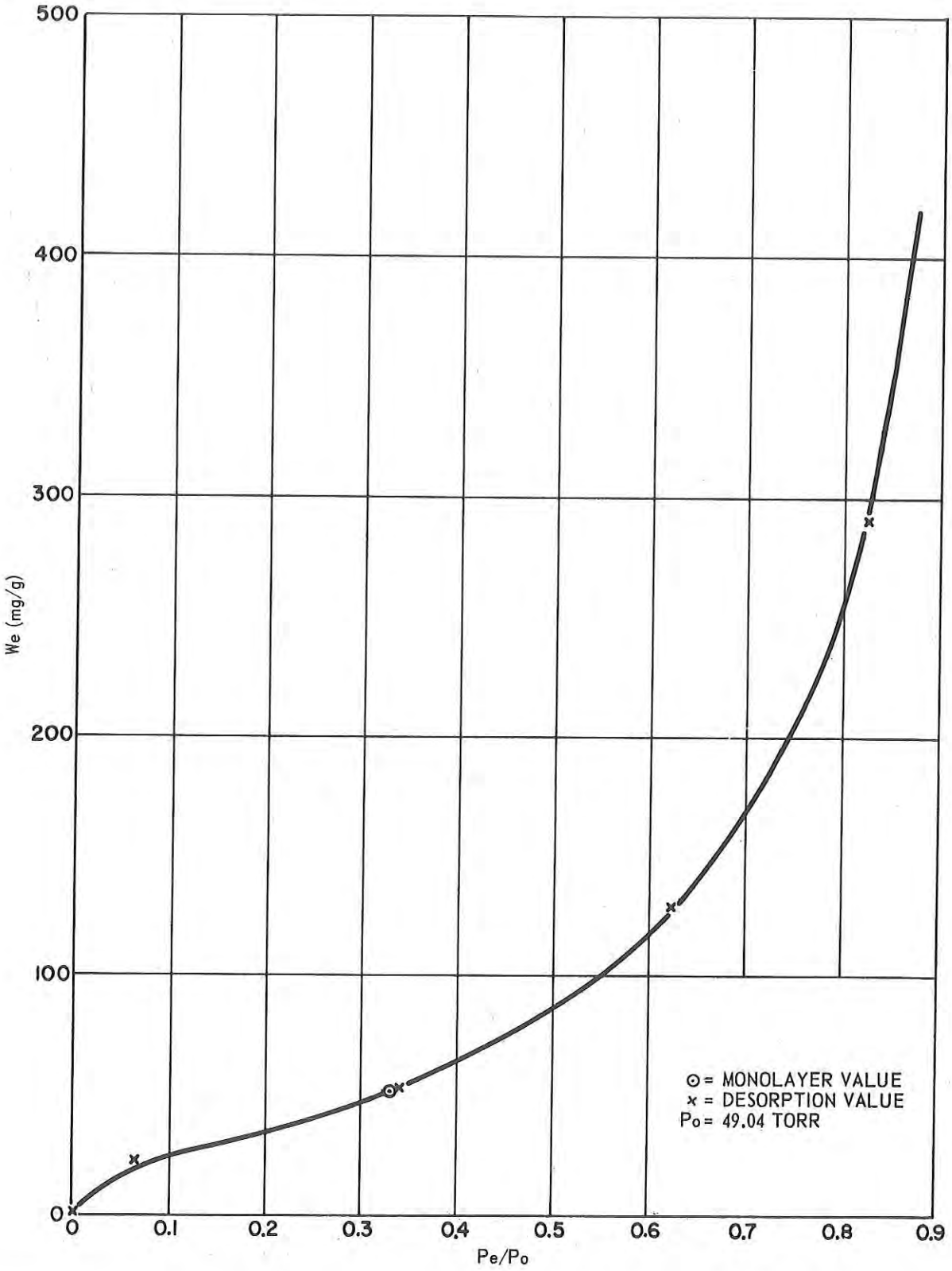


Figure 3. Moisture Sorption Isotherm for Freeze-Dried Spinach Powder at 37.77°C

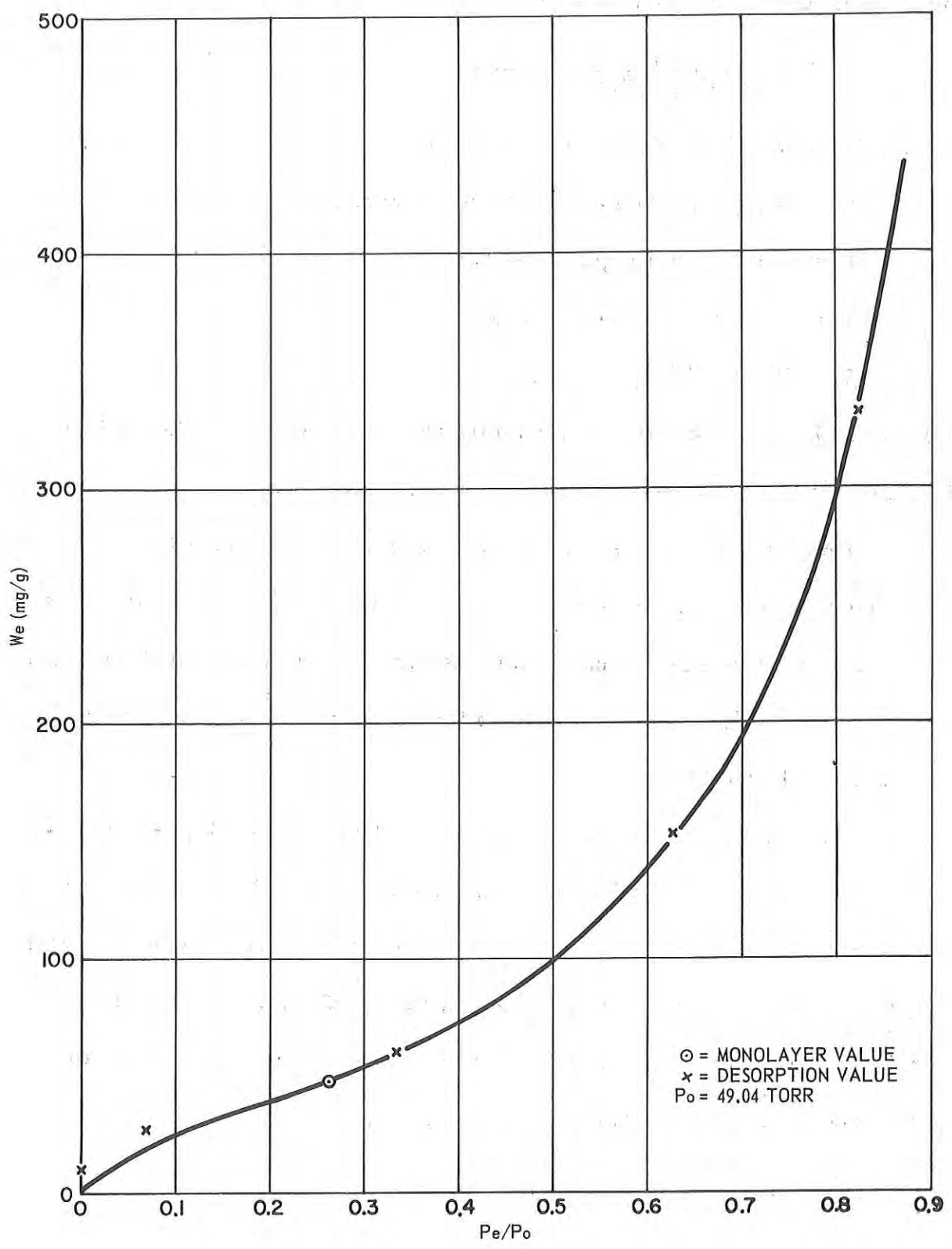


Figure 4. Moisture Sorption Isotherm for Freeze-Dried Sweet Potato Powder at 37.77°C

The BET isotherm equation was used:

$$We = \frac{WmCP}{(Po-P)\,(1+(C-1)P/Po)} \qquad (1)$$

where We = weight sorbed at equilibrium

Wm = weight sorbed at monolayer coverage

P = equilibrium vapor pressure

P0 = saturation vapor pressure

$$C = K\exp.\ \left(\left(E_a - E_b\right)/RT\right) \qquad (2)$$

In equation 2, K is a constant approximately equal to one, $E_a$ is the heat of sorption and $E_b$ the heat of liquefaction of the vapor.

Although a monolayer value can be obtained from the BET equation, this equation has severe limitations. It is, however, usually valid between 0.1 and 0.5 relative pressure. In the case of a swelling gel (such as food), the number of interior sites may be so large that the number of exterior sites are negligible.

The BET monolayer values were calculated by plotting P/We(Po-P) vs P/Po. This gave a straight line whose slope is equal to (c-1)/WmC and intercept equal to 1/WmC. These values were used to calculate BET water surface areas by assuming a cross section area of the water molecule of $10.8A^{0^2}$. The values for Wm and the surface areas for the original and stored foods are given in Tables 28 through 33.

TABLE 28

Equilibrium Moisture Sorption Values at 22.0°C
At the BET Monolayer for Freeze-Dried Beef

| * | Original | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 3.5%$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 34.4 | 41.9 | 36.1 | 36.1 | 39.0 | 36.2 | 37.0 |
| S.A.($m^2$/g) | 124 | 151 | 130 | 130 | 141 | 131 | 134 |
| Pm/Po | 0.295 | 0.285 | 0.305 | 0.275 | 0.275 | 0.320 | 0.260 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 34.4 | 33.9 | 21.9 | 34.4 | 32.2 | 24.1 | 27.0 |
| S.A.($m^2$/g) | 124 | 123 | 79 | 124 | 116 | 87 | 98 |
| Pm/Po | 0.295 | 0.305 | 0.210 | 0.280 | 0.270 | 0.285 | 0.280 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 34.4 | 26.2 | 27.9 | 47.2 | 31.4 | 21.6 | 31.8 |
| S.A.($m^2$/g) | 124 | 95 | 101 | 171 | 114 | 78 | 115 |
| Pm/Po | 0.295 | 0.285 | 0.230 | 0.305 | 0.270 | 0.125 | 0.210 |

* Wm(mg/g) is the milligrams of moisture sorbed per gram of dry food at the BET monolayer.

S.A.($m^2$/g) is the water surface area of the food (in meters $^2$/gram) at the BET monolayer.

Pm/Po is the relative pressure at the BET monolayer.

TABLE 29

Equilibrium Moisture Sorption Values at 37.8°C
At the BET Monolayer for Freeze-Dried Beef

| * | Original | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 3.5$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 34.5 | 33.7 | 33.2 | 32.6 | 32.3 | 29.1 | 31.8 |
| S.A.($m^2$/g) | 125 | 121.7 | 119.8 | 117.9 | 116.8 | 105.0 | 115.0 |
| Pm/Po | 0.280 | 0.323 | 0.330 | 0.317 | 0.296 | 0.304 | 0.312 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 34.5 | 35.2 | 23.6 | 33.1 | 36.1 | 28.5 | 30.4 |
| S.A.($m^2$/g) | 125 | 127 | 101 | 120 | 131 | 103 | 109 |
| Pm/Po | 0.280 | 0.265 | 0.205 | 0.285 | 0.285 | 0.275 | 0.300 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 34.5 | 25.1 | 30.4 | 30.6 | 29.6 | 25.8 | 30.1 |
| S.A.($m^2$/g) | 125 | 91 | 110 | 111 | 107 | 93 | 109 |
| Pm/Po | 0.280 | 0.230 | 0.260 | 0.280 | 0.275 | 0.310 | 0 |

* Wm(mg/g) is the milligrams of moisture sorbed per gram of dry food at the BET monolayer.

S.A.($m^2$/g) is the water surface area of the food (in meters 2/gram) at the BET monolayer.

Pm/Po is the relative pressure at the BET monolayer.

TABLE 30

Equilibrium Moisture Sorption Values at 22.0°C at the BET Monolayer for Freeze-Dried Spinach

| * | Original | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.1%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.1%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 51.2 | 58.1 | 56.5 | 50.3 | 54.9 | 54.6 | 51.3 |
| S.A.($m^2$/g) | 185 | 210 | 204 | 182 | 199 | 198 | 185 |
| Pm/Po | 0.225 | 0.280 | 0.240 | 0.185 | 0.235 | 0.280 | 0.205 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 51.2 | 47.6 | 44.7 | 49.0 | 40.5 | 44.9 | 44.4 |
| S.A.($m^2$/g) | 185 | 172 | 161 | 177 | 147 | 162 | 161 |
| Pm/Po | 0.225 | 0.185 | 0.155 | 0.260 | 0.155 | 0.185 | 0.170 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 51.2 | 51.0 | 44.4 | 43.5 | 52.6 | 40.2 | 44.8 |
| S.A.($m^2$/g) | 185 | 184 | 161 | 157 | 190 | 145 | 162 |
| Pm/Po | 0.225 | 0.195 | 0.105 | 0.145 | 0.180 | 0.105 | 0.175 |

* Wm(mg/g) is the milligrams of moisture sorbed per gram of dry food at the BET monolayer.

S.A.($m^2$/g) is the water surface area of the food (in meters$^2$/gram) at the BET monolayer.

Pm/Po is the relative pressure at the BET monolayer.

TABLE 31

Equilibrium Moisture Sorption Values at 37.8°C at the BET Monolayer for Freeze-Dried Spinach

| * | Original | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.1%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.1%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 50.3 | 54.9 | 43.1 | 39.4 | 50.8 | 50.0 | 42.9 |
| S.A.($m^2$/g) | 182 | 199 | 156 | 142 | 183 | 181 | 155 |
| Pm/Po | 0.325 | 0.270 | 0.210 | 0.245 | 0.280 | 0.275 | 0.265 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 50.3 | 53.5 | 49.0 | 48.3 | 51.0 | 43.9 | 44.7 |
| S.A.($m^2$/g) | 182 | 193 | 177 | 175 | 184 | 159 | 161 |
| Pm/Po | 0.325 | 0.265 | 0.230 | 0.240 | 0.285 | 0.240 | 0.220 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 50.3 | 50.3 | 36.8 | 34.9 | 48.8 | 47.8 | 45.1 |
| S.A.($m^2$/g) | 182 | 182 | 133 | 126 | 176 | 173 | 163 |
| Pm/Po | 0.325 | 0.245 | 0.245 | 0.135 | 0.130 | 0.230 | 0.250 |

* Wm(mg/g) is the milligrams of moisture sorbed per gram of dry food at the BET monolayer. S.A.($m^2$/g) is the water surface area of the food (in meters$^2$/gram) at the BET monolayer. Pm/Po is the relative pressure at the BET monolayer.

TABLE 32

Equilibrium Moisture Sorption Values at 22.0°C at the BET Monolayer for Freeze-Dried Sweet Potatoes

| * | Original | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 39.8 | 49.5 | 39.0 | 38.5 | 48.8 | 39.7 | 32.3 |
| S.A.($m^2$/g) | 114 | 178 | 141 | 102 | 176 | 143 | 116 |
| Pm/Po | 0.290 | 0.295 | 0.255 | 0.265 | 0.295 | 0.275 | 0.320 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 39.8 | 32.8 | 35.7 | 22.4 | 27.9 | 38.8 | 29.6 |
| S.A.($m^2$/g) | 144 | 118 | 128 | 83 | 101 | 140 | 107 |
| Pm/Po | 0.290 | 0.255 | 0.250 | 0.265 | 0.225 | 0.275 | 0.300 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 39.8 | 31.4 | 32.5 | 15.5 | 32.3 | 27.7 | 17.7 |
| S.A.($m^2$/g) | 144 | 114 | 117 | 56 | 117 | 100 | 62 |
| Pm/Po | 0.290 | 0.285 | 0.275 | 0.225 | 0.275 | 0.170 | 0.100 |

*
Wm(mg/g) is the milligrams of moisture sorbed per gram of dry food at the BET monolayer

S.A.($m^2$/g) is the water surface area of the food (in meters$^2$/gram) at the BET monolayer.

Pm/Po is the relative pressure at the BET monolayer.

TABLE 33

Equilibrium Moisture Sorption Values at 37.8°C at the BET Monolayer for Freeze-Dried Sweet Potatoes.

| * | Original | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 46.8 | 57.0 | 37.2 | 29.5 | 51.4 | 38.5 | 24.3 |
| S:A.($m^2$/g) | 169 | 206 | 134 | 107 | 185 | 139 | 33 |
| Pm/Po | 0.265 | 0.320 | 0.260 | 0.265 | 0.305 | 0.270 | 0.265 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 46.8 | 40.2 | 33.0 | 30.7 | 38.7 | 44.4 | 33.7 |
| S.A.($m^2$/g) | 169 | 145 | 119 | 111 | 139 | 160 | 122 |
| Pm/Po | 0.265 | 0.255 | 0.275 | 0.265 | 0.260 | 0.295 | 0.290 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| Wm(mg/g) | 46.8 | 44.1 | 42.5 | 32.3 | 37.5 | 36.8 | 27.8 |
| S.A.($m^2$/g) | 169 | 160 | 154 | 117 | 135 | 133 | 100 |
| Pm/Po | 0.265 | 0.300 | 0.285 | 0.280 | 0.265 | 0.270 | 0.255 |

* Wm(mg/g) is the milligrams of moisture sorbed per gram of dry food at the BET monolayer.

S.A.($m^2$/g)is the water surface area of the food (in meters $^2$/gram at the BET monolayer.

Pm/Po is the relative pressure at the BET monolayer.

3.1.3 Fugassi - Mitchell Isotherm Values

Equation (3) was derived by Fugassi et al [7,8,9,10,11] to cover the case of swelling gels. This equation is valid for swelling gels over the entire relative pressure range.

$$We = \frac{AKK_1PoC}{\left[1+(K_1Po-1)C\right]\left[1-C\right]+KK_1PoC} \qquad (3)$$

Where We = weight of material sorbed at equilibrium (mg/g)

A = total equilibrium sorption at saturation pressure (mg/g)

K = equilibrium constant for the transfer reaction from the surface to the interior of the sample

$K_1$ = equilibrium constant for the sorption from the vapor phase onto the surface ($mm^{-1}$)

$P_o$ = saturation vapor pressure (mm)

C = relative pressure = $P/P_o$

The total amount of sorption occurring at saturation pressure as well as the equilibrium constants can be determined from this equation. Also, the relative importance of the surface and interior sites can be obtained from this equation.

The Fugassi-Mitchell Equation was solved using a summation method. Specifically, nine separate values of We were summed over three sections of the sorption isotherm: one group of values for low relative pressures, one group for medium relative pressures, and one group for high relative pressures. The three resulting equations are solved simultaneously to

give values of A, K and $K_1$. These values are shown in Tables 34 through 40 for original and stored foods.

Negative values of A and K are obtained for sweet potatoes and spinach. These negative values are meaningless. Presumably the negative values of A and K indicate that some chemical interations are occurring between water and the foods.

The Fugassi-Mitchell A and K values were used to $(H_2O)$ exterior and $(H_2O)$ interior. When the number of interior sorption sites is very much greater than the number of exterior sorption sites, the approximate relation

$$K = (H_2O)_{\text{exterior}} / (H_2O)_{\text{interior}} \qquad (4)$$

can be made. In addition, it is implicit in the Fugassi equation that

$$A = (H_2O)_{\text{interior}} + (H_2O)_{\text{exterior}} \qquad (5)$$

Equations (4) and (5) were solved simultaneously to give values for $(H_2O)$ exterior. A comparison of the monolayer sorption values calculated by the BET equation and the Fuggassi-Mitchell equation is shown in Tables 40 through 42.

TABLE 34

Value of the Fugassi-Mitchell Sorption Equation Constants
For Freeze-Dried Beef at 22.0°C

| * | Original | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 3.5%$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | 1,135 | 940 | 1,386 | 2,405 | 807 | 988 | 1,083 |
| K | 0.033 | 0.047 | 0.029 | 0.014 | 0.052 | 0.041 | 0.035 |
| $K_1$ ($mm^{-1}$) | 0.267 | 0.295 | 0.260 | 0.379 | 0.318 | 0.234 | 0.394 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | 1,135 | 681 | 6,060 | 3,597 | 633 | 499 | 926 |
| K | 0.033 | 0.063 | 0.005 | 0.011 | 0.047 | 0.073 | 0.032 |
| $K_1$ ($mm^{-1}$) | 0.267 | 0.176 | 0.222 | 0.256 | 0.197 | 0.238 | 0.303 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | 1,135 | 1,042 | 467 | -10,989 | 280 | 843 | -55,555 |
| K | 0.033 | 0.003 | 0.098 | -0.002 | 0.190 | 0.040 | -0.0004 |
| $K_1$ ($mm^{-1}$) | 0.267 | 0.229 | 0.182 | 0.822 | 0.124 | 0.305 | 3.78 |

TABLE 35

Values of the Fugassi-Mitchell Sorption Equation Constants
For Freeze-Dried Beef at 37.8°C

3 Weeks Storage at 37.8°C

| * | Original | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 3.5%$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| A(mg/g) | 834 | 372 | 453 | 542 | 472 | 586 | 479 |
| K | 0.044 | 0.115 | 0.090 | 0.070 | 0.082 | 0.058 | 0.078 |
| $K_1$ ($mm^{-1}$) | 0.123 | 0.064 | 0.065 | 0.075 | 0.090 | 0.089 | 0.079 |

7 Weeks Storage at 37.8°C

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| A(mg/g) | 834 | 354 | 593 | 417 | 334 | 473 | 453 |
| K | 0.044 | 0.115 | 0.051 | 0.088 | 0.139 | 0.079 | 0.074 |
| $K_1$ ($mm^{-1}$) | 0.123 | 0.121 | 0.135 | 0.135 | 0.114 | 0.096 | 0.102 |

11 Weeks Storage at 37.8°C

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| A(mg/g) | 834 | 549 | 617 | 978 | 462 | 388 | 381 |
| K | 0.044 | 0.068 | 0.068 | 0.033 | 0.093 | 0.113 | 0.106 |
| $K_1$ ($mm^{-1}$) | 0.123 | 0.098 | 0.094 | 0.132 | 0.074 | 0.044 | 0.059 |

TABLE 36

Values of the Fugassi-Mitchell Sorption Equation Constants
For Freeze-Dried Spinach at 22.0°C

3 Weeks Storage at 37.8°C

| * | Original | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.1%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.1%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| A(mg/g) | -1,122 | -4,370 | -3,570 | -1,010 | 13,880 | -52,630 | -1,740 |
| K | -0.044 | -0.013 | -0.015 | -0.050 | 0.004 | -0.001 | -0.027 |
| $K_1$ ($mm^{-1}$) | 0.652 | 0.295 | 0.355 | 0.693 | 0.416 | 0.409 | 1.058 |
| 7 Weeks Storage at 37.8°C | | | | | | | |
| A(mg/g) | -1,122 | -1,190 | -1,120 | 3,484 | 27,800 | -988 | -100,000 |
| K | -0.044 | -0.037 | -0.038 | 0.015 | 0.002 | -0.045 | -0.001 |
| $K_1$ ($mm^{-1}$) | 0.652 | 0.560 | 0.678 | 0.753 | 0.307 | 0.656 | 0.377 |
| 11 Weeks Storage at 37.8°C | | | | | | | |
| A(mg/g) | -1,122 | -1,940 | -4,000 | -3,720 | -876 | -1,610 | -1,115 |
| K | -0.044 | -0.023 | -0.013 | -0.067 | -0.047 | -0.033 | -0.041 |
| $K_1$ ($mm^{-1}$) | 0.652 | 1.015 | 0.389 | 0.409 | 1.629 | 0.591 | 1.242 |

TABLE 37

Values of the Fugassi-Mitchell Sorption Equation Constants for Freeze-Dried Spinach at 37.8°C

| * | Original | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.1%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.1%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | -2,083 | 3,846 | -1342 | 479 | -200,000 | -6993 | -2849 |
| K | -0.023 | 0.015 | -0.035 | 0.162 | -0,246 | -0.007 | -0.016 |
| $K_1(mm^{-1})$ | 0.111 | 0.128 | 0.219 | 0.063 | 0.173 | 0.182 | 0.018 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | -2,083 | -2,450 | -1,900 | 370 | -1,110 | -1,970 | -820 |
| K | -0.023 | -0.021 | -0.028 | 0.134 | -0.040 | -0.025 | -0.050 |
| $K_1(mm^{-1})$ | 0.111 | 0.078 | 0.161 | 0.076 | 0.349 | 0.157 | 0.420 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | -2,083 | -2,720 | -1,348 | -861 | -799 | -2,070 | -1,180 |
| K | -0.023 | -0.020 | -0.036 | -0.005 | -0.055 | -0.025 | -0.143 |
| $K_1(mm^{-1})$ | 0.111 | 0.149 | 0.095 | 0.295 | 1.333 | 0.186 | 0.206 |

TABLE 38

Values of the Fugassi-Mitchell Sorption Equation Constants
For Freeze-Dried Sweet Potato at 22.0°C

| * | Original | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| **3 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | -2,909 | -821 | -703 | -328 | -943 | -830 | -835 |
| K | -0.023 | -0.065 | -0.074 | -0.192 | -0.057 | -0.061 | -0.065 |
| $K_1$ ($mm^{-1}$) | 0.117 | 0.091 | 0.083 | 0.042 | 0.091 | 0.081 | 0.036 |
| **7 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | -2,909 | 319 | -3,105 | 5,208 | 298 | 1,694 | -9,523 |
| K | -0.023 | -0.819 | -0.021 | 0.017 | 1.357 | 0.055 | -0.007 |
| $K_1$ ($mm^{-1}$) | 0.117 | -0.017 | 0.117 | 0.044 | 0.014 | 0.078 | 0.060 |
| **11 Weeks Storage at 37.8°C** | | | | | | | |
| A(mg/g) | -2,909 | 726 | -2,512 | 1,412 | 909 | 3,236 | -3,144 |
| K | -0.023 | 0.196 | -0.023 | 0.066 | 0.119 | -0.017 | -0.016 |
| $K_1$ ($mm^{-1}$) | 0.117 | 0.035 | 0.127 | 0.034 | 0.050 | 0.173 | 0.103 |

TABLE 39

Values of the Fugassi-Mitchell Sorption Equation Constants
For Freeze-Dried Sweet Potato at 37.8°C

3 Weeks Storage at 37.8°C

| * | Original | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| A(mg/g) | -2,597 | -1,595 | 862 | 2,095 | -4,240 | -1,886 | 3,542 |
| K | -0.022 | -0.039 | 0.102 | 0.041 | -0.018 | -0.032 | 0.023 |
| $K_1(mm^{-1})$ | 0.101 | 0.071 | 0.036 | 0.022 | 0.064 | 0.054 | 0.025 |

7 Weeks Storage at 37.8°C

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| A(mg/g) | -2,597 | -3,257 | 2,141 | 851 | 1,545 | 1,953 | 820 |
| K | -0.022 | -0.016 | 0.031 | 0.110 | 0.048 | 0.038 | 0.099 |
| $K_1(mm^{-1})$ | 0.101 | 0.181 | 0.041 | 0.021 | 0.043 | 0.052 | 0.028 |

11 Weeks Storage at 37.8°C

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| A(mg/g) | -2,597 | 3,125 | -9,524 | 3,155 | 4,630 | 9,174 | 2,183 |
| K | -0.022 | 0.025 | -0.007 | 0.023 | 0.015 | 0.007 | 0.035 |
| $K_1(mm^{-1})$ | 0.101 | 0.036 | 0.057 | 0.027 | 0.051 | 0.044 | 0.028 |

TABLE 40
Comparison of the BET and Fugassi-Mitchell Monolayer Values at 22°C for Freeze-Dried Beef

| * | Original | 1.4%$H_2O$ under $N_2$ | 3.5%$H_2O$ under $N_2$ | 7.6%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 3.5%$H_2O$ under Air | 7.6%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| | | | 3 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 34.4 | 41.9 | 36.1 | 36.1 | 39.0 | 36.2 | 37.0 |
| F Wm(mg/g) | 36.3 | 42.2 | 39.1 | 33.2 | 39.9 | 38.9 | 36.6 |
| | | | 7 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 34.4 | 33.9 | 21.9 | 34.4 | 32.2 | 24.1 | 27.0 |
| F Wm(mg/g) | 36.3 | 40.4 | 30.2 | 39.5 | 28.4 | 33.95 | 28.7 |
| | | | 11 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 34.4 | 26.2 | 27.9 | 47.2 | 31.4 | 21.6 | 31.8 |
| F Wm(mg/g) | 36.3 | 3.1 | 41.7 | 22.0 | 44.7 | 32.4 | 22.2 |

Comparison of the BET and Fugassi-Mitchell Monolayer Values at 37.8°C for Freeze-Dried Beef

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | 3 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 34.5 | 33.7 | 33.2 | 32.6 | 32.3 | 29.1 | 31.8 |
| F Wm(mg/g) | 35.15 | 38.4 | 37.4 | 35.5 | 35.8 | 32.1 | 34.7 |
| | | | 7 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 34.5 | 35.2 | 23.6 | 33.1 | 36.1 | 28.5 | 30.4 |
| F Wm(mg/g) | 35.15 | 36.5 | 28.8 | 33.7 | 40.8 | 34.6 | 31.2 |
| | | | 11 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 34.5 | 25.1 | 30.4 | 30.6 | 29.6 | 25.8 | 30.1 |
| F Wm(mg/g) | 35.15 | 34.95 | 39.3 | 31.2 | 39.3 | 39.4 | 36.5 |

TABLE 41
Comparison of the BET and Fugassi-Mitchell Monolayer Values at 22.0°C for Freeze-Dried Spinach

| * | Original | 1.4%$H_2O$ under $N_2$ | 4.8%$H_2O$ under $N_2$ | 11.%$H_2O$ under $N_2$ | 1.4%$H_2O$ under Air | 4.8%$H_2O$ under Air | 11.%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| | | | 3 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 51.2 | 58.1 | 56.5 | 50.3 | 54.9 | 54.6 | 51.3 |
| F Wm(mg/g) | 51.6 | 57.6 | 54.4 | 53.2 | 55.3 | 52.7 | 48.3 |
| | | | 7 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 51.2 | 47.6 | 44.7 | 49.0 | 40.5 | 44.9 | 44.4 |
| F Wm(mg/g) | 51.6 | 45.7 | 44.2 | 51.5 | 55.5 | 46.6 | 100.1 |
| | | | 11 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 51.2 | 51.0 | 44.4 | 43.5 | 52.6 | 40.2 | 44.8 |
| F Wm(mg/g) | 51.6 | 45.7 | 52.7 | 267.1 | 43.2 | 54.9 | 47.7 |

Comparison of the BET and Fugassi-Mitchell Monolayer Values at 37.8°C for Freeze-Dried Spinach

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | 3 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 50.3 | 54.9 | 43.1 | 39.4 | 50.8 | 50.0 | 42.9 |
| F Wm(mg/g) | 49.0 | 56.8 | 48.7 | 66.8 | 48.5 | 49.3 | 46.3 |
| | | | 7 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 50.3 | 53.5 | 49.0 | 48.3 | 51.0 | 43.9 | 44.7 |
| F Wm(mg/g) | 49.0 | 52.6 | 54.7 | 43.7 | 46.3 | 50.5 | 43.2 |
| | | | 11 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 50.3 | 50.3 | 36.8 | 34.9 | 48.8 | 47.8 | 45.1 |
| F Wm(mg/g) | 49.0 | 55.5 | 50.3 | 4.3 | 46.5 | 53.1 | 52.0 |

TABLE 42

Comparison of the BET and Fugassi-Mitchell Monolayer Values at 22.0°C for Freeze Dried Sweet Potato

| * | Original | 1.2%$H_2O$ under $N_2$ | 4.4%$H_2O$ under $N_2$ | 11.0%$H_2O$ under $N_2$ | 1.2%$H_2O$ under Air | 4.4%$H_2O$ under Air | 11.0%$H_2O$ under Air |
|---|---|---|---|---|---|---|---|
| | | | 3 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 39.8 | 49.5 | 39.0 | 38.5 | 48.8 | 39.7 | 32.3 |
| F Wm(mg/g) | 68.5 | 57.1 | 56.2 | 77.9 | 57.0 | 53.9 | 58.0 |
| | | | 7 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 39.8 | 32.8 | 35.7 | 22.4 | 27.9 | 38.8 | 29.6 |
| F Wm(mg/g) | 68.5 | | 66.6 | 87.1 | 111.2 | 88.3 | 67.1 |
| | | | 11 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 39.8 | 31.4 | 32.5 | 15.5 | 32.3 | 27.7 | 17.7 |
| F Wm(mg/g) | 68.5 | 118.98 | 59.1 | 87.4 | 96.7 | 63.8 | 51.1 |

Comparison of the BET and Fugassi-Mitchell Monolayer Values at 37.8°C for Freeze-Dried Sweet Potato

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | 3 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 46.8 | 57.0 | 37.2 | 29.5 | 51.4 | 38.5 | 24.3 |
| F Wm(mg/g) | 58.4 | 64.7 | 79.8 | 82.5 | 77.7 | 62.3 | 79.6 |
| | | | 7 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 46.8 | 40.2 | 33.0 | 30.7 | 38.7 | 44.4 | 33.7 |
| F Wm(mg/g) | 58.4 | 53.0 | 64.4 | 84.3 | 70.8 | 71.5 | 73.9 |
| | | | 11 Weeks Storage at 37.8°C | | | | |
| BET Wm(mg/g) | 46.8 | 44.1 | 42.5 | 32.3 | 37.5 | 36.8 | 27.8 |
| F Wm(mg/g) | 58.4 | 76.2 | 67.1 | 70.9 | 68.4 | 63.8 | 73.8 |

3.1.4 Heats of Sorption

In the calculation of the heats of sorption for the different foods, Clausius-Clapeyron plots were made at a constant amount of moisture sorbed[12] by making the assumption that the heat of sorption was constant in the temperature range 22°C to 37.8°C. These plots were isosteres where ln P was plotted as 1/T in degrees Kelvin. The slope of the line was equal to $-\Delta H/R$, where R is the gas constant and $\Delta H$ is the heat of sorption.

The heats of sorption of the foods used in this study are given in Tables 43 through 44. These values are reported for different surface coverages ranging from less than monolayer coverage up to multilayer coverage. As more sorbate molecules are sorbed on the surface, the heat of interaction approaches the heat of liquefaction of water (10.4 Kcal/Mole at 25°C).

The heats of absorption for the first monolayer can, however, be greater or less than the heat of liquefaction of the vapor.[13] In the former case a Type III isotherm will result. The BET constant C will be equal to one, and the resultant isotherm will be a simple experimental curve.

The measurements of the heats of sorption in the stored foods were meaningless. In many cases, inversions occurred in the isotherms which made it impossible to get $\Delta H$ values for the stored foods.

TABLE 43

ISOTHERMAL HEATS OF SORPTION OF WATER VAPOR ON FREEZE-DRIED FOOD POWDER IN THE TEMPERATURE RANGE 22°C TO 37.77°C

| Beef | | Spinach | | Sweet Potato | |
|---|---|---|---|---|---|
| Wt. of Moisture Sorbed (mg/g) | $-\Delta H$ (kcal/mole) | Wt. of Moisture Sorbed (mg/g) | $-\Delta H$ (kcal/mole | Wt. of Moisture Sorbed (mg/g) | $-\Delta H$ (kcal/mole) |
| 15 | 9.4 | 25 | 5.7 | 20 | 23.2 |
| 25 | 10.0 | 39.8 | 6.9 | 35 | 20.9 |
| 34.5 | 9.8 | 46.8 | 8.0 | 51.0 | 14.7 |
| 50 | 10.3 | 75 | 9.9 | 75 | 12.4 |
| 100 | 10.6 | 100 | 10.2 | 100 | 11.7 |
| 200 | 10.6 | 200 | 10.6 | 200 | 10.9 |

TABLE 44

HEATS OF SORPTION CALCULATED FROM BET "C" VALUES

| Food | 37.77°C Isotherm | | 22°C Isotherm | |
|---|---|---|---|---|
| | C | $E_a$ (kcal/mole) | C | $E_a$ (kcal/mole) |
| Beef | 6.60 | 11.5 | 5.98 | 11.5 |
| Spinach | 9.09 | 11.3 | 6.24 | 11.9 |
| Sweet Potato | 4.50 | 11.7 | 13.0 | 11.5 |

## 3.2 Chemical Evaluation of Stored Foods

### 3.2.1 Freeze-Dried Sweet Potatoes

#### 3.2.1.1 Freeze-Dried Sweet Potatoes Stored at 25°C

The data for the chemical evaluation of freeze-dried sweet potatoes stored at 25°C is given in Table 45.

For sweet potatoes stored for 3 weeks at 25°C, the sample stored in air with a moisture content of 1.2% differed from the other samples in that there was a loss in color. The aqueous extract was colorless while the extracts from the other samples closely resembled orange juice in appearance.

The sweet potatoes packaged in air at a moisture content of 3.8% were also different in that on extraction with water filtration was very slow. This might be due to a fracturing of the sweet potato tissue. The small particles so formed tended to clog the pores of the filter paper. Oxygen undoubtedly played a part in the process.

The greater the moisture content at which the food was stored, the greater the loss in amino nitrogen and concomitant increase in the titratable acidity. The loss was less in the food packaged under nitrogen.

Minor changes occurred in the texture of the food with a moisture content of 1.2%. Above the monolayer moisture level, however, the texture of the food was ruined. These changes in texture occurred practically to the same extent in the food stored under nitrogen as when packaged in air.

TABLE 45

CHEMICAL EVALUATION OF FREEZE-DRIED SWEET POTATOES AT 25°C *

| | Composition of Food per 100 grams Anhydrous Sweet Potatoes Extraction of Sweet Potatoes with Water | | | | | | | | | | | | | | Titration of the Aqueous Extract with 0.01 N Iodine Solution Results Expressed as Mgs Ascorbic Acid/100 grams Food. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Formol Titration of Residue | | | | Formol Titration of Aq. Extract | | | | | | | Reducing Substances reacted With Iodine 20 sec. | | | | | Reducing Sustances reacted With Iodine over 10 min. Period | | | | |
| | Initial % Water | % Water | Color | pH Slurry | N NaOH to pH 8.5 | Versene N NaOH to pH 8.5 | Mgs Amine Nit. | pH | N NaOH to pH 8.5 | Versene N NaOH to pH 8.5 | Mgs Amine Nit. | Total Titratable Acidity | Total Versene N NaOH to pH 8.5 | Total Mgs. Amine Nit. | No Additives | Formalin | Versene | Formalin & Versene | Average | No Additives | Formalin | Versene | Formalin & Versene | Average |
| | | | | | | | | | | | | 3 Weeks Analyzed September 29, 1965 | | | | | | | | | | | | |
| Ref 4.5% R.H. Air | 1.2 | | Or | | | | | 5.8 | 11.6 | 4.5 | 118 | | | | 46 | | | | | 134 | | | | |
| 4.5%R.H. | 1.2 | 1.4 | Pink | 5.8 | .8 | 1.5 | 14 | 5.8 | 11.1 | 3.4 | 120 | 11.9 | 4.9 | 134 | 46 | | | | | 141 | | | | |
| 21.5%R.H. | 3.8 | 4.3 | Or | 5.9 | .8 | 1.6 | 14 | 5.8 | 11.6 | 4.1 | 109 | 12.4 | 5.7 | 123 | 48 | | | | | 145 | | | | |
| 58.2%R.H. $N_2$ | 11.0 | 11.1 | Or | 5.9 | .8 | 1.4 | 11 | 5.8 | 12.1 | 3.8 | 108 | 12.9 | 5.2 | 119 | 31 | | | | | 145 | | | | |
| 6.5%R.H. | 1.6 | 1.6 | Or | 5.9 | .7 | 1.3 | 12 | 5.8 | 12.1 | 3.8 | 116 | 12.8 | 5.1 | 128 | 57 | | | | | 147 | | | | |
| 27.5%R.H. | 3.8 | 4.4 | Or | 5.9 | .8 | 1.6 | 13 | 5.8 | 11.8 | 3.5 | 115 | 12.6 | 5.1 | 128 | 37 | | | | | 125 | | | | |
| 58.0%R.H. | 10.9 | 10.9 | Or | 6.0 | .8 | 1.5 | 13 | 5.8 | 12.2 | 5.3 | 108 | 13.0 | 6.8 | 121 | 35 | | | | | 130 | | | | |
| | | | | | | | | | | | | 7 Weeks Analyzed November 20, 1965 | | | | | | | | | | | | |
| Ref. 4.5%R.H. Air | 1.2 | | Or | 5.3 | 1.4 | .7 | 17 | 5.7 | 11.4 | 3.8 | 125 | 12.8 | 4.5 | 142 | 48 | 53 | 48 | 42 | 48 | 132 | 167 | 123 | 130 | 138 |
| 4.5%R.H. | 1.2 | 1.6 | Wh Pink Tint | 5.3 | 1.1 | 1.0 | 13 | 5.6 | 12.0 | 3.1 | 117 | 13.1 | 4.1 | 130 | 70 | 57 | 66 | 61 | 64 | 176 | 194 | 163 | 172 | 176 |
| 21.5%R.H. | 3.8 | 4.7 | Or | 5.7 | 1.1 | .9 | 14 | 5.7 | 11.2 | 2.7 | 125 | 12.3 | 3.6 | 139 | 44 | 62 | 53 | 70 | 57 | 128 | 176 | 136 | 163 | 151 |
| 58.2%R.H. $N_2$ | 11.0 | 11.0 | Tan | 5.3 | 1.1 | 1.0 | 15 | 5.7 | 12.8 | 4.2 | 104 | 13.9 | 5.2 | 119 | 18 | 58 | 40 | 44 | 39 | 119 | 167 | 128 | 141 | 139 |
| 6.5%R.H. | 1.6 | 1.8 | Or | 5.3 | 1.1 | 1.1 | 17 | 5.7 | 11.3 | 2.5 | 125 | 12.4 | 3.6 | 142 | 57 | 48 | 66 | 79 | 63 | 145 | 172 | 150 | 172 | 159 |
| 27.5%R.H. | 3.8 | 4.4 | Or | 5.6 | 1.0 | 1.1 | 16 | 5.7 | 11.6 | 3.4 | 118 | 12.8 | 4.2 | 128 | 53 | 57 | 42 | 81 | 63 | 141 | 158 | 132 | 178 | 152 |
| 58.0%R.H. | 10.9 | 10.8 | Tan | 5.4 | 1.0 | 1.2 | 15 | 5.6 | 12.7 | 2.4 | 116 | 13.7 | 3.6 | 131 | 44 | 57 | 44 | 40 | 46 | 136 | 189 | 132 | 141 | 150 |
| | | | | | | | | | | | | 11 Weeks Analyzed November 16, 1965 | | | | | | | | | | | | |
| Ref. 4.5%R.H. Air | 1.2 | | Or | 5.3 | 1.1 | 1.8 | 16 | 5.6 | 11.8 | 4.7 | 107 | 12.9 | 6.5 | 123 | 64 | 35 | 57 | 40 | 49 | 178 | 130 | 155 | 155 | 155 |
| 4.5%R.H. | 1.2 | 1.4 | Wh-Pink Pink | 5.6 | 1.1 | 1.5 | 15 | 5.7 | 11.8 | 3.3 | 114 | 12.9 | 4.8 | 129 | 55 | 79 | 79 | 53 | 66 | 188 | 180 | 220 | 170 | 190 |
| 21.5%R.H | 3.8 | 4.6 | Wh-Or Tint | 5.3 | 1.0 | 1.9 | 14 | 5.6 | 11.7 | 3.3 | 100 | 12.7 | 5.2 | 114 | 46 | 35 | 57 | 62 | 50 | 163 | 115 | 165 | 180 | 156 |
| 58.2%R.H. $N_2$ | 11.0 | 10.7 | Or-Gn Tint | 5.3 | 1.0 | 1.7 | 12 | 5.5 | 13.5 | 3.6 | 92 | 14.5 | 5.3 | 104 | 51 | 44 | 31 | 22 | 37 | 180 | 155 | 155 | 145 | 159 |
| 6.5%R.H. | 1.6 | 1.7 | Pink | 5.3 | 1.0 | 1.8 | 15 | 5.7 | 11.4 | 4.8 | 92 | 12.4 | 6.6 | 107 | 42 | 88 | 53 | 62 | 61 | 168 | 195 | 175 | 165 | 176 |
| 27.5%R.H. | 3.8 | 4.7 | Or | 5.7 | 1.0 | 1.9 | 16 | 5.7 | 11.3 | 3.7 | 118 | 12.3 | 5.6 | 134 | 59 | 22 | 44 | 26 | 38 | 175 | 135 | 140 | 125 | 144 |
| 58.0%R.H. | 10.9 | 10.4 | Or | 5.2 | 1.0 | 1.9 | 12 | 5.6 | 13.3 | 3.5 | 104 | 14.3 | 5.4 | 116 | 37 | 30 | 79 | 44 | 48 | 163 | 140 | 200 | 160 | 166 |

* Placed in Storage August 20, 1965

In sweet potatoes stored 7 weeks at 25°C, a formol titration of the aqueous extract stored in air and under nitrogen above the monolayer moisture level and below the monolayer value in air indicated a reduction in amino nitrogen. The titratable acidity of these samples was also elevated.

The titration of the aqueous extracts with iodine showed significant changes due to the formation and/or destruction of reducing compounds. There are several classes of reducing compounds in sweet potatoes either normally present or capable of appearing on storage. Substances capable of being oxidized by iodine are vitamin C, stannous and ferrous sulfate, sulfhydryl compounds, sulfides, thiosulfates, reductinic acid and reductones, pentoses, dextrins and saturated or unsaturated aldehydes, either aromatic or aliphatic.

An aliquot of the extract was titrated with iodine in the presence of formalin, Versens, a combination of Versene and formalin and without the presence of any additives. Versene did not seem to change the amount of iodine required probably because the concentration of cations was not large enough to associate with a sufficient number of groups to block their oxidation by iodine. Formalin, however, consistently increased the amount of iodine required. This may be accounted for by an association between amino and aldehyde groups. The formalin reacts with the amino groups freeing the aldehyde groups which can then react with the iodine.

Titration of the aqueous extract with iodine does not give a sharp end point because of the large number of compounds reacting with different reaction rates, the difficulty of arriving at the end point in a specified length of time, namely 20 seconds, and the difficulty of arriving at the end point with the same intensity of blue color. However, the data are believed to be quite accurate and reproducible when 4 titrations are averaged.

The sweet potatoes stored in air at an initial moisture content of 1.2% had an enhanced iodine titration value. There was an almost complete disappearance of the pigment of the sweet potatoes and the compounds involved are believed to be formed from the oxidation of these pigments.

The food stored in air with an initial moisture content of 11.0% had a reduced iodine titration value (20 second reaction time) which was due to the loss of ascorbic acid. At the monolayer moisture level in air, the pigment of the sweet potatoes visually appeared stable and the ascorbic acid seemed stable.

Sweet potatoes stored 11 weeks at 25°C under nitrogen at the monolayer moisture level appeared to be stable according to the chemical analysis and by appearance. It did, however, increase in moisture 0.9% which means that chemical reactions were taking place but the analyses performed did not measure the reactions involved.

The sweet potatoes stored below the monolayer moisture level under nitrogen changed in color from an orange to a pink. The aqueous extract had an elevated iodine titration value. Even though the food was packaged under nitrogen there was a small amount of oxygen absorbed and dissolved in the food. Since the amount of lipids in sweet potatoes was small, there was an ample amount of oxygen to react with them.

The food stored above the monolayer under nitrogen had an elevated titratable acidity accompanied by a loss of amino nitrogen. Although their color did not change, the data from Phase I of this contract showed that at this moisture level the lipids decompose through oxidative-hydrolytic reactions. The products enter into reactions which change the texture of the sweet potatoes so that they are hard and rubbery.

The sweet potatoes stored in air also showed extensive degradation through lipid oxidation and/or hydrolysis. The products so formed then took part in a wide variety of chemical reactions.

3.2.1.2 Freeze-Dried Sweet Potatoes Stored at 37.8°C

Data on the chemical evaluation of freeze-dried sweet potatoes stored at 37.8°C is given in Table 46. It was found that the general rule which states that a chemical reaction increases in velocity by a factor of 2 to 3 for each 10°C rise in temperature does not apply to the decline in amino nitrogen. The amino nitrogen of the sweet potatoes stored with an initial moisture content of 10.9% fell 6.9 times faster at 37.8°C than at 25°C.

Table 46

CHEMICAL EVALUATION OF FREEZE-DRIED SWEET POTATOES AT 37.8°C*

Composition of Food per 100 grams Anhydrous Sweet Potatoes
Extraction of Sweet Potatoes with Water

Titration of the Aqueous Extract with 0.01 N Iodine Solution
Results Expressed as Mgs Ascorbic Acid/100 grams Food

| Initial Relative Humidity | Initial % Water | % Water | Color | Formol Titration of Residue: pH Slurry | N NaOH to pH 8.5 | Versene N NaOH to pH 8.5 | Mgs Amine Nit. | Formol Titration of Aq. Extract: pH | N NaOH to pH 8.5 Versene N NaOH to pH 8.5 | Mgs Amine Nit. | Total Titratable Acidity | Total Versene N NaOH to pH 8.5 | Total Mgs. Amine Nit. | Reducing Sustances reacted With Iodine 20 sec.: No Additives | Formolin | Versene | Formolin & Versene | Average | Reducing Sustances reacted With Iodine over 10 min. Period: No Additives | Formolin | Versene | Formolin & Versene | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 Weeks Analyzed October 3, 1965 | | | | | | | | | | | | | | | | | | | | | | | |
| Ref. 4.5%R.H. | 1.2 | | Or | 6.0 | .8 | 1.6 | 10 | 5.8 | 11.0 | 3.3 | 118 | 11.8 | 4.9 | 128 | 53 | 24 | | 18 | 32 | 114 | 110 | | 110 | 111 |
| Air | | | | | | | | | | | | | | | | | | | | | | | |
| 4.5%R.H. | 1.2 | 1.8 | Wh-faint Bn | 6.0 | .8 | 1.6 | 10 | 5.8 | 11.3 | 4.3 | 109 | 12.1 | 5.9 | 119 | 48 | 40 | | 53 | 47 | 119 | 141 | | 174 | 145 |
| 21.5%R.H. | 4.5 | 4.9 | Or-faint Bn | 5.7 | .8 | 1.5 | 9 | 5.7 | 13.9 | 3.8 | 81 | 14.7 | 5.3 | 90 | 26 | 42 | | 35 | 28 | 114 | 97 | | 143 | 118 |
| 58.2%R.H. | 11.0 | 11.1 | Or-faint Bn | 5.8 | 1.3 | 1.5 | 10 | 5.6 | 16.2 | 3.0 | 67 | 17.5 | 4.5 | 77 | 22 | 37 | | 37 | 32 | 141 | 114 | | 158 | 138 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | |
| 6.5%R.H. | 1.6 | 1.2 | Or | 5.9 | .8 | 1.4 | 12 | 5.8 | 11.7 | 3.7 | 115 | 12.5 | 5.0 | 127 | 53 | 24 | | 22 | 33 | 114 | 101 | | 132 | 116 |
| 27.5%R.H. | 4.4 | 5.3 | Or | 5.6 | .9 | 1.7 | 11 | 5.7 | 13.5 | 3.5 | 87 | 14.4 | 5.2 | 98 | 37 | 13 | | 37 | 29 | 106 | 79 | | 161 | 115 |
| 58.0%R.H. | 10.9 | 10.9 | Or | 5.9 | .9 | 1.5 | 11 | 5.6 | 15.8 | 3.3 | 73 | 16.7 | 4.8 | 84 | 40 | 44 | | 37 | 40 | 208 | 149 | | 172 | 176 |
| 7 Weeks Analyzed December 6, 1965 | | | | | | | | | | | | | | | | | | | | | | | |
| Ref. 4.5%R.H. | 1.2 | | Or | 5.5 | 1.0 | 1.4 | 18 | 5.7 | 11.4 | 1.8 | 122 | 12.4 | 3.2 | 140 | 40 | 22 | 53 | 31 | 37 | 114 | 119 | 110 | 101 | 111 |
| Air | | | | | | | | | | | | | | | | | | | | | | | |
| 4.5%R.H. | 1.2 | 1.8 | Wh-pink tint | 5.5 | 1.0 | 1.3 | 18 | 5.7 | 11.4 | 1.5 | 116 | 12.4 | 2.8 | 134 | 31 | 40 | 66 | 40 | 44 | 101 | 150 | 141 | 123 | 129 |
| 21.5%R.H. | 4.5 | 5.4 | Lt tan Gr tint | 5.7 | 1.0 | 1.2 | 18 | 5.6 | 11.5 | 2.5 | 77 | 12.5 | 3.7 | 95 | 13 | 26 | 31 | 26 | 24 | 101 | 114 | 119 | 110 | 111 |
| 58.2%R.H. | 11.0 | 11.4 | Tan | 5.2 | 1.4 | 1.4 | 18 | 5.4 | 17.7 | 2.0 | 55 | 19.1 | 3.4 | 73 | 31 | 57 | 101 | 75 | 66 | 132 | 180 | 198 | 163 | 168 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | |
| 6.5%R.H | 1.6 | 1.8 | Light Or | 5.5 | 1.0 | 1.4 | 19 | 5.7 | 11.4 | 1.7 | 117 | 12.4 | 3.1 | 136 | 35 | 22 | 53 | 18 | 32 | 101 | 110 | 123 | 92 | 107 |
| 27.5%R.H. | 4.4 | 3.8 | Or | 5.5 | 1.0 | 1.1 | 19 | 5.6 | 14.2 | 2.1 | 84 | 15.2 | 3.2 | 103 | 26 | 22 | 35 | 26 | 27 | 106 | 123 | 123 | 106 | 115 |
| 58.0%R.H. | 10.9 | 11.2 | Tan | 5.3 | 1.3 | 1.2 | 17 | 5.5 | 17.5 | 3.1 | 54 | 18.8 | 4.3 | 71 | 72 | 40 | 53 | 66 | 58 | 163 | 163 | 163 | 158 | 162 |
| 11 Weeks Analyzed December 13, 1965 | | | | | | | | | | | | | | | | | | | | | | | |
| Ref. 4.5%R.H. | 1.2 | | Or | 5.5 | 1.1 | 1.5 | 20 | 5.7 | 11.5 | 3.4 | 112 | 12.6 | 4.9 | 132 | 48 | 31 | 57 | 57 | 48 | 110 | 136 | 132 | 145 | 131 |
| Air | | | | | | | | | | | | | | | | | | | | | | | |
| 4.5%R.H. | 1.2 | 1.8 | Wh | 5.6 | 1.0 | 1.5 | 20 | 5.7 | 11.7 | 2.3 | 112 | 12.7 | 3.8 | 132 | 36 | 26 | 44 | 35 | 35 | 106 | 120 | 114 | 128 | 117 |
| 21.5%R.H | 4.5 | 4.8 | Wh-gr tint | 5.5 | 1.1 | 1.5 | 17 | 5.6 | 15.5 | 3.6 | 56 | 16.6 | 5.1 | 73 | 22 | 31 | 35 | 40 | 32 | 101 | 132 | 119 | 172 | 131 |
| 58.2%R.H. | 11.0 | 11.0 | tan-gr tint | 5.2 | 1.5 | 1.7 | 18 | 5.3 | 14.2 | 2.9 | 42 | 15.7 | 4.6 | 60 | 66 | 88 | 79 | 75 | 77 | 163 | 198 | 185 | 158 | 176 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | |
| 6.5%R.H. | 1.6 | 1.7 | Lt.Or. | 5.6 | 1.0 | 1.8 | 20 | 5.7 | 11.5 | 3.4 | 118 | 12.5 | 5.2 | 138 | 48 | 22 | 57 | 40 | 42 | 106 | 109 | 119 | 128 | 118 |
| 27.5%R.H. | 4.4 | 5.4 | Or | 5.5 | 1.1 | 1.5 | 20 | 5.6 | 15.0 | 2.5 | 64 | 16.1 | 4.0 | 84 | 22 | 26 | 40 | 53 | 35 | 97 | 123 | 128 | 141 | 122 |
| 58.0%R.H. | 10.9 | 10.9 | Tan-or brn | 5.3 | 1.4 | 1.8 | 16 | 5.3 | 18.8 | 2.0 | 32 | 20.2 | 3.8 | 55 | 44 | 40 | 70 | 70 | 55 | 150 | 163 | 176 | 154 | 161 |

* Placed in Storage August 20, 1965

Thus, 3 weeks at 37.8°C would correspond to 21 weeks at 25°C. At the monolayer moisture level, the rate was 4.4 times faster and at 1.2% moisture the rate was very little different, if any, than the reference. The loss of amino nitrogen paralleled the rise in titratable acidity. Under nitrogen, the loss of amino groups was not quite as large. The titratable acidity data supports these observations.

The sweet potatoes stored below the monolayer moisture level in air but not under nitrogen had an elevated iodine titration value. The color of the food was badly faded so carbonyl compounds derived from the oxidation of the lipids must be responsible. The moisture content increased from 1.2 to 1.8%. These carbonyls then reacted with polar groups as amino which accounts for their decrease.

Above the monolayer moisture level under nitrogen, the food had an enhanced iodine titration value which was due presumably to the hydrolysis of starch to dextrins. In air these reducing compounds were not detected because they were immediately oxidized by the oxygen in the package.

At the monolayer moisture level in air and under nitrogen there was a loss of amino nitrogen and an increase in the titratable acidity. The loss was slightly greater when packaged in air. In air the moisture increased 0.4% while under nitrogen 0.9%. The oxidative hydrolytic products of the lipids reacted with the amino nitrogen of free amino acids and proteins.

Polymerization reactions were also going on between lipid decomposition products with the formation of water. Less water was formed in air because a portion of these carbonyl compounds were oxidized and so could no longer enter into polymerization reactions.

For sweet potatoes stored 11 weeks at 37.8°C, the reference and samples stored in air and nitrogen below the monolayer moisture level behaved differently than the other samples upon extraction with water. The portion of the food insoluble in water floated to the surface instead of sinking to the bottom. The lipids are believed to be responsible. Upon freeze dehydration as well as on storage at low moisture levels, the lipids dissolve in one another forming a wax-like coating over the proteins, starches, hemicelluloses, etc. Both these physical changes result in the sweet potatoes being poorly hydrated, causing them to float. On storage at and above the monolayer moisture level, these changes disappear so the food rehydrates properly.

### 3.2.2 Freeze-Dried Beef

#### 3.2.2.1 Freeze-Dried Beef Stored at 25°C

Data on the chemical evaluation of freeze-dried beef stored at 25°C is given in Table 47. In ground beef stored 3 weeks at 25°C, the sample stored in air with a moisture content of 7.9% showed quite extensive chemical changes and has probably deteriorated to the point of being inedible. The color of the meat has changed from a red to a tan-green.

TABLE 47

CHEMICAL EVALUATION OF FREEZE-DRIED GROUND BEEF AT 25°C*

| Initial Relative Humidity | Initial % Water | % Water | Color | Odor | Extraction with Ether: Wt. of Defatted Residue | Extraction with Ether: Gms. Fat Extracted | Comp. per 100 gms of Defatted Beef: Total | Lipase Digestion of Defatted Beef: pH Slurry | Lipase Digestion of Defatted Beef: N NaOH to pH 8.5 | Lipase Digestion of Defatted Beef: % FFF as Oleic | Lipase Digestion of Defatted Beef: Total N NaOH to Formalin | Lipase Digestion of Defatted Beef: Gms Amino Nit. | Lipase Digestion of Defatted Beef: Odor of Digest | Fractionation of Lipids with Acetone, Phosphidipid Precipitation: Digestion with Lipase: Gms Insol. in Acetone | Digestion with Lipase: FFF as Oleic/ 100 gms Fat. | Digestion with Lipase: Mg Amino Nit/ 100 gms Fat. | Digestion with Lipase: Gms Sol. in Acetone | Digestion with Lipase: FFF as Oleic/ 100 gms Fat. | Digestion with Lipase: Mgs Amino Nit/ 100 gms Fat. | Digestion with Lipase: FFA as Oleic 100 gms Fat. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **3 Weeks Analyzed October 18, 1965** | | | | | | | | | | | | | | | | | | | | | |
| Reference | 1.2 | | Red | | 60.8 | 42.1 | 102.9 | 5.8 | 39.7 | 3.12 | 137.4 | 2.65 | Slighty Putrid | 16.6 | 7.6 | | 25.5 | 2.0 | | |
| Air | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.4 | 1.6 | Red | | 59.5 | 41.8 | 101.3 | 5.8 | 39.9 | 2.70 | 132.7 | 2.56 | Sweet | 12.0 | 15.9 | | 29.9 | 1.1 | | |
| 28.0%R.H. | 3.5 | 3.8 | Red Tan-tint | | 62.3 | 41.4 | 103.7 | 5.8 | 39.2 | 2.72 | 132.4 | 2.36 | Sweet | 20.0 | 6.8 | | 21.5 | 1.4 | | |
| 61% R.H. | 7.9 | 8.0 | Tan green | | 59.5 | 39.5 | 99.0 | 5.8 | 42.4 | 4.21 | 193.9 | 2.83 | Putrid | 10.7 | 28.1 | | 28.9 | 2.6 | | |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.5 | 1.5 | Red | | 60.8 | 43.6 | 104.4 | 5.8 | 40.0 | 2.43 | 122.2 | 2.61 | Sweet | 21.1 | 5.7 | | 22.5 | .6 | | |
| 28.0%R.H. | 3.4 | 3.7 | Red | | 60.0 | 42.8 | 102.8 | 5.8 | 41.8 | 2.48 | 131.6 | 2.49 | Sweet | 27.8 | 5.7 | | 14.5 | 3.4 | | |
| 59.%R.H. | 7.5 | 7.8 | Red Tan-tint | | 60.8 | 42.4 | 103.2 | 5.8 | 41.9 | 2.72 | 135.5 | 2.42 | Sweet | 3.8 | 13.3 | | 38.6 | 17.8 | | |
| **7 Weeks Analyzed December 10, 1965** | | | | | | | | | | | | | | | | | | | | | |
| Reference | 1.2 | | Red | | 59.0 | 42.6 | 101.6 | 5.8 | 38.5 | 2.85 | 143.9 | 2.82 | Sl.off | 6.1 | 32.8 | 182 | 36.5 | 28.2 | 71 | .7 |
| Air | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.4 | 1.5 | Red | | 58.2 | 40.6 | 98.8 | 5.8 | 38.0 | 2.98 | 148.0 | 2.77 | Sweet | 5.0 | 35.7 | 84 | 35.6 | 33.6 | 136 | .7 |
| 28.0%R.H. | 3.5 | 4.2 | Red-Tan | | 59.8 | 39.4 | 99.2 | 5.8 | 40.4 | 3.16 | 156.9 | 3.00 | Sl.off | 11.6 | 40.2 | 182 | 27.8 | 33.2 | 123 | 2.4 |
| 61.0%R.H | 7.9 | 7.2 | Gray Brn | | 60.6 | 39.6 | 100.2 | 5.7 | 45.2 | 3.08 | 158.6 | 3.32 | Sl.off | 6.6 | 38.8 | 84 | 33.0 | 33.9 | 242 | 8.4 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.5 | 2.1 | Red | | 58.8 | 41.4 | 100.2 | 5.8 | 38.8 | 2.88 | 143.5 | 2.77 | Sweet | 8.9 | 39.5 | 88 | 32.5 | 34.8 | 170 | 1.0 |
| 28.0%R.H. | 3.4 | 3.8 | Red Tan tint | | 59.5 | 38.7 | 98.2 | 5.8 | 40.9 | 4.61 | 208.6 | 2.80 | Sweet | 8.8 | 38.5 | 115 | 29.9 | 36.3 | 242 | 2.8 |
| 59.0%R.H. | 7.5 | 8.0 | Grey Brn | | 60.2 | 42.3 | 102.5 | 5.8 | 46.0 | 6.24 | 271.4 | 3.04 | Putrid | 8.3 | 37.4 | 95 | 34.0 | 37.1 | 240 | 8.2 |
| **11 Weeks Analyzed October 27, 1965** | | | | | | | | | | | | | | | | | | | | | |
| Reference | 1.2 | 1.2 | Red | None | 58.1 | 43.2 | 101.3 | 5.8 | 39.8 | 4.01 | 141.8 | 3.24 | Sweet | 9.5 | 33.6 | 39 | 33.7 | 11.8 | 45 | | |
| Air - S | | | | | | | | | | | | | | | | | | | | | |
| 8.2%R.H. | 1.4 | 1.6 | Red | Metallic | 57.5 | 43.6 | 101.1 | 5.8 | 40.8 | 3.36 | 118.8 | 3.17 | Sweet | 25.1 | 33.6 | 20 | 18.5 | 10.4 | -32 | | |
| 28.0%R.H. | 3.5 | 4.0 | Red | Metallic | 58.0 | 43.1 | 101.1 | 5.8 | 42.2 | 3.64 | 128.7 | 3.07 | Sweet | 8.8 | 31.5 | 3 | 34.3 | 15.4 | 31 | | |
| 61.0%R.H. | 7.0 | 6.5 | Tan-Sl Green | Sl Cooked | 60.5 | 41.6 | 102.1 | 5.7 | 48.4 | 3.30 | 116.7 | 2.63 | Sweet | 6.7 | 31.9 | -39 | 34.9 | 24.0 | 56 | | |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.5 | 2.8 | Red | Sl Metallic | 57.0 | 42.3 | 99.3 | 5.8 | 39.1 | 3.70 | 130.8 | 3.25 | Sweet | 7.8 | 29.6 | -57 | 34.5 | 8.6 | 10 | | |
| 28.0%R.H. | 3.4 | 3.4 | Sl Off Red | Sl cooked metallic | 56.3 | 42.3 | 99.5 | 5.8 | 42.4 | 3.67 | 129.8 | 3.13 | Sweet | 8.6 | 35.7 | -26 | 34.6 | 10.2 | 3 | | |
| 59.0%R.H. | 7.5 | 8.1 | Red Tan | Fresh meat cooked | 60.5 | 41.3 | 101.8 | 5.8 | 49.6 | 3.60 | 127.1 | 2.72 | Sweet | 7.3 | 33.0 | 3 | 34.0 | 21.4 | 28 | | |

* Placed in storage August 4, 1965

The defatted ground beef from the meat stored in air at a moisture content of 7.9% differed markedly from the other samples on digestion with pancreatic lipase. The amount of free fatty acids liberated, presumably from the lipoproteins, was greater, the amount of alkali required to adjust the pH to 8.5 prior to the addition of formalin was enhanced and the formal titration showed the liberation of a greater amount of amino nitrogen.

The defatted beef from the other storage conditions was digested less readily by the lipase than the reference. The percent free fatty acids liberated as oleic acid was the lowest for the meat stored under nitrogen with a moisture content of 1.5%.

When the defatted beef was digested with lipase, the storage samples showed a progressively small amino nitrogen content with increase in moisture at which they were stored. Those packaged under nitrogen lost less than those in air. The food stored in air at a moisture level of 7.9% had an elevated amino nitrogen but that was due to the greater activation of the lipase by bile pigments which were formed during storage. The components combining with the amino groups may be compounds formed through oxidation and/or hydrolysis of unsaturated lipids or lipoproteins as the heme. Polar groups as amino play an important role in the water holding properties of a food so their decline accounts for the decreased uptake of water when stored at progressively higher moisture levels.

The data on the lipase digestion of the phospholipids and the lipids soluble in acetone are very erratic. Apparently the lipids were not digested by the lipase because on freeze dehydration there was a change in their physical properties so that they were no longer readily hydrated. This difficulty was not encountered after 7 weeks storage at 25°C. The cause shall be discussed in detail at a later storage interval.

In freeze-dried beef stored 7 weeks at 25°C, the defatted residue from the beef stored under nitrogen with a moisture content of 7.5% was digested more than the other samples by the lipase. Also it was the only one that developed a putrid odor. The amount of free fatty acids liberated expressed as oleic was double that of the reference. Bile pigments formed from the decomposition of the heme of the myoglobin apparently caused the lipase to function more efficiently. The defatted residue from the beef stored under air with a moisture content of 7.9% did not display this phenomenon as it did after storage for 3 weeks at 25°C. The bile pigments must have continued to decompose until they no longer could activate the lipase.

A free fatty acid was run on the lipids soluble in acetone and showed a progressive rise in concentration with increase in moisture content of storage. In Phase 1 of this contract a free fatty acid was run on Wilson's

Dehydrated Fully Cooked Ground Beef. At none of the storage intervals was there a rise in the free fatty acids of beef with a moisture content comparable to this now under study. Lipases normally present in the beef must have hydrolyzed the fat during storage.

The phospholipids separated from the lipids derived from the storage samples by means of acetone were digested more completely than the reference. The digest of the reference was thick, the digests from the beef stored under air at a moisture content of 1.4% and from the food under nitrogen at a moisture content of 1.5% were not quite as viscous while in the remainder of the storage samples the digests were very thin. The rate of digestion of the phospholipids from the samples, however, was very different. The meat with a moisture content above the monolayer moisture level in air as well as under nitrogen were rapidly attacked by the enzyme solution so that after 1 hour at 37.8° C the fat was completely disintegrated and had formed a homogeneous solution. After 2 1/2 hours at 37.8° C the phospholipids from the reference, the sample stored under air with a moisture content of 1.4% and under nitrogen with a moisture content of 1.5% were little attacked by the lipase. The light yellow fat particles remained discrete with their surface white where the enzyme had commenced to attack the fat. The enzyme is not able to penetrate the fat particles so as to soften and disintegrate them. A physical barrier seems to exist between the fat and enzyme.

The phospholipids of the beef separated by the acetone fractionation procedure were white in color but tend to turn brown if left exposed to the air. They melted at about 70° C. Free fatty acids and bile pigments should not be present since the phospholipids were unique in being insoluble in acetone. Bile pigments should not be present in the first place because they were very slightly soluble in ether so would not be extracted when the fat was extracted with ether.

When ground beef samples stored for 11 weeks at 25° C were extracted with ether, the 2 samples stored above the monolayer moisture level differed from the reference and other storage samples in having a large amount of fines. After agitating with ether and allowing the residue to settle, a layer of fines about 3 mm thick settled down on the main mass of beef. For both samples, the residue insoluble in ether weighed more than the reference and other storage samples and the weight of the fat extracted was correspondingly less. The cause is believed to be due to the hydrolysis of a lipoprotein fraction which prior to hydrolysis was soluble in ether and after hydrolysis the protein moiety was no longer soluble and appears as a fine light precipitate above the main mass of the meat. These samples have changed in moisture indicating the occurrence of various reactions on, some of which consume water while in others water is formed.

In the beef stored above the monolayer value, the sample packaged in air lost 1.4% water presumably due to hydrolysis of fats. The same sample stored under nitrogen increased in moisture 0.6%. Here water consumed by hydrolysis was slightly less than that formed through polymerization reactions of the lipids. The defatted residues of these 2 samples weighed 2.4 grams more than the reference which is apparently derived from lipoproteins which prior to hydrolysis were soluble in ether.

The beef stored at the monolayer moisture level in air and under nitrogen seemed quite stable as to moisture change. The defatted residue of the meat stored under nitrogen weighed 1.8 grams less than the defatted residue of the reference. Although the data is not complete enough to prove the following supposition, it is believed that the phospholipid moiety of the lipoproteins on storage reacts with the residual oxygen in the food to form peroxides which decompose to carbonyls when they enter into polymerization reactions with all sorts of compounds being formed. Under this particular storage condition one of the reactions products is not water but other compounds. Head space gas analysis would probably reveal their identity. The sample stored in air resembles the reference in this regard as well as in the other analyses.

The meat stored above the monolayer moisture level differed from the others in that the fines on the filter paper after the paper had dried in the hood overnight were devoid of any fatty residue on the paper. During

the 5 extractions with ether when the supernatant was decanted on to the filter paper a small amount of solid material was carried over, particularly small meat particles, which were somewhat slow in settling. This residue flaked off very readily. In the other storage samples this residue had to be scraped from the filter paper and was of a slightly greasy consistency. The beef contains a material which is borderline in regard to being soluble in ether and disappeared on storage presumably by hydrolysis.

When the defatted residues were digested with lipase the storage samples appeared to digest fairly well although less free fatty acids were released as compared to the reference. The two samples with a moisture content above the monolayer moisture level had an elevated titratable acidity.

The phospholipids and lipids soluble in acetone were digested by the lipase to about the same extent as the reference except for the sample stored under nitrogen with a moisture content of 1.5%. The cause is believed to be due to a chemical modification of the fat through peroxide formation and decomposition to carbonyls which polymerize producing lipids which the lipase can no longer hydrolyze or more than likely a portion of the fat just simply disappears as water and other volatile compounds.

3.2.2.2 Freeze-Dried Beef Stored at 37.8°C

The data for freeze-dried beef stored 3, 7, and 11 weeks at 37.8°C is shown in Table 48. In the samples of ground beef stored 3 weeks at 37.8°C, it was found that the phospholipids from the reference sample were attacked by lipase more rapidly than the beef stored in air below the monolayer moisture level and in air at the monolayer moisture level. The remaining storage samples of beef were attacked slightly less rapidly than the reference. When the samples were held overnight in the incubator cell, the digests were so viscious that they had to be diluted with water before the free fatty acids as oleic acid could be determined.

Ground beef stored for 7 weeks at 37.8°C under nitrogen as well as air at a moisture level above the monolayer value possessed a fine white flocculent material which was soluble in ether and settled above the beef residue. Also, no greasy material was left on the filter paper after the ether extract was filtered as was in the case of the reference and other storage samples. The residue insoluble in ether flaked off the filter paper while the other samples had to be scraped off, some of which were quite greasy to the touch. The food constituent involved may be a lipoprotein which is on the borderline with regard to its solubility in ether. During storage this lipoprotein was probably hydrolyzed with a portion of the hydrolyzate. Presumably the protein moiety was rendered insoluble in

TABLE 48

CHEMICAL EVALUATION OF FREEZE-DRIED GROUND BEEF AT 37.8°C*

| Initial Relative Humidity | Initial % Water | % Water | Color | Odor | Extraction with Ether: Wt. of Defatted Residue | Extraction with Ether: Gms Fat Eltd. | Lipase Digestion of Defatted Beef Comp. per 100 gms of Defatted Beef: Total | pH Slurry | N NaOH to pH 8.5 | %FFA as Oleic | Total N NaOH prior to Formolin | Gms Amino Nit. | Odor of Direct | Precipitation of Phospholipids with Acetone Digestion with Lipase: Gms Insol in Acetone | FFA aq. Oleic/100 gm Fat | Mgs Amino Nit./100 Grn Fat | Gms Sol. in Acetone | FFA as Oleic /100 gms Fat | Mgs Amino Nit/100 gms Fat | Lipids Sol in Acetone: FFA Oleic/100 gm Fat | Peroxide No |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **3 Weeks Analyzed January 3, 1966** | | | | | | | | | | | | | | | | | | | | | |
| Reference | 1.2 | | Red | | 57.5 | 48.0 | 105.5 | 5.8 | 39.1 | 3.36 | 157.9 | 3.17 | Sweet | 4.46 | 35.5 | -1.4 | 43.5 | 7.5 | 28.0 | 0.6 | |
| Air | | | Red Tan tint | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.4 | 1.7 | | | 57.3 | 42.7 | 100.0 | 5.8 | 41.2 | 3.40 | 161.4 | 3.16 | Sweet | 3.80 | 29.0 | -23.8 | 38.9 | 5.8 | 7.0 | 1.4 | |
| 28.0%R.H. | 3.5 | 3.5 | Tan Red Brn tint | | 56.5 | 43.2 | 99.7 | 5.8 | 43.3 | 3.75 | 175.9 | 3.33 | Putrid | 3.78 | 28.6 | -74.2 | 39.4 | 12.0 | 86.8 | 2.9 | |
| 61.0%R.H. | 7.9 | 7.4 | Light Brn | | 59.3 | 42.3 | 101.6 | 5.7 | 47.5 | 3.00 | 146.5 | 2.67 | Sl Off | 3.93 | 23.4 | -14.0 | 38.4 | 13.2 | 107.8 | 8.3 | |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.5 | 1.4 | Red | | 57.3 | 39.3 | 96.8 | 5.8 | 40.2 | 3.69 | 170.8 | 3.54 | Putrid | 3.40 | 27.7 | 7 | 36.1 | 8.8 | 29.4 | 1.0 | |
| 28.0%R.H. | 3.4 | 3.4 | Red | | 57.5 | 43.8 | 101.3 | 5.8 | 41.8 | 3.78 | 176.0 | 3.48 | Putrid | 4.44 | 34.2 | 28 | 39.4 | 8.4 | 61.6 | 3.0 | |
| 59.0%R.H. | 7.5 | 7.4 | Red-tint | | 58.9 | 42.3 | 101.2 | 5.7 | 49.4 | 3.88 | 186.6 | 3.15 | Putrid | 3.85 | 28.7 | -16.8 | 38.5 | 13.2 | 191.8 | 8.3 | |
| **7 Weeks Analyzed January 24, 1966** | | | | | | | | | | | | | | | | | | | | | |
| Reference | 1.2 | | Red | | 57.9 | 42.6 | 100.5 | 5.8 | 39.8 | 2.92 | 148.7 | 3.06 | Sweet | 4.08 | 12.3 | 98.8 | 38.52 | 29.4 | 242 | 0.6 | 22 |
| Air | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.4 | 1.4 | Red Tan | | 56.6 | 43.0 | 99.6 | 5.8 | 42.5 | 3.97 | 188.4 | 3.71 | Putrid | 4.30 | 11.2 | 23.0 | 38.7 | 27.7 | 287 | 1.1 | 7 |
| 28.0%R.H. | 3.5 | 3.4 | Grey Brn | | 56.8 | 43.3 | 100.1 | 5.8 | 44.5 | 3.97 | 190.5 | 3.15 | Sl Off | 4.08 | 14.6 | 95.2 | 39.22 | 27.5 | 288 | 4.5 | 0 |
| 61.0%R.H. | 7.9 | 7.6 | Brn | | 61.3 | 40.5 | 101.8 | 5.7 | 53.2 | 3.05 | 166.6 | 2.51 | Sl Off | 3.88 | 25.7 | 300.1 | 37.62 | 29.7 | 287 | 13.1 | 44 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.5 | 1.9 | Red | | 56.3 | 43.0 | 99.3 | 5.8 | 41.8 | 3.80 | 181.7 | 3.71 | Putrid | 5.89 | 9.4 | 14.2 | 37.11 | 24.00 | 441 | 1.2 | 7 |
| 28.0%R.H. | 3.4 | 3.7 | Red-Tan tint | | 58.0 | 43.4 | 101.4 | 5.8 | 43.4 | 3.89 | 186.4 | 3.31 | Putrid | 3.86 | 16.9 | 76.9 | 39.54 | 26.0 | 357 | 5.0 | 0 |
| 59.0%R.H. | 7.5 | 7.1 | Red Brn | | 60.6 | 40.5 | 101.1 | 5.7 | 55.7 | 2.88 | 163.3 | 2.09 | Sl.Off | 5.53 | 27.2 | 119.8 | 34.97 | 22.4 | 532 | 13.2 | 15 |
| **11 Weeks Analyzed February 16, 1966** | | | | | | | | | | | | | | | | | | | | | |
| Reference | 1.2 | 1.2 | Red | None | 57.3 | 41.1 | 98.4 | 5.9 | 38.5 | 2.25 | 118.3 | 2.81 | Sl.Off | 7.3 | 17.3 | 164.7 | 38.8 | 9.7 | 108 | 0.6 | 29 |
| Air | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.4 | 1.5 | Red Tan tint | Metallic | 56.9 | 43.0 | 99.9 | 5.8 | 44.4 | 2.19 | 121.8 | 2.55 | Sweet | 6.73 | 22.4 | 188.7 | 36.27 | 10.2 | 133 | 1.5 | 15 |
| 28.0%R.H. | 3.5 | 3.0 | Tan Gn tint | Cooked | 58.8 | 42.3 | 101.1 | 5.8 | 47.6 | 2.11 | 122.2 | 2.11 | Sweet | 4.26 | 21.5 | 128.5 | 38.04 | 10.2 | 108 | 5.7 | 22 |
| 61.0%R.H. | 7.9 | 7.6 | Golden Bright Brn | Burned | 62.3 | 40.0 | 102.3 | 5.7 | 58.8 | 2.13 | 134.1 | 1.82 | Sweet | 3.44 | 25.2 | 163.8 | 36.56 | 12.0 | 101 | 14.9 | 58 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | |
| 8.5%R.H. | 1.5 | 2.5 | Red | None | 56.7 | 39.9 | 96.6 | 5.9 | 42.0 | 2.10 | 116.4 | 2.59 | Sl.Off | 6.90 | 17.2 | 79.4 | 33.0 | 6.6 | 169 | 1.9 | 15 |
| 28.0%R.H. | 3.4 | 3.3 | Tan Gn-tint | Burnt | 58.3 | 40.8 | 99.1 | 5.8 | 48.4 | 2.16 | 125.0 | 2.27 | Sweet | 3.81 | 23.9 | 211.7 | 36.99 | 10.6 | 129 | 6.5 | 123 |
| 59.0%R.H. | 7.5 | 7.7 | Red-tan | Burnt | 61.8 | 39.1 | 100.9 | 5.8 | 57.4 | 2.22 | 135.9 | 1.89 | Sweet | 3.96 | 22.7 | 149.9 | 35.14 | 14.0 | 167 | 17.5 | 102 |

* Placed in storage August 4, 1965

ether and appeared as a fine flocculent material which settled above the main mass of material insoluble in ether. The amount of material formed at this storage interval was about 2.5 grams per 100 grams of anhydrous beef, after 3 weeks at 37.8°C, 1.4 grams; after 11 weeks at 37.8°C, 4.5 grams and after 11 weeks at 25°C, 2.4 grams. The rate of this hydrolytic reaction at 37.8°C was about double that at 25°C.

The weight of the defatted residue from the beef stored in air below the monolayer moisture level was 1.3 grams less than the reference and the same sample stored under nitrogen 1.6 grams less than the reference. This loss in weight is believed to be due to the disintegration of the phospholipid moiety of the lipoproteins to water and other volatile compounds. Head-space gas analysis would probably reveal their identity.

When the titratable acidity was determined in the beef stored 3 weeks at 37.8°C, it was observed that the aqueous suspension of the reference was thick while the aqueous suspensions from the storage samples were thin. Thus, the constituent(s) responsible for the body or viscosity of the beef had disappeared.

After the beef had been digested with lipase and the formol titration completed, the storage samples were allowed to settle and the residue was collected. The residues contained less collagen and less grey material which appeared as thin flat ribbons. The volume of the collagen layer and

the layer of grey ribbons was about 1/3 that of the reference sample. For samples stored at and below the monolayer moisture level, the amount of collogen layer and the layer of grey ribbons increased slightly. A major portion of the grey filaments and collagen had disintegrated on storage to mole particles and some had become soluble in water. Also, the small amount of collogen remaining had changed from a white to a tan color at the lower moisture levels and to a brown color at the highest moisture level.

In adding the enzyme solution to the phospholipids, the fat from the ground beef stored under nitrogen at a moisture content of 7.5% had almost completely gone into solution in 5 minutes. The reference and 2 samples stored below the monolayer moisture level were attacked very slightly, if at all, by the enzyme. Every hour during the day the samples were taken from the incubator and examined to observe the progress of the digestion. The lipids from the reference and 2 samples stored below the monolayer moisture level were digested slowly.

The cause was probably due to a physical change induced in the lipids during freeze dehydration. The lipids from the sample stored in air at a moisture content of 7.9% were digested slower than the other samples. The enzyme apparently could not soften the individual fat particles. The reason for this probably is due to the occurrence of a chemical change such as oxidative rancidity instead of a physical modification of the lipids. The lipids soluble in acetone from this sample had a peroxide value of 44.

Since the phospholipids are even more unsaturated, their peroxide value would probably be much higher.

When the phospholipid-enzyme solution was held in the incubator overnight, the samples stored in air and nitrogen below the monolayer moisture level contained slightly less free fatty acids as oleic acid. This appeared to be due to the modification of the phospholipids through oxidative-hydrolytic-polymerization reactions. Also, the weight of the defatted residue stored in air was 1.3 grams less than the reference and for the sample stored under nitrogen 1.6 grams less than the reference. This loss was believed due to the disappearance of the phospholipid moiety of lipoproteins by degradation to volatile compounds. Thus the two samples in question were digested less completely because they were changed chemically through oxidation and a portion simply disappeared through polymerization reactions of the resulting carbonyl compounds to water and other volatile compounds. The samples at the monolayer moisture level and above the phospholipids were digested to a greater extent by the lipase than the reference.

In ground beef stored 11 weeks at 37.8°C, it was found that the recovery of phospholipids was less than in the reference sample. This reduction in the recovery of phospholipids is believed to be due to the hydrolysis by enzymes, chemical hydrolysis and chemical decomposition. The volume of phospholipids recovered from the reference sample and the two samples stored below the monolayer moisture level was approximately double that of the other storage samples.

The phospholipids of the reference and stored beef were digested about the same extent in 24 hours. Further, there was no great difference in the rate of digestion. The lipids from the beef sample with a moisture content above the monolayer were rapidly attacked by the lipase. The lipids went into solution in about 15 minutes. The reference and sample stored in air below the monolayer moisture level were barely attacked after 2 hours. The samples stored at the monolayer moisture level and the sample stored under nitrogen at a moisture content of 1.5% were moderately attacked. After digesting overnight at 37.8° C, the solution of the reference was viscous, the digests from the samples with a moisture content below the monolayer were less viscous while the remaining 4 samples were very thin.

When the defatted beef residue was digested with lipase, the amount of amino nitrogen liberated was less than that from the digestion of the reference sample for all the storage conditions. The amount of amino nitrogen declined more the higher the moisture content at which the beef was stored. This loss in amino nitrogen must be directly related to the magnitude of the titratable acidity. Storage of the beef in air or nitrogen did not appear to influence the reactions which caused the disappearance of the amino groups. Fewer amino groups, however, were lost in beef stored under nitrogen.

The lipids of the ground beef soluble in acetone were digested with lipase. All the storage samples were digested to about the same extent as the reference sample except for the meat stored under nitrogen at a moisture content of 1.5%.

The defatted residue from the beef stored under nitrogen below the monolayer moisture level weighed 0.6 grams less than the reference and the lipids extracted weighed 1.2 grams less than the reference. This loss was probably due to the decomposition of phospholipids and lipoproteins into volatile compounds. The residual oxygen in the beef reacted with highly unsaturated fatty acids of the lipoproteins producing peroxides which decomposed into aldehydes and ketones. Aldehydes and ketones contain the reactive carbonyl group, C = 0, which is characterized by extreme unsaturation or ability to add a great variety of agents. The activated form will add H or a metal atom from a great variety of reagents. The rest of the addend may add to the electronically deficient carbon or elsewhere in the molecule if a shift first takes place (1:4 addition). Water is often lost.[14]

In the digestion of the defatted residue with lipase, the amount of amino nitrogen liberated was less than the reference for all the storage samples and declined more the higher the moisture content at which the beef was stored. The loss in amino nitrogen must be directly related to the magnitude of the titratable acidity. Storage of the beef in air or nitrogen

does not seem to influence the reactions which caused the disappearance of the amino groups; however, slightly fewer amino groups were lost under nitrogen.

In the digestion of the lipids soluble in acetone with lipase, all the storage samples were digested to about the same extent as the reference except for the meat stored under nitrogen at a moisture content of 1.5%. The moisture content of the beef increased from 1.5% to 2.5% during storage. As discussed previously, the moisture was thought to come from polymerization reactions of the unsaturated lipids. Residual oxygen in the food adds to the double bonds of unsaturated fatty acids which break down to carbonyl compounds. The latter polymerize to form water and other volatile compounds. The lipids modified in this manner apparently are attacked less readily by the lipase. The peroxide value of the lipids soluble in acetone was 15 in this sample while samples stored at the 2 higher moisture levels had peroxide values in excess of 100. In the presence of a little water the peroxides must be more stable and do not decompose.

### 3.2.3 Changes in the Reference Ground Beef while Stored in the Frozen State

#### 3.2.3.1 Digestion of the Defatted Protein with Lipase

The reference was packaged under nitrogen in metallized aluminum pouches and held at -20°C.

The titratable acidity of the defatted protein and the amino nitrogen liberated by the lipase did not change. However, the amount of free fatty acids as oleic acid liberated by the lipase seemed to decline with the amount of time the beef was held in the frozen state. The data for the beef stored 11 weeks at 25° C was high because after the addition of the enzyme solution the pH was adjusted to pH 8.5 twice instead of once during the day. Apparently, the phospholipid moiety of the lipoproteins is gradually undergoing oxidative rancidity, a change which causes it to be less readily digested by lipase. The peroxide value of the lipids soluble in acetone was 29 at the time of the last analysis of the beef. Probably the peroxides were decomposing. Headspace gas analyses might support such a hypothesis.

3.2.3.2 Extraction of the Beef with Ether

The combined weight of the defatted residue and fat extracted on October 18, 1966 was 102.9 grams. There was a gradual decline in this value so that on February 16, 1966, the combined weight was 98.4 grams. Again the possible explanation for this could be the decomposition of the phospholipids to volatile compounds.

3.2.4 Freeze-Dried Spinach

3.2.4.1 Freeze-Dried Spinach Stored at 25° C

Data on the chemical evaluation of freeze-dried spinach stored at 25° C is given in Table 49.

TABLE 49

CHEMICAL EVALUATION OF FREEZE-DRIED SPINACH AT 25°C*

Composition of Food per 100 grams Anhydrous Spinach
Extraction of Spinach with Water

Titration of the Aqueous Extract with 0.01 N Iodine Solution
Results Expressed as Mgs Ascorbic Acid/100 grams Food

| Initial Relative Humidity | Initial % Water | % Water | Color | Formol Titration of Residue: pH Slurry | Formol Titration of Residue: N NaOH to pH 8.5 | Formol Titration of Residue: Versene N NaOH to pH 8.5 | Formol Titration of Residue: Mgs Amine Nit. | Formol Titration of Aq. Extract: pH Slurry | Formol Titration of Aq. Extract: N NaOH to pH 8.5 | Formol Titration of Aq. Extract: Versene N NaOH to pH 8.5 | Formol Titration of Aq. Extract: Mgs. Amine Nit. | Total Titratable Acidity | Total Versene N NaOH to pH 8.5 | Total Mgs. Amine Nit. | Color | Odor | Reducing Substances reacted With Iodine 20 sec.: No Additive | Reducing Substances reacted With Iodine 20 sec.: Formalin | Reducing Substances reacted With Iodine 20 sec.: Versene | Reducing Substances reacted With Iodine 20 sec.: Formalin and Versene | Reducing Substances reacted With Iodine 20 sec.: Average | Reducing Sustances reacted With Iodine over 10 min.Period: No Additive | Reducing Sustances reacted With Iodine over 10 min.Period: Formalin | Reducing Sustances reacted With Iodine over 10 min.Period: Versene | Reducing Sustances reacted With Iodine over 10 min.Period: Formalin and Versene | Reducing Sustances reacted With Iodine over 10 min.Period: Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | 4 Weeks Analyzed January 17, 1966 | | | | | | | | | | | | | | |
| Ref. 6% R.H. | 1.4 | 1.4 | Gn | 6.45 | 5.7 | 47.1 | 175 | 6.37 | 12.1 | 19.5 | 145 | 17.8 | 66.6 | 320 | Gn-tan tint | Grass | 62 | 53 | 53 | 70 | 60 | 172 | 172 | 163 | 198 | 176 |
| Air | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.5 | Gn | 6.38 | 5.8 | 46.2 | 195 | 6.42 | 11.6 | 20.3 | 139 | 17.4 | 66.3 | 334 | Gn | Grass | 66 | 75 | 57 | 66 | 66 | 194 | 194 | 163 | 194 | 186 |
| 21.5%R.H. | 4.8 | 4.9 | Gn | 6.44 | 5.8 | 49.2 | 175 | 6.21 | 12.8 | 18.2 | 131 | 18.6 | 67.4 | 306 | Gn | Grass | 53 | 70 | 53 | 70 | 62 | 158 | 198 | 163 | 194 | 178 |
| 54.5%R.H. | 11.1 | 11.5 | Gn | 6.32 | 6.4 | 47.9 | 191 | 6.23 | 12.8 | 17.2 | 131 | 19.2 | 65.1 | 322 | Gn-Tan tint | Grass | 48 | 53 | 40 | 53 | 49 | 158 | 172 | 141 | 185 | 164 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.6 | Gn | 6.47 | 6.2 | 48.4 | 184 | 6.10 | 14.3 | 18.2 | 135 | 20.5 | 66.6 | 319 | Gn-tan tint | Grass | 48 | 44 | 62 | 79 | 58 | 141 | 167 | 163 | 211 | 171 |
| 21.5%R.H. | 4.8 | 4.9 | Gn | 6.40 | 5.3 | 48.4 | 178 | 6.41 | 11.3 | 19.4 | 141 | 16.6 | 67.8 | 319 | Gn-tan tint | Grass | 75 | 97 | 66 | 66 | 76 | 189 | 220 | 176 | 194 | 195 |
| 54.5%R.H. | 11.1 | 10.8 | Gn | 6.38 | 5.9 | 49.5 | 165 | 6.33 | 11.9 | 18.3 | 141 | 17.8 | 67.8 | 306 | Tan-Gn | Hay | 66 | 48 | 66 | 66 | 62 | 180 | 180 | 154 | 172 | 172 |
| | | | | | | | | | | | | 8 Weeks Analyzed February 15, 1966 | | | | | | | | | | | | | | |
| Ref. 6% R.H. | 1.4 | 1.3 | Gn | 6.35 | 6.7 | 46.5 | 165 | 6.48 | 11.2 | 16.8 | 130 | 17.9 | 63.3 | 295 | Gn-faint tan | Faint Grass | 48 | 53 | 57 | 57 | 54 | 150 | 172 | 167 | 172 | 165 |
| Air | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.4 | Gn | 6.39 | 6.2 | 45.5 | 172 | 6.42 | 11.3 | 17.0 | 124 | 17.5 | 62.5 | 296 | Gn | Grass | 53 | 40 | 53 | 57 | 51 | 163 | 154 | 145 | 167 | 157 |
| 21.5%R.H. | 4.8 | 5.1 | Gn | 6.38 | 6.1 | 45.7 | 163 | 6.42 | 11.5 | 16.9 | 156 | 17.6 | 62.6 | 319 | Gn | Grass | 26 | 40 | 44 | 62 | 43 | 128 | 154 | 136 | 180 | 150 |
| 54.5%R.H. | 11.1 | 10.5 | Gn | 6.28 | 7.7 | 46.3 | 171 | 6.25 | 12.9 | 17.1 | 115 | 20.6 | 63.4 | 286 | Gn-slight Tan | Grass | 22 | 31 | 31 | 48 | 38 | 119 | 141 | 114 | 158 | 133 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.4 | Gn | 6.42 | 6.2 | 45.1 | 163 | 6.45 | 11.2 | 16.9 | 125 | 17.4 | 62.0 | 288 | Gn-faint Tan | Faint Grass hay | 40 | 44 | 53 | 53 | 48 | 141 | 163 | 141 | 167 | 153 |
| 21.5%R.H. | 4.8 | 5.4 | Gn | 6.38 | 6.1 | 44.9 | 163 | 6.48 | 11.5 | 17.6 | 130 | 17.6 | 62.5 | 293 | Gn-faint Tan | none | 62 | 53 | 62 | 53 | 58 | 180 | 167 | 167 | 176 | 173 |
| 54.5%R.H. | 11.1 | 10.8 | Gn | 6.30 | 6.6 | 45.8 | 167 | 6.30 | 13.0 | 16.9 | 118 | 19.6 | 62.7 | 285 | Gn-tan | none | 31 | 35 | 40 | 62 | 42 | 136 | 145 | 132 | 198 | 153 |
| | | | | | | | | | | | | 12 Weeks Analyzed March 14, 1966 | | | | | | | | | | | | | | |
| Ref. 6.0%R.H. | 1.4 | 1.9 | Gn | 6.50 | 32.4 | 80.6 | 258 | 6.62 | 10.6 | 20.9 | 152 | 43.0 | 101.5 | 410 | Gn-tan | Faint Hay | 66 | 66 | 53 | 53 | 59 | 194 | 189 | 145 | 158 | 172 |
| Air | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.6 | Gn | 6.69 | 30.0 | 67.5 | 288 | 6.59 | 11.0 | 20.7 | 136 | 50.0 | 88.2 | 424 | Gn | Grass | 62 | 48 | 53 | 53 | 54 | 180 | 176 | 150 | 158 | 166 |
| 21.5%R.H. | 4.8 | 5.3 | Gn | 6.79 | 5.7 | 95.3 | 152 | 6.55 | 11.0 | 21.4 | 136 | 16.7 | 116.7 | 288 | Gn | Grass | 44 | 53 | 53 | 48 | 50 | 154 | 189 | 158 | 158 | 165 |
| 54.5%R.H. | 11.1 | 10.9 | Gn-tan tint | 6.64 | 131.7 | 49.7 | 178 | 6.33 | 12.8 | 20.5 | 126 | 134.5 | 70.2 | 304 | Bn-Gr tint | Hay | 31 | 35 | 48 | 53 | 42 | 150 | 167 | 141 | 154 | 153 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.8 | Gn | 6.59 | 43.4 | 62.3 | 343 | 6.59 | 10.6 | 21.0 | 147 | 54.0 | 83.3 | 490 | Gn-tan | Faint grass hay | 84 | 53 | 62 | 70 | 67 | 207 | 185 | 158 | 176 | 182 |
| 21.5%R.H. | 4.8 | 5.6 | Gn | 6.55 | 32.4 | 81.2 | 231 | 6.60 | 10.6 | 20.8 | 150 | 43.0 | 102.0 | 380 | tan-gn tint | faint hay | 75 | 57 | 62 | 62 | 64 | 194 | 194 | 154 | 176 | 180 |
| 54.5%R.H. | 11.1 | 10.7 | Gn-tan tint | 6.69 | 21.7 | 88.3 | 157 | 6.39 | 12.5 | 20.4 | 129 | 34.2 | 108.7 | 286 | Gn | faint hay | 44 | 31 | 40 | 40 | 39 | 128 | 158 | 128 | 145 | 140 |

* Placed in storage December 21, 1965

Spinach stored 4 weeks at 25°C at the monolayer moisture level in air and under nitrogen behaved very peculiarly when the 250 ml Erlenmeyer flasks containing the spinach were opened to run the moisture determinations. When the spinach was poured from the flasks, spinach particles aggregated around the neck of the flask in large clusters presumably because they possessed a static charge. This made it difficult to remove the spinach from the flasks.

The color of the aqueous extract of the spinach stored at and below the monolayer moisture level in air was green and remained green after storing in the refrigerator overnight. The odor of the spinach resembled freshly cut grass. These samples probably had an oxidative-reduction equilibrium which leaned toward an oxidized condition.

The color of the aqueous extracts of the reference and other storage samples of spinach were green with a tan tint and the tan color intensified when the samples were stored overnight in the refrigerator. The oxidative-reduction equilibrium of these extracts are believed to lean toward a reduced state. When the aqueous extracts of the spinach stood overnight in the refrigerator, the hydrolytic products of the pentosans as pentoses polymerized further. The chlorophyll in the spinach was also reduced. Both reactions are believed to be responsible for the production of a brown color. In the extract from the spinach stored at a moisture content of 11.1%, these reactions had progressed sufficiently for enough to give the extract a hay-like odor.

For spinach stored 8 weeks at 25° C, the iodine titration values are essentially the same as those for spinach stored at 25° C. The sample stored under nitrogen at the monolayer moisture level had an elevated iodine titration value. Also, the dry spinach had a relatively high static charge.

The aqueous extracts from the food stored at a moisture content of 1.4 and 4.8 % were green in color, had a grass-like odor, and remained unchanged after storing overnight in the refrigerator. As discussed previously, this was believed to be due to the oxidation by the air in the package of any pentoses initially present or formed by hydrolysis of pentosans. Therefore, the pentoses could not polymerize or reduce the highly unsaturated chlorophyll molecule. Both of these reactions produce a brown color. The aqueous extracts of the reference and other samples had a tan tint which intensified after storage overnight in the refrigerator. Probably, there was not enough oxygen present to eliminate the pentoses so they polymerized and/or reduced the chlorophyll with the formation of a tan color.

A vacuum was created in the 250 ml Erlenmeyer flasks containing the spinach stored 12 weeks at 25° C in air at the monolayer moisture level. The sample of spinach stored in air above this moisture level appeared to have an even greater vacuum. It appears that the oxygen of the container was consumed through various chemical reactions. This vacuum was not observed in the flasks containing the reference and other storage samples.

Furthermore, the food stored under nitrogen at the monolayer moisture level was the only sample in which the food particles possessed a static charge. However, the static charge was not as large as that for the previous storage periods.

When the spinach was extracted with water, the food stored in air at a moisture content of 1.4% behaved differently than the reference and the other storage samples. The aqueous slurry was thick, having about twice the viscosity of the reference. Also, filtration was very slow due to the presence of a large number of very small particles which clogged the filter paper and which were very difficult to remove. It appears that the spinach particles broke down to a smaller size.

The color of the aqueous extracts from the spinach stored in air at moisture levels of 1.4 and 4.8% was a rich green with an odor of newly mowed grass. When these extracts were stored in the refrigerator overnight their color or odor did not change. The reference and other samples were green but tinged with a tan or brown color, the intensity increasing at the higher moisture levels of storage. Also, a hay-like odor appeared in these samples. The tan color and hay odor intensified after these extracts had stood in the refrigerator overnight.

The reference and spinach sample stored under nitrogen at an initial moisture content of 1.4% increased approximately 0.5% in moisture. These two samples were packaged under nitrogen and differed in that the

former was stored at -20°C and the latter at room temperature. Apparently, the water was formed through polymerization reactions of the lipids, particularly the chlorophyll. The reaction rate and induction period seemed to be the same at the two temperatures which began somewhere between eight and twelve weeks of storage. More water was formed under nitrogen than in the same sample stored in air because possibly oxygen kept breaking the polymerization reaction by oxidation of a participant as a carbonyl compound.

The spinach stored under nitrogen at an initial moisture content of 1.4 and 4.8% had elevated iodine titration values and an increase in moisture content of 0.4 and 0.8%, respectively. More brown was present in the color of the aqueous extract. It would appear that the water is needed to initiate the polymerization reaction which results in the formation of water and a brown color. The more water initially present, the faster the reaction occurs. Also, exclusion of oxygen favors the reaction since less water and color was formed in these same samples stored in air. The reactions involved in these changes must be very complex. A simplified version is that water adds across a double bond of the chlorophyll molecule to produce carbonyl compounds which polymerize with the formation of water and a brown pigment.

3.2.4.2 Freeze-Dried Spinach Stored at 37.8°C

Data on the chemical evaluation of freeze-dried spinach stored at 37.8°C is given in Table 50.

For spinach stored 3 weeks at 37.8°C, the samples stored under air and nitrogen at a moisture content of 11.1% deteriorated very rapidly. Due to the complexity of the reactions taking place, no differences were detected in these samples with regard to the formation of a hay-like odor and brown color of the aqueous extract. When the samples were extracted with water, filtration was very rapid as compared with the reference.

The spinach stored in air with an inititial moisture content of 1.4% filtered more slowly than the reference sample. The number of small spinach particles or fines had increased during storage and these clogged the pores of the filter paper. Apparently, the spinach was fractured through the oxidative processes.

An elevated iodine titration value was observed for spinach stored at this temperature. Probably, reactive carbonyls formed from the chlorophyll or reducing sugars formed by the hydrolysis of the pentosans immediately took part in the various chemical reactions. The spinach stored at an initial moisture content of 11.1% had less reducing compounds present which were probably due to the destruction of ascorbic acid.

TABLE 50

CHEMICAL EVALUATION OF FREEZE-DRIED SPINACH AT 37.8°C*

| | Composition of Food per 100 grams of Anhydrous Spinach Extraction of Spinach with Water | | | | | | | | | | | | | | | Titration of the Aqueous Extract with 0.01 N Iodine Solution Results Expressed as Mgs Ascorbic Acid/100 grams Food. | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Formol Titration of Residue | | | | Formol Titration of Aq. Extract | | | | | | | | Reducing Sustances reacted With Iodine 20 sec | | | | | Reducing Sustances reacted With Iodine over 10 min. Period | | | | |
| Initial Relative Humidity | Initial % Water | % Water | Color | pH Slurry | N NaOH to pH 8.5 | Versene N NaOH to pH 8.5 | Mgs Amine Nit. | pH Slurry | N NaOH to pH 8.5 | Versene N NaOH to pH 8.5 | Mgs Amine Nit. | Total Titratable Acidity | Total Versene N NaOH to pH 8.5 | Total Mgs Amine Nit. | Color | Odor | No Additives | Formolin | Versene | Formolin & Versene | Average | No Average | Formolin | Versene | Formolin & Versene | Average |
| 3 Weeks Analyzed January 10, 1966 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Ref. 6.0%R.H. | 1.4 | 1.4 | Gn | 6.2 | 5.8 | 45.2 | 175 | 6.4 | 12.2 | 21.1 | 137 | 18.0 | 66.3 | 312 | | | 35 | 66 | 57 | 70 | 57 | 123 | 167 | 141 | 176 | 152 |
| Air | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.6 | Gn | 6.3 | 6.6 | 49.1 | 178 | 6.3 | 11.3 | 19.9 | 136 | 17.9 | 69.0 | 314 | | | 48 | 66 | 53 | 48 | 54 | 154 | 167 | 136 | 158 | 154 |
| 21.5% R.H. | 4.8 | 5.2 | Gn | 6.2 | 6.4 | 44.3 | 176 | 6.3 | 13.3 | 22.8 | 95 | 19.7 | 67.1 | 271 | | | 35 | 58 | 66 | 66 | 66 | 128 | 170 | 150 | 167 | 154 |
| 54.5% R.H. | 11.1 | 11.4 | Gn-tan tint | 6.1 | 8.4 | 46.4 | 174 | 6.0 | 14.6 | 21.4 | 108 | 23.0 | 67.5 | 282 | Brn | | 31 | 35 | 44 | 48 | 40 | 114 | 136 | 119 | 145 | 129 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.6 | Gn | 6.4 | 6.3 | 48.2 | 189 | 6.3 | 11.9 | 20.0 | 140 | 18.2 | 68.2 | 329 | | | 62 | 70 | 62 | 62 | 64 | 158 | 180 | 150 | 163 | 163 |
| 21.5%R.H. | 4.8 | 5.1 | Gn | 6.4 | 6.7 | 44.1 | 191 | 6.3 | 12.9 | 19.4 | 113 | 19.6 | 63.5 | 304 | | | 53 | 62 | 62 | 57 | 59 | 150 | 154 | 141 | 154 | 150 |
| 54.5%R.H. | 11.1 | 10.7 | Gn-Sl. Tan tint | 6.2 | 8.4 | 48.6 | 166 | 6.0 | 13.7 | 17.9 | 116 | 22.1 | 66.5 | 282 | Brn | | 35 | 40 | 48 | 44 | 42 | 136 | 145 | 123 | 150 | 139 |
| 7 Weeks Analyzed February 7, 1966 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Ref. 6.0%R.H. | 1.4 | 1.4 | Gn | 6.2 | 8.4 | 48.3 | 170 | 6.4 | 11.2 | 17.2 | 141 | 19.6 | 65.5 | 311 | Gn-Tan Tint | Grass | 48 | 40 | 44 | 40 | 43 | 132 | 141 | 150 | 150 | 143 |
| Air | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.2 | Gn | 6.2 | 6.8 | 47.6 | 157 | 6.3 | 12.5 | 17.9 | 139 | 19.3 | 65.5 | 296 | Gn | Grass | 35 | 31 | 48 | 53 | 42 | 123 | 119 | 158 | 154 | 139 |
| 21.5%R.H. | 4.8 | 5.4 | Gn | 6.2 | 7.4 | 46.6 | 155 | 6.1 | 14.5 | 20.8 | 99 | 21.9 | 67.4 | 254 | Gn-tan tint | Grass Hay | 22 | 31 | 44 | 57 | 39 | 110 | 123 | 136 | 150 | 130 |
| 54.5%R.H. | 11.1 | 11.2 | Tan-Gn | 6.0 | 10.9 | 48.4 | 153 | 5.8 | 15.5 | 17.9 | 112 | 26.4 | 66.3 | 265 | Brn | Hay | 18 | 22 | 26 | 31 | 24 | 101 | 110 | 114 | 141 | 117 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.5 | Gn | 6.4 | 6.0 | 44.6 | 169 | 6.4 | 11.8 | 18.6 | 136 | 17.8 | 63.2 | 305 | Gn-Sl | Grass | 53 | 44 | 62 | 57 | 54 | 150 | 110 | 167 | 163 | 148 |
| 21.5%R.H. | 4.8 | 5.6 | Gn | 6.3 | 7.1 | 45.4 | 152 | 6.2 | 14.5 | 19.5 | 137 | 21.6 | 64.9 | 289 | Tan-Gn | Hay | 70 | 62 | 66 | 70 | 67 | 172 | 172 | 176 | 189 | 177 |
| 54.5%R.H. | 11.1 | 11.1 | Tan-Gr | 6.0 | 9.6 | 46.6 | 162 | 6.0 | 14.0 | 17.9 | 126 | 23.6 | 64.5 | 288 | Dark Brn | Hay | 22 | 18 | 31 | 48 | 30 | 106 | 106 | 136 | 136 | 121 |
| 11 Weeks | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Ref.6.0%R.H. | 1.4 | 1.8 | Gn | 6.7 | 7.2 | 91.3 | 173 | 6.6 | 10.5 | 18.5 | 148 | 17.7 | 109.8 | 321 | Gn-tan tint | Grass | 66 | 92 | 48 | 53 | 66 | 172 | 220 | 141 | 158 | 173 |
| Air | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 1.7 | Gn | 6.7 | 6.6 | 87.2 | 164 | 6.5 | 11.5 | 19.5 | 134 | 18.1 | 106.7 | 298 | Gn | Grass | 44 | 57 | 57 | 57 | 54 | 150 | 189 | 154 | 158 | 163 |
| 21.5%R.H. | 4.8 | 5.7 | Gn | 6.6 | 7.4 | 87.9 | 150 | 6.3 | 13.5 | 21.0 | 113 | 20.9 | 108.9 | 263 | Bn-Tan tint | Grass | 40 | 48 | 53 | 48 | 47 | 145 | 172 | 145 | 163 | 156 |
| 54.5%R.H. | 11.1 | 11.0 | Brn Gn tint | 6.1 | 11.5 | 75.9 | 148 | 5.8 | 15.6 | 20.5 | 92 | 27.1 | 96.4 | 240 | Brn | Faint Hay | 40 | 44 | 53 | 31 | 42 | 132 | 154 | 150 | 145 | 145 |
| $N_2$ | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0%R.H. | 1.4 | 2.2 | Gn | 6.7 | 7.0 | 83.8 | 162 | 6.5 | 11.0 | 19.0 | 148 | 18.0 | 102.8 | 310 | Gn | Grass | 53 | 57 | 57 | 44 | 53 | 163 | 176 | 154 | 154 | 162 |
| 21.5%R.H. | 4.8 | 5.8 | Gn | 6.7 | 6.7 | 89.1 | 159 | 6.4 | 13.3 | 19.5 | 125 | 20.0 | 108.6 | 284 | Gn-tan | Faint grass | 84 | 79 | 57 | 48 | 67 | 189 | 224 | 172 | 172 | 189 |
| 54.5%R.H. | 11.1 | 11.2 | Brn Gn tint | 6.3 | 10.4 | 82.6 | 159 | 6.0 | 13.6 | 18.0 | 109 | 24.0 | 100.6 | 268 | Brn | Faint hay | 26 | 44 | 31 | 35 | 34 | 136 | 158 | 128 | 145 | 142 |

*Placed in storage December 21, 1965

In spinach stored 7 weeks at 37.8°C, the sample stored at the monolayer moisture level under nitrogen had high iodine titration value while the same sample stored in air did not. The reducing compounds responsible for the high iodine titration values are believed to be pentoses formed from the hydrolysis of pentosans. In air they disappear through oxidation while under nitrogen they are stable. Further analytical work, of course, would be necessary to prove this hypothesis.

There was a greater loss of amino nitrogen in the food stored in air than in nitrogen. The reactants involved are believed to be between oxidative-hydrolytic fragments of the chlorophyll and the free amino groups of proteins and amino acids.

For spinach stored 11 weeks at 37.8°C, the reference and samples stored in air and in nitrogen with a moisture content of 1.4% gained 0.4, 0.3 and 0.8% moisture, respectively. The same thing occurred after 12 weeks storage at 25°C. Thus, there was a latent period of 7 to 12 weeks whereby a reaction occurred which formed water. The latent period presumably was the same at -20°C, 25°C, and 37.8°C. This was typical of lipids and chlorophyll and these were probably the compounds involved. The same thing happened with freeze-dried beef stored under nitrogen at 25°C and 37.8°C with an initial moisture content of 1.5%. The amount of

water formed at 25° C was 1. 0% with 1. 3% at 37. 8° C. The reference stored at -20° C did not change in this manner. The latent period was thus the same duration as the spinach. Polymerization reactions involving the phospholipids were believed to be responsible.

The food stored at a moisture content of 4. 8% increased in water 1%. Polymerization reactions of the chlorophyll again are believed to be the cause with a latent period of less than 3 weeks at 37. 8° C and approximately 7 weeks at 25° C. The food stored at a moisture content of 11. 1% did not change in moisture since reactions which formed and consumed water balanced one another out.

The spinach stored at the monolayer moisture level under nitrogen had an enhanced iodine titration value. This was probably due to the pentoses derived from the hydrolysis of pentosans. They were formed in the same sample stored in air but were destroyed through oxidation. At a moisture content of 1. 4% there was insufficient water for such hydrolytic reactions to occur. Above the monolayer moisture level the pentoses were formed but they disappeared along with other reducing chemicals as ascorbic acid by reducing the chlorophyll to a brown compound, combining with amino groups and through participation in polymerization reaction with the formation of a brown pigment.

At this storage interval the procedure for the formol titration of the residue insoluble in water was changed. After the addition of a solution of Versene the pH was raised to 11.5 and held there for 5 minutes. Then it was lowered to below 8.0 and the formol titration then performed. The Versene was allowed to react with the spinach at a pH below 7.0. At the high pH level the amount of cations chelated was increased two fold. The amount of calcium which was combined with the carboxyl groups of the hemicelluloses was 2.2 grams per 100 grams of anhydrous spinach. The data does not indicate that there was a disappearance of carboxyl groups in any of the storage samples. The differences in the various samples appear to be within the error of the titration. Spinach contains approximately 1.15 grams of calcium and iron per 100 grams of anhydrous food while according to the amount of carboxyl groups freed upon removal of the cations by Versene there were 2.2 grams of cations present so each calcium or iron ion was associated with two carboxyl groups.

## 4. DISCUSSION OF RESULTS

### 4.1 General

In general, deterioration did occur in the stored foods. This was indicated by changes in the color, texture, and chemical results. Also, the sorption isotherm measurements indicated that structural changes took place which caused a decrease in the number of moisture sorption sites on the foods.

The chemical reactions causing the stored foods to deteriorate were very complex. Many reactions were taking place at the same time. Hence, a select number of chemical tests were run to ascertain the mechanism of a few of the reactions causing deterioration of foods during storage. Some trends were observed in the sorption isotherms and chemical evaluation results of the stored foods. These trends have been utilized to attempt to determine the moisture level at which the foods exhibited the best storage stability. It was found that the moisture level at which the food was stored had an effect on the storage stability. Foods stored at or below the moisture monolayer level showed less deterioration than foods stored at a moisture level above the monolayer value.

## 4.2 Moisture Sorption Results

In general, the measurement of the sorption isotherms showed that less moisture was resorbed on foods stored at the high moisture levels than on foods stored at the low moisture levels; on foods stored under air than on foods stored under nitrogen; and on foods stored for a longer period of time. Presumably, the reason for these results is that oxygen and moisture reacted irreversibly with the foods during storage to block reactive sites. These reactions, of course, would be more extensive at the high moisture levels, in air atmosphere, and at the longer storage intervals. While sweet potatoes followed this pattern constantly, many exceptions were shown in the beef and spinach samples. These irregularities in the beef and spinach

samples are attributed to the heterogeneity of these foods. Reference to Table 1 shows that beef consists of approximately equal amounts of protein and fat while spinach consists of protein, fat, and carbohydrate in ratios of approximately 1:2:1. Sweet potatoes are basically all carbohydrates.

Plots of the sorption isotherms for the foods show that the beef-moisture sorption isotherms belong to Type II, sigmoid-shaped isotherms. The moisture isotherms of sweet potatoes and spinach were stepwise isotherms. These stepwise isotherms were more pronounced for the foods stored at high moisture levels, for foods stored under air, and foods stored for longer intervals. Also the stepwise isotherms were more pronounced for stored spinach samples than for stored sweet potato samples.

Stepwise isotherms for multilayer absorption on uniform and non-uniform surfaces were examined by Champion and Halsey.[15] These authors concluded that stepwise isotherms were characteristic of absorption on uniform surfaces while the smoothness of a large variety of isotherms were due to surface heterogeniety. The stepwise isotherms for the sweet potatoes and spinach, therefore, might indicate that the surfaces of spinach and sweet potatoes were more uniform than the surface of ground beef. This heterogeniety in the beef has been pointed out earlier.

One of the trends noted in the measurement of the sorption isotherms on the stored foods was that there was generally lowered moisture sorption on foods after storage. This was exemplified by the isotherm values and the BET values. One of the possible reasons for the lower moisture sorption is that moisture and oxygen react with the active sites of foods. After an appreciable time in storage, therefore, there are fewer active sites left for the sorption of water. In addition, other reactions may be occurring in the foods on storage which utilize the available active sites for moisture sorption. The existence of these reactions was evident in the chemical evaluation of the stored foods.

As was indicated earlier, the Fugassi-Mitchell equation was derived to analyze the sorption of vapors on a swelling gel. Since food resembles a swelling gel, the Fugassi-Mitchell equation was applied to the analysis of the sorption isotherms. The only discernible trend in the Fugassi-Mitchell values was that A increased as the temperature decreased and K increased as the temperature increased. These observations were made on beef. Negative A, and K values were obtained for spinach and sweet potatoes. The negative results were meaningless.

Foods were specifically chosen for this study which were found to yield negative A values. Previous studies[2] indicated that beef and sweet potatoes gave negative A values and both of these foods as well as spinach

exhibited storage instability. Negative values, therefore, appeared to indicate a basic instability of the foods in contact with moisture.

On the other hand, negative as well as positive Fugassi-Mitchell constants gave values for $(H_2O)_{exterior}$ which was very close to the BET monolayer value. This phenomenon is not fully understood at present, and no significance can be attached to the agreement o disagreement between the BET monolayer value and $(H_2O)_{exterior}$.

Heat of sorption results were impossible to calculate for the stored foods due to inversions of the isotherms occurring even after short storage intervals. These inversions made it impossible to use the Clausius-Clapeyron equation.

All results indicate that an irreversible chemical interaction was occurring between water and the foods. This can be seen from the irrevisibility of the moisture sorption isotherm of the original foods which took two weeks to measure. There was some bound water which could not be removed from the foods upon evacuation.

## 4.3 Chemical Evaluation Results

### 4.3.1 Sweet Potatoes

The loss in color of sweet potatoes stored 3 weeks at 25°C was believed to be due to the destruction of beta carotene by oxidation. The carotenoids are a class of labile easily oxidizable pigments which are noted for their preferential solubility in fats and solvents for fats. The color varies with the number and conjugation of the double bonds. Each carotenoid

pigment may occur in several interconvertible modifications -- the common, relatively more stable, trans isomer and its several less common and less stable cis isomers. Inter-conversion of the trans form and its several cis modifications takes place when the solutions of each isomer are heated or exposed to light and iodine. Although carotenoids are usually present in fats, some may be linked to proteins. They are invariably associated with the chlorophylls and are an essential part of the photosynthetic apparatus.[16]

These compounds are terpenic in that they can be formulated or constructed from isoprene residues. The two chief hydrocarbons of this series, lycopene and carotene, have the formula $C_{40}H_{56}$. Although the hydrocarbons do not contain 8H atoms, they are tetraterpenes containing 8 isoprene units. Conjugated double bonds differ radically from the same number of isolated double bonds. An approximation of the facts is that a system of conjugated double bonds involves something like a uniform flux of valence forces all along the chain.[17]

The decrease in amino nitrogen and simultaneous increase in the titratable acidity for foods stored at the high moisture levels may be due to the formation of carbonyl compounds through the oxidative-hydrolytic decomposition of the lipids. The carbonyl compounds then react with the amino groups of free amino acids and proteins. This would result in the various components of the sweet potatoes being linked together with the

formation of large macromolecules which would give the food a hard and rubbery texture.

An increase in the titratable acidity and concomitant decrease in amino nitrogen of sweet potatoes stored 7 weeks at 75°C could possibly be due to the nullification of the amino groups of amino acids through chemical reactions leaving the carboxyl groups free to exert their acidity. Some of the compounds thought to be involved are oxidative and hydrolytic products of the lipids as carotenoid pigments. In sweet potatoes stored above the monolayer moisture level in air, the lipids enter into oxidative as well as hydrolytic reactions and oxidative reactions below the monolayer in air. The amino nitrogen and titratable acidity of the food at the monolayer moisture level appear to be stable.

In Phase 2 of this contract, sweet potatoes stored in air at 25°C after 9 weeks had a greater loss in percent fat extracted and beta carotene at the lowest and highest moisture levels of storage. These data would further support the data outlined in the previous paragraph. The percent sugar as dextrose had declined the same amount for all the storage conditions. This would indicate that reducing sugars were not responsible for the decline in amino nitrogen.

The compounds in the aqueous extract of the sweet potatoes responsible for reacting with iodine are believed to be ascorbic acid, carbonyl compounds

formed from the oxidative deterioration of lipids as the carotenoids and dextrins derived from the hydrolysis of the starch. In order to ascertain their identity and their separation, characterization and quantitative determination would be necessary. Possibly the aqueous extract could be fractionated by means of column chromatography. The fraction containing the ascorbic acid could be reacted with 2, 4 dinitrophenylhydrazine and the color measured photometrically. Suitable derivatives of the compounds in the fraction containing the aldehydes could be made and thereby identify and determine the amount of aldehydes present. The fraction containing the dextrins might be treated with alcohol to precipitate the dextrins. The precipitate could then be separated and hydrolyzed by acid and the amount of glucose determined. Even though these compounds were not separated from each other, the data nevertheless would provide considerable insight with regard to the production and disappearance of compounds which are capable of being oxidized by iodine.

In sweet potatoes stored 3 weeks at 37.8°C the reactions involved in the disappearance of the amino nitrogen are believed to be between the decomposition products, hydrolytic as well as oxidative, of lipids as the carotenes and the amino nitrogen of free amino acids and proteins. Below the monolayer moisture level the lipids are prone to be oxidized and above this level they are prone to be oxidized and hydrolyzed. At the monolayer

moisture level both reactions occur but hydrolysis is less than at a higher moisture level and oxidation is less than at a lower moisture level.

The weight of material involved in the above reaction is rather small, say a maximum of 60 mgs of amino nitrogen and 200 mgs of lipid per 100 grams of anhydrous sweet potatoes. The changes in texture, appearance and rehydratability though are large. The lipid oxidative-hydrolytic products link together large molecules of starch and protein together intermolecularly through polar groups as amino and hydroxyl causing profound changes in the physical properties of the food. The weights of the polar groups involved were small but the weight of the material linked together was large.

4.3.2 Ground Beef

The change in color of the ground beef stored at 25°C in air at a moisture content of 7.9% from a red to a tan green may be accounted for by the decomposition of the heme of the myoglobin. Heme is very unstable and among the many potential decomposition products are the bile pigments which are green and or yellowish-brown in color. The first step in the formation of bile pigments appears to involve an oxidative scission of the porphyrin ring to produce carbon dioxide and an open ring compound. In Phase I of this contract, headspace gas analysis of beef stored at high moisture levels showed the consumption of oxygen and the production of carbon dioxide. In general, these pigments are green in color and the opening of the porphyrin ring has apparently rendered the iron labile so that it is easily split off.[18]

Myoglobin is a water-soluble protein similar to hemoglobin with a molecular weight of 17,000 as compared to 67,000 for hemoglobin. It is a heme protein or lipoprotein which combines reversibly with oxygen. Its affinity for oxygen is higher than that of hemoglobin so that the intracellular pigment may be fully oxygenated at oxygen tensions which cause unloading of the blood pigment. The myoglobin thus acts as a store for oxygen.[19]

The enhanced activity of the pancreatic lipase on the sample of beef stored at 25° C for 3 weeks may be explained by the presence of bile pigments which were derived from the breakdown of the heme of the myoglobin. Lipase is a water soluble enzyme with an optimum pH of 7. The bile pigments accelerate lipase activity by permitting closer contact between the water-soluble lipase and the fat globule. Digestion is further enhanced by the bile pigments through removal of the end products of lipase digestion.[20] The putrid odor of the digest of this sample may be attributed to the more complete digestion of the beef.

The low percentage of free fatty acids liberated as oleic acid for beef stored 3 weeks at 25° C under nitrogen is believed to be due to the modification of the phospholipid moiety of the proteins during storage. The phospholipid moiety is modified to such an extent that it is not readily attacked by lipase. Fat peroxides presumably have de - composed to carbonyl compounds which are characterized by extreme

unsaturation or ability to add a great variety of agents. The activated form will add H and the rest of the addend may add to the electronically deficient carbon or elsewhere in the molecule if a shift first takes place (1:4-addition). Water is often lost.[21] The meat stored in air at this moisture level did not show as great a diminished attack by lipase. Possibly the chain reaction in which the carbonyls take part was stopped by oxidation of the activated forms.

Apparently, the reason for the difference in the rate of attack of the phospholipids extracted from beef stored 7 weeks at 25° C by lipase was due to a difference in the physical properties of the fat. The phospholipids which were not readily attacked by the lipase when isolated on the Buchner funnel were quite voluminous, about double the volume of the others. An interesting experiment would be to take some fresh beef, separate the fat without first dehydrating the meat, precipitate the phospholipids with acetone and digest them with lipase. The phospholipids would probably go rapidly into solution in the same manner as the high moisture beef samples. If this were true, then the reference and samples stored below the monolayer moisture level have undergone a physical change whereupon lipase attacks them very slowly. This physical change probably occurred during the dehydration and during the storage of the beef.

The lipids soluble in acetone from all the storage samples were digested more completely by the lipase than the reference. The reason may be a change in the physical properties of the fat on storage which permits the enzyme to digest it more completely.

The consumption of water by beef stored 11 weeks at 25° C may be explained by the following. A reaction which consumes water is the hydrolysis of the fat with the formation of free fatty acids which occurs to about the same extent when the food was stored in air or under nitrogen. A reaction which forms water was the decomposition of peroxides of the lipids to form carbonyl compounds which polymerize with water as one of the many possible reaction products. This reaction apparently occurred only in the meat packaged under nitrogen because in the presence of air the chain reaction was broken before any water was produced. The beef packaged under nitrogen below the monolayer moisture level increased 1.3% in water which presumably was derived from a polymerization reaction of the lipids. Since hydrolysis at this low a moisture level is quite minimal, more than this amount of water probably was not formed. The defatted residue weighed 1.1 grams less than that of the reference presumably due to a polymerization reaction with the formation of water from the phospholipid moiety of the lipoproteins. Likewise therewas 0.9 gram less fat extracted from this sample than from the reference due to polymerization reactions of the lipids and more than likely it was the phospholipids because they are quite

highly unsaturated. The combined weight of the defatted residue and lipids was 2 grams less than the reference although there was only 1.3 grams of water formed. This can be accounted for by volatile reaction products other than water being formed in the polymerization of the carbonyl compounds derived from the lipids.

The elevated titratable acidity in the two samples stored above the monolayer moisture level may be attributed to a chemical reaction between amino groups and hydrolytic products of the lipids particularly phospholipids. At this moisture level there were less phospholipids recovered by the acetone precipitation procedure. One of the hydrolytic products of the phospholipids is phosphoric acid and this compound being very acid would readily react with basic groups of the proteins such as the strongly basic ring nitrogens of histidine.

The reason for the difference in attack of the phospholipids by lipase in beef stored 3 weeks at 37.8°C was that the physical properties of the beef had been altered in such a way that the difference in the speed of attack by lipase had been smoothed out. In this sample of extracted fat, the last traces of ether were removed by holding the fat for 5 hours in a vacuum oven at 70°C with 29" of vacuum. The fat from the beef stored for 7 weeks at 25°C was held under the same conditions in the vacuum oven for 2 hours. It is believed that the extra 3 hours at 70°C had brought about a change in the lipids.

There were other indications that the physical properties of the phospholipids isolated from the reference and storage samples were nearly the same. When the phospholipids were removed by filtration, filtration was very rapid for all the samples and the volume of the precipitate was the same. In those storage samples where the reference and 2 samples stored below the monolayer moisture level were attacked very slowly by the lipase, the speed of filtration of the phospholipids was slow and the volume of the precipitate approximately double those which were attacked by the lipase very rapidly.

A small difference in the amount of ether left in the fat extracted from the beef may cause a large difference in the weight of the phospholipids isolated. In this storage period where the phospholipids were held for 5 hours at 70°C, about 4 grams of phospholipids were recovered from 100 grams of beef. When the sample was held for 2 hours at 70°C, as was done for the storage period of 7 weeks at 25°C, 8 grams of phospholipids were isolated. The amount of ether remaining in the lipids in each case was probably small. When the ether was allowed to evaporate from the lipids by placing the extract in the hood overnight, it was found that the ether accounted for about 10% of the weight of the residue. It is almost impossible to remove all the ether since it is so tenaciously bound to the fat.

4.3.3 Spinach

When the spinach stored 4 weeks at 25°C was titrated with iodine, the aqueous extract of the spinach stored under nitrogen at the monolayer moisture level contained a greater concentration of reducing compounds than the reference and other storage samples. The source of the reducing compounds may be from the hydrolysis of pentosans associated with the hemicelluloses as pectins. They were not formed in the food stored under nitrogen below the monolayer moisture level because there was an insufficient amount of water present for hydrolysis to take place. Above the monolayer moisture level under nitrogen, the pentosans were hydrolyzed but the hydrolytic products were not stable as at the monolayer moisture level. The reason for this is that sufficient water was available so that the pentosans could take part in such diverse reactions as polymerization to a brown pigment and reduction of such extremely reactive groups of the chlorophyll molecule as the ethylene side chains, methylene bridges and unsaturated bonds of the porphyrin rings. These reactions were more than likely responsible for the change in color of the chlorophyll from a green to a brown. The greater stability of the pentoses at the monolayer moisture level may account for the high static charge observed in these samples.

In the spinach stored 8 weeks at 25° C the reducing compounds were formed from the pentosans as a result of their participation in polymerization reactions or their hydrolysis to pentoses. The polymerization reaction, which has water as one reaction product had progressed to the extent that the moisture increased from 4.8 to 5.4%. These reactions did not occur as much in the spinach stored in air at the monolayer moisture level so this food appears more stable in the presence of air than it does under nitrogen by this criterion.

The amino nitrogen of the aqueous extracts for the spinach stored in air and under nitrogen above the monolayer moisture level decreased with the loss a little less under nitrogen. The reactants are soluble proteins, amino acids, lipids and their oxidative-hydrolytic products and sugars normally present or more than likely formed from the hydrolysis of the pentosans. The product has the amino group blocked so that the carboxyl group of the amino acid can exert its acidity thus accounting for the elevated titratable acidity of the aqueous extract for these two samples. The magnitude of this change for that portion of the spinach insoluble in water was not large enough to be picked up by the formol titration.

In spinach stored under nitrogen, the oxidation-reduction equilibrium shifts from an oxidized to a reduced state. Reducing compounds as ascorbic acid, reducing sugars as pentoses etc. initially present are free to reduce the very reactive and highly unsaturated chlorophyll molecule which results in the formation of a brown color and hay-like odor. Also under these reducing conditions, the chlorophyll fragments formed by

reduction could also enter into polymerization reactions. On storage under nitrogen at the monolayer moisture level the pentosans are hydrolyzed liberating pentoses which are powerful reducing agents and reduce the chlorophyll even more. At the monolayer moisture level in the presence of air the chlorophyll would not be reduced because compounds as ascorbic acid, reducing sugars as the pentoses were oxidized. These compounds take part in reactions which form water but polymerization reactions do not progress to the extent that a brown pigment was formed. Possibly in the presence of air the chain reaction between carbonyls derived from the chlorophyll and other compounds involved were stopped through their oxidation.

Above the monolayer moisture level in air the pentosans had been hydrolyzed to the extent that the reducing sugars liberated have combined with all the oxygen in the package and in addition have reduced or combined with the chlorophyll to the extent that it has a hay-like odor and brown color. The same food packaged under nitrogen, the brown color and hay-like odor were more intense because it was not necessary to first remove the oxygen in the package before reducing the chlorophyll.

The hay-like odor and brown color of the aqueous extracts of spinach stored 3 weeks at 37.8° C was attributed to the polymerization of the chlorophyll and pentosans which resulted in the disappearance of fine spinach particles with the texture of the food becoming more coarse and

woody in character. The amino nitrogen also decreased so it probably also took part in these reactions. The brown color was believed to be due to a loss of some of the double bonds of the chlorophyll plus polymerization reactions of the pentosans. The pentosans were hydrolyzed to pentoses which then reduced a sufficient number of double bonds in the chlorophyll molecules so that its color changed from a green to a brown.

In spinach stored 7 weeks at 37.8°C, it was found that a more intense brown color was observed in the aqueous extract of food stored under nitrogen than the same sample stored in air. The probable reason for this is that the pentoses polymerize with the formation of water and a brown pigment. In air, the pentoses do not polymerize because they are bleached by being oxidized.

In spinach stored 11 weeks at 37.8°C, the data from the titratable acidity and the number of carboxyl groups liberated by chelation of calcium indicate that the molecule of hemicellulose insoluble in water may be visualized as a straight chain folded around several times. The folds are held together by means of hydrogen bonds and calcium was through combination with carboxyl groups of the gluconic acid residues. When the molecule is unwound by the Versene, each fold has a titratable acidity in excess of 14.

## 4.4 Conclusions and Interrelationships Between Moisture Sorption and Chemical Analysis Results

The chemical analysis and moisture sorption isotherm results tended to complement each other. In general, the moisture sorption isotherm values and the BET values showed that less reactive sites were available for the water molecule for food stored over a large storage interval. The number of reactive sites decreased indicating that structural changes had occurred in the foods on storage. Chemical evaluatibns of the stored foods further elucidated the typical chemical reactions occurring in the deterioration of stored foods. In one instance, a very good correlation was found between the moisture levels of storage and chemical results. In ground beef stored 7 and 11 weeks at 37.8°C, the peroxide value increased with increased moisture storage levels. There was no change in the peroxide value for food stored 7 weeks at 37.8°C. The results indicated an oxidation of the fatty acids at the high moisture levels.

The study showed that freeze dehydration of the beef, sweet potatoes and spinach to a moisture content of 1.2% was detrimental to these foods. Also, it is inadvisable to store these foods at this low moisture level. There are not enough water molecules to maintain the spacing between the nonpolar groups of the lipids, proteins, lipoproteins, etc. These groups reorientate themselves with respect to one another to form a waxy water-repellant coating over the starches, hemicellulose, proteins, etc.

Probably the solubility relationships between the various food components change at the low moisture level of 1.2%. Chemicals insoluble at a high moisture level like carbonyl compounds derived from the oxidative decomposition of the lipids react with each other and other food components like amino groups and unsaturated linkages of the lipids. A portion of the lipid is then changed to water and volatile compounds. The lipid modified chemically in the above manner are digested less completely than those from the control sample.

Storage of these freeze-dried foods at the 1.2% moisture level under nitrogen was more detrimental than storage in air. In the presence of air, the carbonyl compounds formed on storage are immediately inactivated through oxidation by the free oxygen in the package. Packaging under nitrogen reduces the oxygen content of the food to the optimum value where deterioration takes place.

The ground beef, sweet potatoes and spinach were most stable at the monolayer moisture level. When the dehydration of the food is stopped at the monolayer value there is probably enough water present to preserve the natural space relationships of the nonpolar groups of such food constituents as the fats and oils, phospholipids, lipoproteins, proteins, etc.

The beef, sweet potatoes and spinach deteriorated in the same manner. After a latent period, oxidative-hydrolytic decomposition products of the lipids began to appear and reacted with the amino nitrogen of the free amino acids and proteins. These reactions caused the food to change in odor, flavor and to take on a woody and/or rubbery texture. The higher the moisture content at which the food was stored, the shorter was this latent period. The spinach deteriorated by reduction. Reducing compounds initially present or formed on storage reduced the chlorophyll causing it to turn brown and assume a hay-like odor.

The following procedure is recommended for the production of stable and nutritionally sound dehydrated foods. Foods in their fresh state are in a reduced condition and upon dehydration tend to change to an oxidized state. This has to be prevented. The oxidation-reduction equilibrium should be kept in the reduced state where the concentration of oxidants would be nil. A sulfur compound soluble in water as cysteine and a sulfur compound soluble in lipids as iso-octyl thioglycolate would be blended in the food. In the absence of oxygen the moisture would be removed evenly from the food down to the monolayer value. The dried food would be placed in a container and flushed with a gas as nitrogen or carbon dioxide until the effuent gas was devoid of oxygen. The container would then be sealed.

The oxidation-reduction equilibrium of the food should not be shifted too far to the reduced state since this could very well be more harmful than if the equilibrium were in the oxidized state. The additives should stabilize the foods at the normal reduced state. Compounds such as ascorbic acid, diacetyl, acetone, etc., would be neither oxidized nor reduced. The freshness of the food would be preserved causing it to be easily and readily digested

The analytical tests were selected to identify the nature of the chemical reactions involved in the deterioration of the foods. It came quite as a surprise to discover that the foods changed physically. One example is the change in the collagen. It soon became apparent that the physical changes were more important than the chemical changes because more food was involved. In most instances the weight of the lipids which decomposed through oxidative-hydrolytic reactions was only a few milligrams. The flavor and texture was damaged but the food was not hurt nutritionally.

The collagen changed to a more soluble form. This change is important since meat is one-third collagen. The collagen is a waste product since it is not digested by the enzymes of the digestive tract. It is also responsible for the toughness of meat. The meat after storage at low moisture levels might be more tender and more of the collagen would be broken down in the digestive tract and utilized by the body.

The superior quality characteristics of foods stored at the monolayer moisture level might have been verified by several additional chemical tests, if time and funds had been available. The changes in the lipids which occurred in foods dehydrated below the monolayer value affected markedly the rehydration characteristics. These changes in lipids were evidently not reversible for when the moisture in the over dehydrated food was readjusted to the monolayer value the rehydration characteristics were not improved. This irreversability of some physical or chemical changes could be of great significance in explaining the variation in rehydration characteristics of freeze dehydrated foods. It might account for difference in rehydration of various size particles within a batch as well as differences between batches of food which were presumably dehydrated to the same moisture level. It points out definitely the importance of process control particularly when other variables are being evaluated as to effects on storagability. A detailed study of these irreversible changes could be very significant in determining the variations in moisture removal which could be tolerated with affecting the rehydration of the food.

The loss of amino groups was greater in foods stored at the monolayer moisture level than in those stored at moisture levels either above or below the monolayer value. This loss in amino groups could probably be prevented if the food were stored in the complete absence of oxygen.

## SUMMARY

Water sorption isotherm measurements were made on freeze-dried beef, spinach and sweet potatoes stored 3, 7, and 11 weeks at 37.8°C. These measurements were made to determine a moisture range for optimum storage stability of the freeze-dried foods. The BET and Fugassi-Mitchell theories were applied to the sorption isotherms to interpret the results. According to Salwin[22], a monomolecular layer of water as ascertained by the BET theory may be regarded as a protective film which protects the food particles from attack by oxygen. It was stated previously in this report that oxygen was responsible for the deterioration of dehydrated foods. Salwin[22] points out that the monolayer does not represent a continuous film but more accurately corresponds to the number of available reactive absorption sites in the fat, carbohydrate and protein components of the food. In addition, the bond energy of absorbed water could inhibit the interactions between polar groups or adjacent protein or carbohydrate molecules. This would preserve the reconstitutability, hydratability and texture of the food.[22]

Since food behaves like a swelling gel, the Fugassi-Mitchell equation was applied to the results of the isotherm measurements. The Fugassi-Mitchell constant A and K were determined for the freeze-dried beef, spinach and sweet potato at various storage intervals. The constant A represents the total number of moles of sorption sites per gram of food and K is the rate of the overall reaction

$$\underset{\text{(surface)}}{H_2O} + D \rightleftharpoons D \cdot H_2O$$

where D represents the number of interior sites in the food. For beef stored three weeks at 37.8°C, the 22° and 37.8°C isotherms showed an increase in the value of A and a corresponding decrease in the value of K with moisture content. The values of A for beef stored under nitrogen were higher than the corresponding values of A for beef stored under air. In addition, the values of A for the 22° isotherms were higher than those for the 37.8°C isotherms. This was generally true for values of A for beef stored 7 and 11 weeks. From the above results, it appears that more moles of sorption sites per gram of beef were present at 22°C than at 37.8°C and for beef stored under $N_2$ than for beef stored in air. One reason for these results might be an increase in the number of chemical interactions which occurred between adjacent polar groups in proteins and carbohydrates for beef stored under air and at higher temperatures. This would leave fewer sites for the absorption of water. The increase in the value of A with increasing moisture content could also possibly be attributed to the absorption of more than one molecule of water per site. The Fugassi-Mitchell equation assumes that one molecule of water is absorbed per site.

As stated previously, K represents the equilibrium constant for the reaction between surface sorbed water and the internal sites. The constant K for beef was found to decrease with increasing moisture content and increase with increasing temperatures. These results indicate that for beef stored at the higher moisture levels, the rate of diffusion of surface bound water to the internal sites decreased. This is reasonable since more sites near the surface are filled thereby increasing the distance a surface sorbed water molecule diffuses to the interior sites. Also, the rate of diffusion of surface bound water to the internal sites increased with increasing temperatures.

Table 51 gives a short summary of the conclusions drawn from the analytical and chemical changes in the foods stored 3, 7, and 11 weeks below, at and above the monolayer moisture level at 37.8°C. From a consideration of the BET values for foods stored 3 weeks under $N_2$, the rate of rehydration at practically all moisture levels was spinach > sweet potatoes > beef. The table also shows that spinach deteriorates very rapidly at the higher moisture levels.

The desorption results indicate that hysteresis is taking place on the stored foods. Also some water is absorbed irreversibly. The irreversibility is due to the chemical interactions occurring between the foods and moisture. The hysteresis loop in the sorption-desorption cycle may arise from a number of causes--e.g., chemical interactions and capillary condensation. It is believed that the hysteresis effect is due to chemical interactions rather than capillary condensation. In the sorption phase, insufficient water is available to effect capillary condensation at low relative pressures when the first and second monolayers are forming. Since most of the water appears to be transferred to internal surface sites, capillary condensation would be delayed until saturation of the surface sites is approached.

Table 51 Summary of Significant Results in the Interrelationships Between Moisture and the Storage Stability of Foods at 37.8°C

| Food | Moisture Level(%) | BET Monolayer * Value for Foods Stored 3 Weeks Under $N_2$ 22°C | BET Monolayer * Value for Foods Stored 3 Weeks Under $N_2$ 37.8°C | Physical & Chemical Changes on Storage ** | Conclusions |
|---|---|---|---|---|---|
| Ground Beef | 1.4 | 41.9 | 33.7 | Stable color for food stored under $N_2$, color changed to reddish tan for food stored in air, Fugassi A values at 22°C and 37.8°C increase with increased storage. | Beef appears to be more stable just below the monolayer level $N_2$ atmosphere. The beef is uncooked and enzymatic reaction probably has an effect at higher moisture levels. |
| | 3.5 | 36.1 | 32.2 | Color change from red to brown tint for food stored under $N_2$, Fugassi A values at 22°C decrease with increased storage, opposite effect observed at 37.8°C for food stored under $N_2$. | |
| | 7.6 | 36.1 | 32.6 | Color change from tan tint to reddish tan for food stored under $N_2$. Bright brown color observed for food stored in air. Negative Fugassi A values at 22°C occur for food stored under $N_2$. At 37.8°C, Fugassi A values increase with increased storage. | |
| Spinach | 1.4 | 58.1 | 54.9 | Color stable for spinach stored under $N_2$ and air atmospheres, elevated iodine titration, negative Fugassi values. | Spinach appears to be more stable at the monolayer moisture level. Fewer reducing substances are found in spinach stored in air than in nitrogen. |
| | 4.8 | 56.5 | 43.1 | Color stable for spinach stored under $N_2$ and air atmospheres, sample stored in nitrogen exhibited higher iodine titration, Negative Fugassi values, amine $N_2$ greater for sample stored in nitrogen atmosphere. | |
| | 11.1 | 50.3 | 39.4 | Spinach grows at this moisture level, rapid deteriorations detected by production of hay-like odor and brown color of aqueous extract, negative Fugassi values. | |

* Wm(mg/g), milligrams of moisture sorbed per gram of dry food at the BET monolayer

** Summary of changes taking place over storage intervals of 3, 7 and 11 weeks

Table 51 (Continued)

| Food | Moisture Level | BET Monolayer Value for Foods Stored 3 Weeks Under $N_2$ 22°C | 37.8°C | Physical & Chemical Changes on Storage* | Conclusions |
|---|---|---|---|---|---|
| Sweet Potatoes | 1.2 | 49.5 | 57.0 | Faded color; elevated iodine titration, mgs. amine of $N_2$ derived from titration of residue increased from 12 to 20 for food stored under $N_2$, elevated iodine titration value. | Sweet potato appears to be more stable at the monolayer level under $N_2$ atmosphere. |
| | 4.4 | 39.0 | 37.2 | Color stable on food stored under $N_2$, mgs. of amine $N_2$ derived from titration of residue increased from 11 to 20 for food stored under $N_2$. | |
| | 11.0 | 38.5 | 29.5 | Color turned from orange to orange brown color for food stored under $N_2$, mgs. of amine $N_2$ increased from 11 to 16 for food stored under $N_2$, elevated iodine titration value. | |

*Summary of changes taking place over storage intervals of 3, 7 and 11 weeks.

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13. ABSTRACT

Investigations were made on the moisture sorption behavior and the chemical stability upon storage of freeze-dried beef, sweet potatoes and spinach. Moisture sorption isotherms were determined at 22.0 and 37.8 C using a constant temperature spring balance. On the basis of these isotherms the B.E.T. monomolecular layer of water, heat of adsorption and constants of the Fugassi-Mitchell equation were determined. The foods were stored at three moisture levels, representing values below, at and above the B.E.T. monolayer, under air and under nitrogen, at 25 and 37.8 C for 3, 7 and 11 weeks. To foods were evaluated by a total of ten chemical tests and by limited sensory evaluation. Results showed that deterioration was more pronounced for foods stored under air than under nitrogen. In addition, foods stored at high moisture contents showed more deterioration than foods stored at lower moisture levels. All foods were more stable at the monolayer moisture level.

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| Storage stability | 7 | | 8 | | 6,7 | |
| Sorption | 7 | | 8 | | 6,7 | |
| Dehydrated foods | 7 | | 9 | | 6,7,9 | |
| Freeze dried foods | 7 | | 9 | | 6,7,9 | |
| Isotherms | | | 8 | | | |
| Water vapor | | | | | 6,7 | |
| Temperature | | | | | 6 | |
| Nitrogen | 5 | | | | | |
| Air | 5 | | | | | |
| Packaging | 6 | | | | | |