MELPAR INC
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FINAL REPORT (Phase I)

INTERRELATIONSHIPS BETWEEN STORAGE STABILITY AND MOISTURE SORPTION PROPERTIES OF DEHYDRATED FOODS

Period Covered

27 March 1964 to 26 March 1965

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1. INTRODUCTION

This report describes the work completed during the first phase of Contract # DA 19-129-AMC-252(N). The objective of the program was to study the interrelationships between storage stability and moisture sorption properties of dehydrated foods selected as representatives of various types (e.g., high sugar, protein, starch). The foods that were studied are:

Banana flakes
Egg yolk solids
Oven-dried cooked ground beef
Shrimp
Freeze-dried apple
White potato
Sweet potato
Rice
Non-fat dry milk solids

The storage stability of the foods was determined as a function of storage temperature and moisture level by chemical analysis and taste panel evaluation.

In order to maximize the usefulness of this report as a reference document it has been organized so that the data and discussions pertaining mainly to the sorption measurements are together in Section 2. Section 3 describes the studies on storage stability of each of the foods separately. Section 4 contains general comments relating to the over-all program and to the interrelationships between the sorption measurements and storage stability.

2. WATER SORPTION MEASUREMENTS

2.1 Experimental Program

2.1.1 <u>Constant Temperature Sorption Apparatus</u>

Sorption isotherms of water vapor onto dehydrated food powders were measured using a modified McBain-Baker[1] sorption balance. This is a constant temperature spring balance which can be loaded with food, evacuated, and filled with pure sorbate vapor. The apparatus used in this study is shown in Figures 1, 2, and 3. It consisted essentially of a source of pure water vapor, large expansion bulbs, a differential manometer, six sorption tubes, vacuum pumps, and a temperature controller.

The constant temperature box was constructed of 3/4-inch thick plywood except for the front, which was 1/4-inch thick Plexiglas. The front and one side are removable to enable work to be done on the vacuum system. The temperature in the box was maintained by two 200-watt light bulbs whose power input could be varied by slide wire resistors. One of the light bulbs was on continuously and its power input was set so that it heated the box to within one-half degree of the desired temperature. The other light bulb was an intermittent bulb used as a fine control. An Aminco (American Instrument Company, Silver Springs, Maryland) temperature controller controlled the intermittant light bulb. Two heavy duty squirrel cage centrifugal blowers curculated the air in the box. The box temperature could be maintained within $\pm 0.05^{\circ}C$ of a fixed setting between

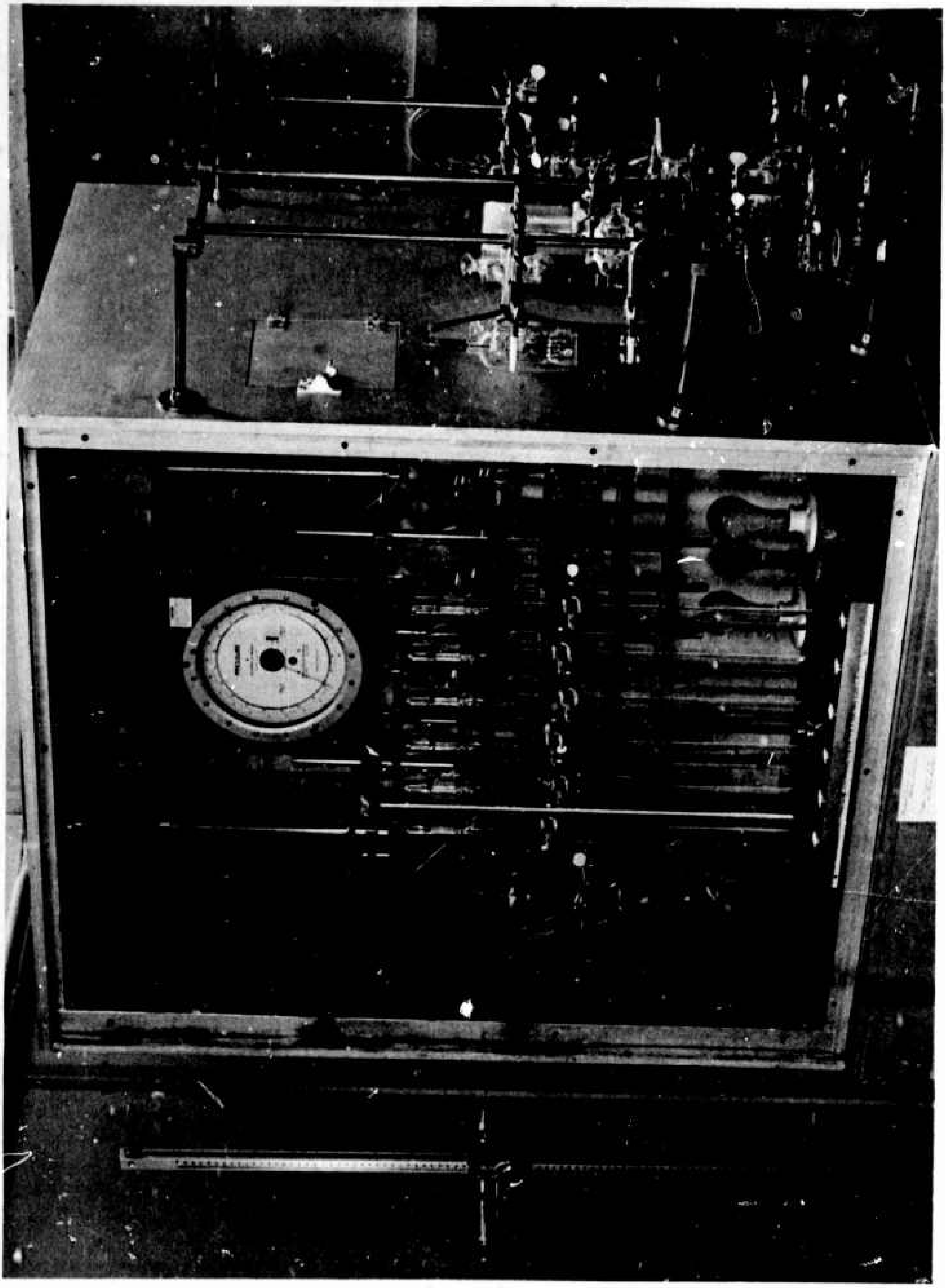

Figure 1. View of Constant Temperature Box, Sorption Apparatus, and Cathetometer

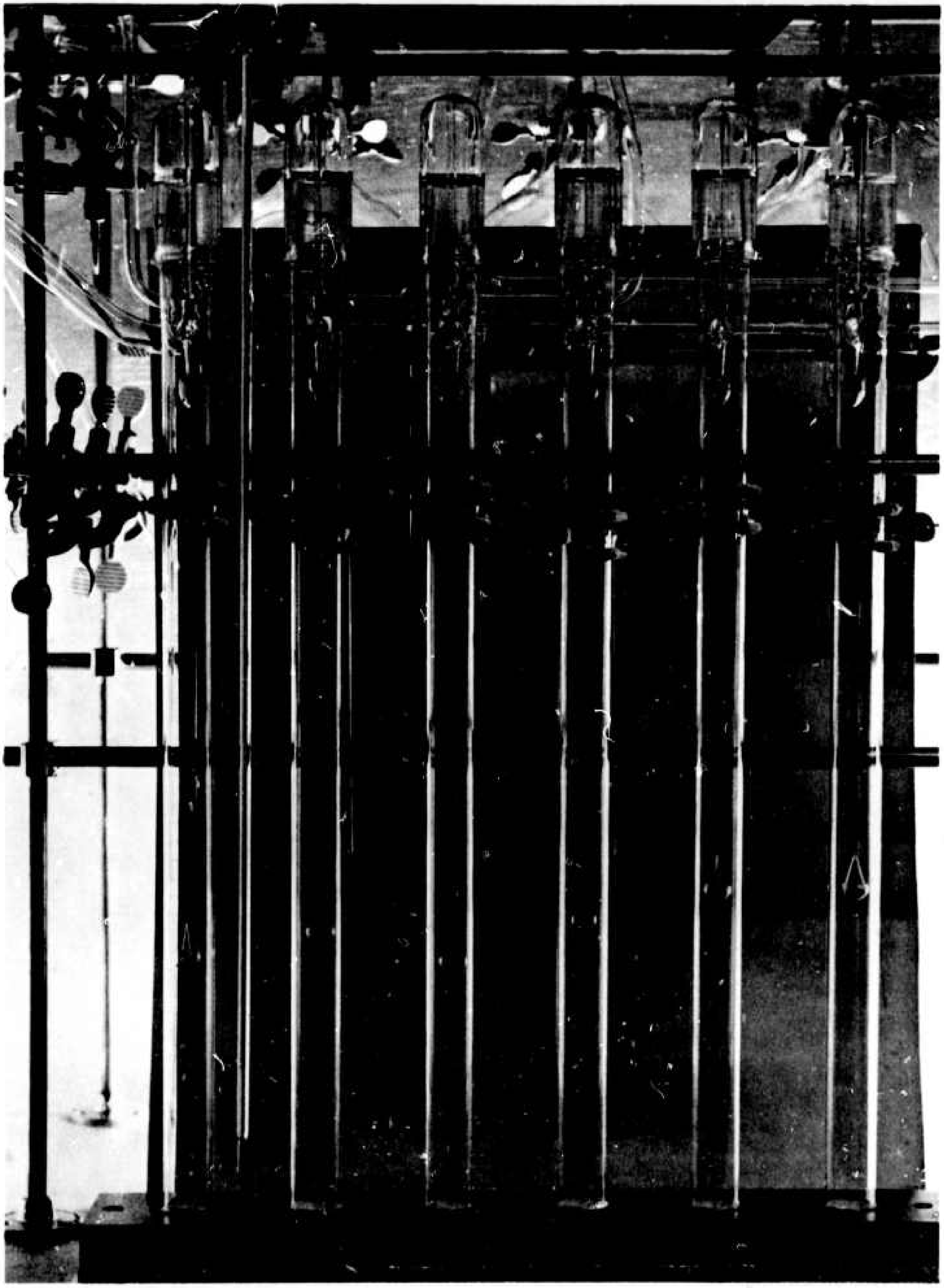

Figure 2. Close-up View of Sorption Tubes Showing Ni-Span-C Springs and Foods Suspended in Quartz Buckets

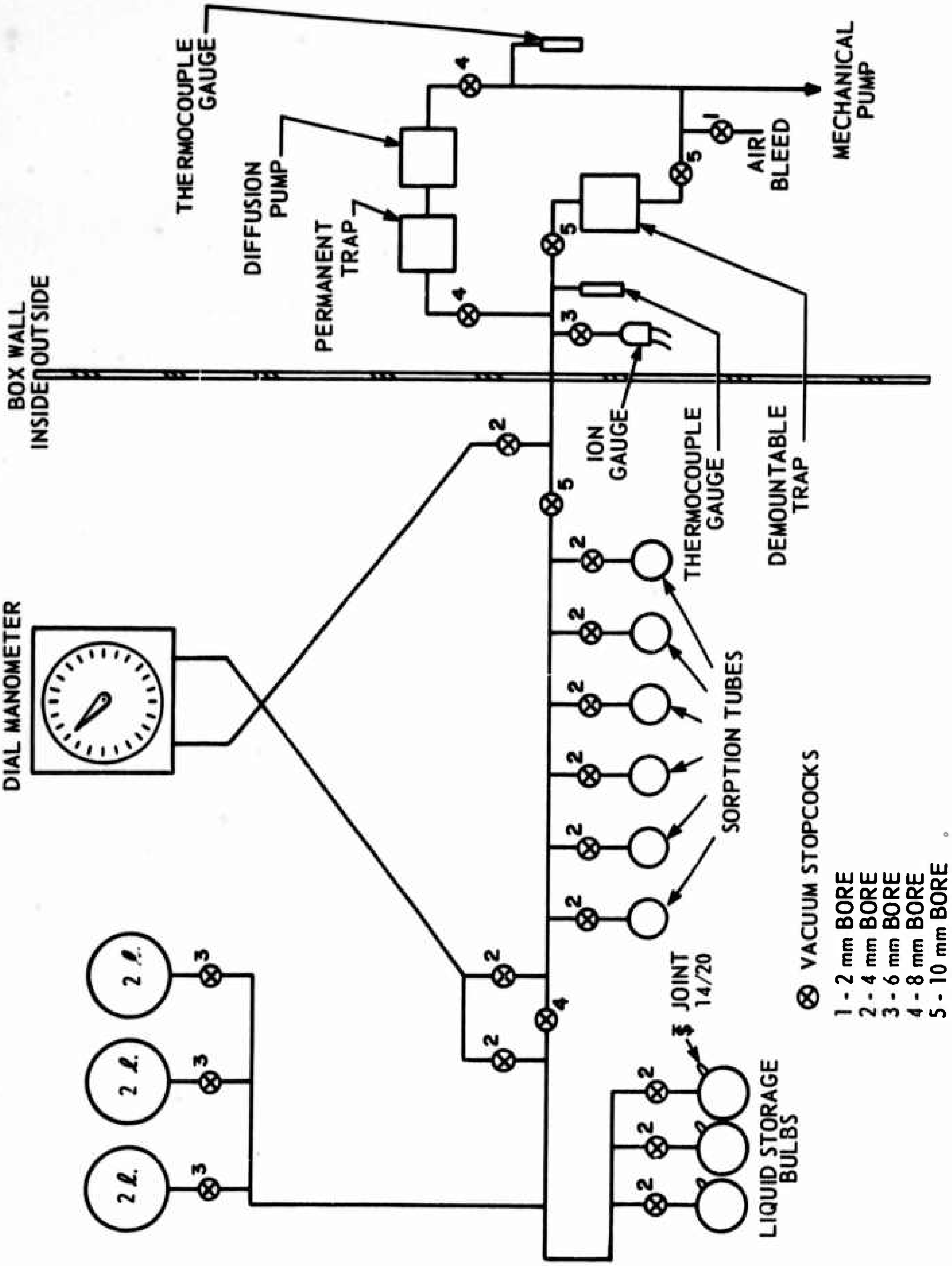

Figure 3. Schematic Diagram of Moisture Sorption Apparatus

30°C and 45°C, although at the lower temperature one 100-watt light bulb (intermittent) was sufficient to maintain fine control.

A Welch, two-stage, Duo-seal, oil pump was used to provide a rough vacuum. A Consolidated Vacuum Corporation one stage, oil diffusion pump was used to obtain the high vacuum (1×10^{-6} torr). Two liquid nitrogen cold traps were used to prevent water vapor fouling the pumps. The pressure in the system was measured with a Wallace and Tiernan differential manometer, while the system vacuum was measured with a CVC ion gauge and thermocouple gauges.

The spring balances were constructed from 5-mil diameter Ni-Span-C wire. The springs were made by winding the wire on a stainless steel mandrel (12 threads per inch, 0.5 inches inner thread diameter) and annealing the wire, *in vacuo*, at 600°C for six hours. Ni-Span-C wire was chosen because of its low spring-constant temperature coefficient and because water adsorption on this wire is extremely low and does not affect the spring constant up to a relative pressure of 0.95. The springs used in these sorption measurements had about 28 turns and had spring constants in the range 1.5 to 1.8 mg/mm with a standard deviation of ± 0.01 mg/mm. A cathetometer was used to tollow the spring expansion. This cathetometer could be read to ± 0.05 mm.

2.1.2 Isotherm Measurements

The moisture sorption isotherms were measured by the static equilibrium method. Food samples (about 0.2g), ground to less than 40 mesh size, were placed in quartz buckets which were suspended on the springs in the sorption tubes. The entire apparatus was evacuated to a vacuum of about 1×10^{-5} torr. Usually the food samples took about 30 hours before they ceased evolving moisture. When cathetometer readings of the spring movement showed no contractions for six straight hourly readings, complete dehydration of the foods was assumed. The sorption tubes were closed and water vapor was let into the system to the desired pressure and then 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 six hourly cathetometer readings were the same. In the measurement of the isotherms nine pressure points were taken. An effort was made to spread these points uniformly over the vapor pressure range at the temperature used. Moisture sorption isotherms were measured at 30°C, 35°C, 40°C, and 45°C with the 37.77°C (100°F) isotherm determined by interpolation. Equilibrium of the foods with moisture usually took about 12 to 24 hours; and a complete isotherm at any temperature took about two weeks, including the desorption measurements. For each sample, four or five desorption points were measured, including complete desorption. For desorption, the sample tubes were closed while water vapor was pumped out of the system and then the tubes were opened and the equilibrium point determined. During complete desorption the sample tubes were open during evacuation.

2.1.3 Materials Used

The water used in these experiments was triply distilled water with a conductivity of 3.0×10^6 $ohms^{-1}$-cm^{-1}. It was completely degassed before use.

The foods used in these isotherm measurements are described completely in Section 3. These foods were ground to less than 40 mesh for the isotherm measurements. Three samples of each food were used at each temperature with fresh samples of food being used at each temperature. The sample weights were approximately 0.2 grams. This weight was dictated by the volume of the quartz sample buckets and the length of the sorption tubes (approximately 60 cm). At high relative pressure the extended springs occupied the whole length of the tubes.

2.2 Experimental Results

2.2.1 Moisture Sorption Isotherms

In this experimental study, moisture sorption and desorption isotherms were measured for various dried food powders over the temperature range of 30° to 45°C. The dried food powders used were: banana, egg yolk, beef, shrimp, apple, cooked white potato, cooked sweet potato, rice and milk. All the food powders passed through a 40 mesh screen. These foods were examined in pairs and were not all examined at the same temperatures. The pairs were: bananas and egg yolk, beef and shrimp, apple and cooked white potato, rice and milk, and cooked sweet potato singly. The first two pairs were studied at 35°, 40°, and 45°C and the remainder at 30°, 35°, and 40°C.

The equilibrium sorption values for these foods are given in Tables 1 through 9 and the equilibrium desorption values are given in Tables10 through 18. For all foods the equilibrium sorption values at 37.77°C (100°F) were obtained by interpolation. The sorption values shown were all taken from sorption plots and were taken at standardized relative pressures to make comparisons easier. The desorption points, however, are the actual experimental values except for milk at 30°C. A detailed desorption measurement was made for milk at 30°C for comparison with the stepwise sorption isotherm.

TABLE 1

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR DRIED BANANA POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	45°C	40°C	37.77°C	35°C
0.05	6.0	5.0	4.8	4.5
0.1	11.7	11.5	10.5	9.3
0.2	28.1	27.0	24.8	22.6
0.3	49.3	45.6	42.8	39.9
0.4	75.3	72.1	69.0	65.9
0.5	105.6	109.3	105.0	100.6
0.6	146.9	155.9	152.6	149.2
0.7	212.1	221.6	220.8	220.9
0.8	314.0	323.0	334.4	345.8
0.85	396.2	418.4	440.6	462.7
Po (mm)	71.9	55.3	49.0	42.1

TABLE 2

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES FOR DRIED EGG YOLK POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	45°C	40°C	37.77°C	35°C
0.05	11.5	10.8	10.4	9.5
0.1	19.6	18.2	17.2	15.9
0.2	30.3	26.8	26.0	25.0
0.3	38.0	33.8	33.3	32.3
0.4	46.4	41.1	39.7	39.3
0.5	56.8	51.1	49.7	48.1
0.6	69.5	63.9	62.4	60.4
0.7	86.1	80.6	79.0	77.0
0.8	110.9	106.1	105.3	104.6
0.85	130.9	126.0	125.1	123.9
Po (mm)	71.9	55.3	49.0	42.1

TABLE 3

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR OVEN-DRIED BEEF POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	45°C	40°C	37.77°C	35°C
0.05	14.5	15.0	12.4	9.1
0.1	22.8	22.3	20.0	17.1
0.2	32.9	33.1	31.3	29.0
0.3	42.3	41.2	40.2	39.0
0.4	54.1	50.3	50.3	50.2
0.5	68.0	62.1	63.1	64.4
0.6	85.1	79.4	81.7	84.7
0.7	112.5	112.2	114.7	117.9
0.8	163.7	166.6	167.2	168.0
0.85	197.0	208.5	210.5	213.0
Po (mm)	71.9	55.3	49.0	42.1

TABLE 4

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR FREEZE-DRIED SHRIMP POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	45°C	40°C	37.77°C	35°C
0.05	21.0	23.0	20.8	18.0
0.1	32.0	32.9	31.2	29.1
0.2	46.9	47.1	46.5	45.8
0.3	62.3	58.2	60.2	62.7
0.4	78.4	70.8	75.0	80.4
0.5	98.3	88.3	93.7	100.6
0.6	122.7	116.2	120.7	126.4
0.7	158.8	156.5	162.2	169.4
0.8	220.0	223.0	227.2	232.5
0.85	256.2	282.0	287.7	294.0
Po (mm)	71.9	55.3	49.0	42.1

TABLE 5

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR FREEZE-DRIED APPLE POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	40°C	37.77°C	35°C	30°C
0.05	8.0	8.4	8.8	9.2
0.1	17.1	17.7	18.5	19.4
0.2	37.5	38.8	40.4	42.4
0.3	60.0	62.7	66.2	68.0
0.4	88.2	92.0	96.9	99.0
0.5	128.0	131.1	135.0	140.8
0.6	180.7	186.8	193.3	208.0
0.7	260.5	273.7	290.5	301.8
0.8	404.0	435.6	476.0	437.0
0.85	525.0	566.0	595.5	606.0
Po (mm)	55.3	49.0	42.1	31.8

TABLE 6

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES FOR FREEZE-DRIED COOKED WHITE POTATO POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	40°C	37.77°C	35°C	30°C
0.05	26.3	27.0	28.0	27.0
0.1	38.5	39.7	41.3	43.1
0.2	58.8	59.5	60.4	63.5
0.3	75.9	76.1	76.3	80.0
0.4	91.9	92.4	93.0	94.9
0.5	109.1	110.0	111.0	113.7
0.6	128.5	131.7	133.4	138.2
0.7	154.7	158.7	163.7	167.0
0.8	195.3	202.0	210.6	210.5
0.85	237.0	248.1	262.0	265.0
Po (mm)	55.3	49.0	42.1	31.8

TABLE 7

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR FREEZE-DRIED COOKED SWEET POTATO POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	40°C	37.77°C	35°C	30°C
0.05	14.8	15.7	16.8	14.4
0.1	24.2	26.3	29.0	25.8
0.2	38.3	41.2	44.8	42.0
0.3	50.0	53.0	56.8	52.0
0.4	64.1	68.2	73.4	67.0
0.5	94.0	96.6	99.9	88.8
0.6	133.5	135.3	137.6	122.4
0.7	187.0	191.0	196.0	173.4
0.8	286.0	288.0	292.2	263.6
0.85	366.4	372.5	380.0	340.0
Po (mm)	55.3	49.0	42.1	31.8

TABLE 8

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR DRIED RICE POWDER

Relative Pressure	Equilibirum Weight (mg/g)			
P/Po	40°C	37.77°C	35°C	30°C
0.05	17.8	19.5	21.5	16.4
0.1	31.5	34.4	38.0	30.2
0.2	49.5	55.1	62.1	54.1
0.3	64.6	70.4	77.5	73.0
0.4	77.0	81.7	87.6	86.0
0.5	90.0	94.5	100.0	97.7
0.6	105.0	109.5	115.0	112.3
0.7	124.1	129.4	136.0	131.5
0.8	157.8	163.5	170.5	161.9
0.85	185.5	189.3	194.0	184.5
Po (mm)	55.3	49.0	42.1	31.8

TABLE 9

AVERAGE EQUILIBRIUM MOISTURE SORPTION VALUES
FOR FREEZE-DRIED MILK POWDER

Relative Pressure	Equilibrium Weight (mg/g)			
P/Po	40°C	37.77°C	35°C	30°C
0.05	15.7	18.5	20.7	14.5
0.1	23.0	25.1	27.8	22.0
0.2	33.2	34.9	37.1	30.7
0.3	49.0	51.2	53.9	42.7
0.4	57.6	65.1	74.6	66.2
0.5	67.5	75.4	85.5	76.9
0.6	36.2	90.5	96.0	89.3
0.7	115.3	120.5	127.0	119.0
0.8	179.0	177.2	175.0	158.5
0.85	234.0	231.8	229.0	197.5
Po (mm)	55.3	49.0	42.1	31.8

TABLE 10

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR DRIED BANANA POWDER

45°C Isotherm		40°C Isotherm		35°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.88	440.0	0.68	205.0	0.77	292.5
0.54	137.0	0.29	48.9	0.46	90.5
0.11	27.5	0.07	27.3	0.13	24.9
0.0	5.0	0.0	4.0	0.0	4.0

TABLE 11

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR DRIED EGG YOLK POWDER

45°C Isotherm		40°C Isotherm		35°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.88	146.0	0.68	78.1	0.77	94.5
0.54	75.0	0.29	36.5	0.46	45.3
0.11	37.9	0.07	25.4	0.13	20.1
0.0	6.0	0.0	4.0	0.0	0.0

TABLE 12

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR OVEN-DRIED BEEF POWDER

45°C Isotherm		40°C Isotherm		35°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.49	67.6	0.71	123.1	0.78	168.8
0.32	47.1	0.56	82.9	0.44	64.5
0.14	31.2	0.31	48.2	0.23	46.7
0.08	24.8	0.13	30.3	0.0	2.4
0.0	1.9	0.0	0.0		

TABLE 13

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR FREEZE-DRIED SHRIMP POWDER

45°C Isotherm		40°C Isotherm		35°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.49	94.0	0.71	169.9	0.78	227.1
0.32	67.6	0.56	117.1	0.44	93.7
0.14	45.1	0.31	70.9	0.23	67.4
0.08	36.9	0.13	45.0	0.0	00.7
0.0	0.8	0.0	0.0		

TABLE 14

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR FREEZE-DRIED COOKED SWEET POTATO POWDER

40°C Isotherm		35°C Isotherm		30°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.68	173.1	0.78	275.0	0.69	169.5
0.38	61.6	0.59	132.5	0.59	110.0
0.10	25.9	0.30	76.8	0.38	62.1
0.0	6.2	0.10	31.3	0.15	36.3
		0.0	6.0	0.0	6.5

TABLE 15

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR DRIED RICE POWDER

40°C Isotherm		35°C Isotherm		30°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.65	137.5	0.78	179.3	0.74	157.2
0.43	104.5	0.59	147.0	0.59	127.7
0.27	76.4	0.30	91.7	0.37	86.4
0.11	45.3	0.10	49.1	0.0	5.3
0.0	0.8	0.0	7.1		

TABLE 16

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR FREEZE-DRIED APPLE POWDER

40°C Isotherm		35°C Isotherm		30°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.75	326.4	0.73	327.7	0.75	360.6
0.63	203.1	0.37	88.1	0.57	183.6
0.33	71.4	0.13	30.4	0.27	59.9
0.19	35.7	0.0	0.6	0.10	21.0
0.0	0.0			0.0	0.9

TABLE 17

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES
FOR FREEZE-DRIED COOKED WHITE POTATO POWDER

40°C Isotherm		35°C Isotherm		30°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.75	164.3	0.73	173.8	0.75	180.1
0.63	135.1	0.37	101.1	0.57	130.9
0.33	85.7	0.13	55.7	0.27	79.1
0.19	64.7	0.0	0.0	0.10	46.9
0.0	0.0			0.0	1.8

TABLE 18

AVERAGE EQUILIBRIUM MOISTURE DESORPTION VALUES FOR FREEZE-DRIED MILK POWDER

40°C Isotherm		35°C Isotherm		30°C Isotherm	
P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)	P/Po	Equilibrium Wt. (mg/g)
0.65	100.0	0.78	166.7	0.80	160.0
0.43	61.4	0.59	100.3	0.70	118.2
0.27	47.6	0.30	63.6	0.60	89.0
0.11	35.0	0.10	36.2	0.50	76.7
0.0	3.0	0.0	2.1	0.40	68.0
				0.30	58.9
				0.20	48.2
				0.10	34.6
				0.05	24.5
				0.0	4.1

Typical moisture sorption isotherms at 37.77°C are shown for all the foods in Figures 4 through 12. In addition, the moisture sorption isotherm for milk at 30°C is shown in Figure 13. This latter sorption isotherm plot was included because of the interesting stepwise sorption which is shown, there being three distinct sorption steps. At 30 to 40°C, only two sorption steps appear for milk. The sorption isotherm of moisture onto milk is a unique type in this study. It is essentially a type II isotherm which is indicative of multilayer sorption. The various sorption steps were probably due to the formation of the second and third monolayers. The isotherms for the other foods can be divided into type II and type III isotherms. Bananas and apples gave type III isotherms while the others gave type II isotherms. Both types of isotherms are indicative of multilayer sorption.

For all foods, with the possible exception of sweet potatoes, the desorption of moisture exhibited a hysteresis loop with the maximum hysteresis occurring between the first and second monolayers. In most cases a small amount of water was irreversibly sorbed. The desorption of moisture from sweet potato powder exhibited hysteresis only at nearly complete desorption, there being some irreversible sorption. The desorption of moisture from milk powder was not stepwise and hysteresis only occurred between the first and second monolayer. At the second and third steps the desorption curve was actually below the sorption curve.

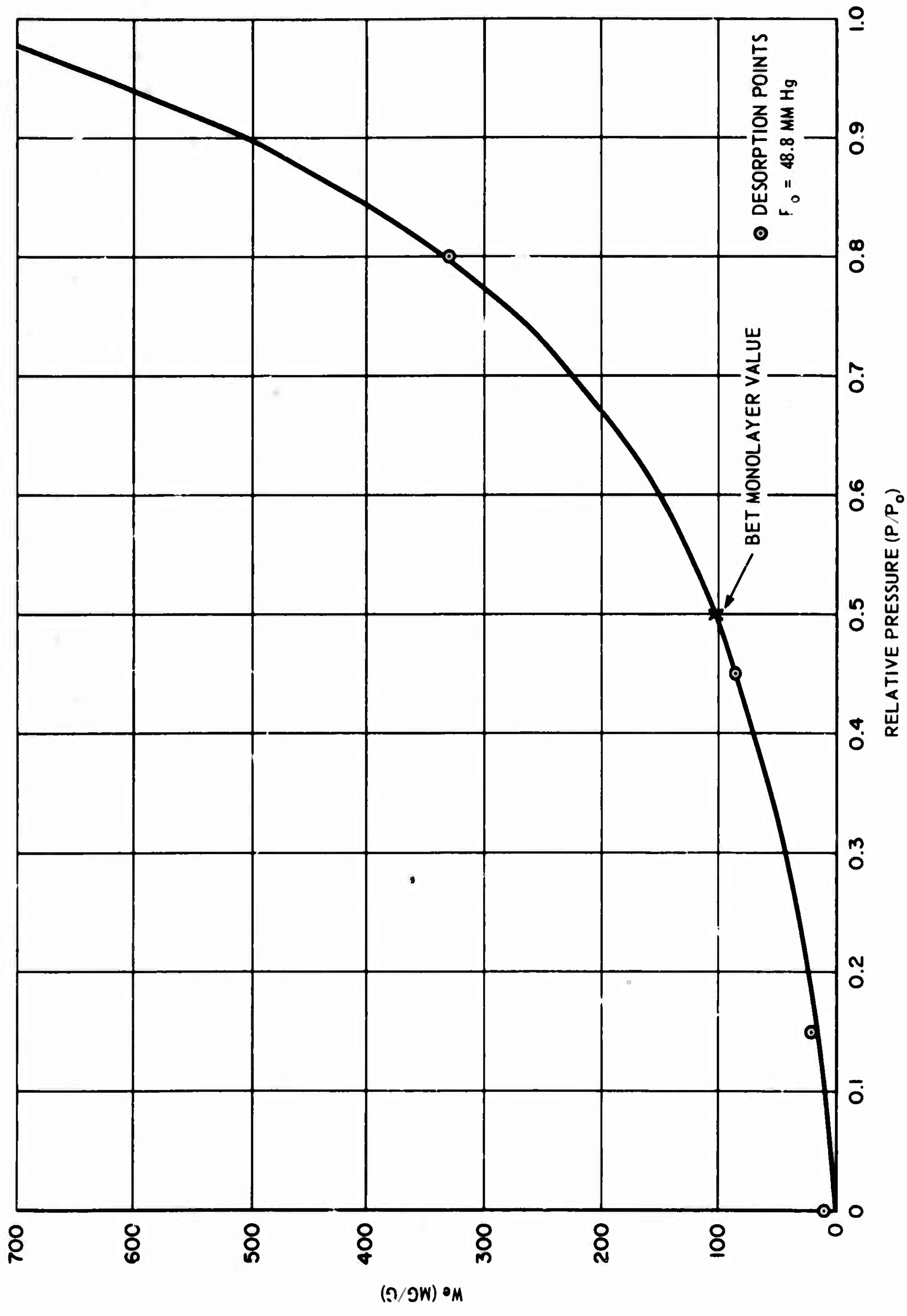

Figure 4. Water Sorption Isotherm for Dried Banana Powder at 37.77°C

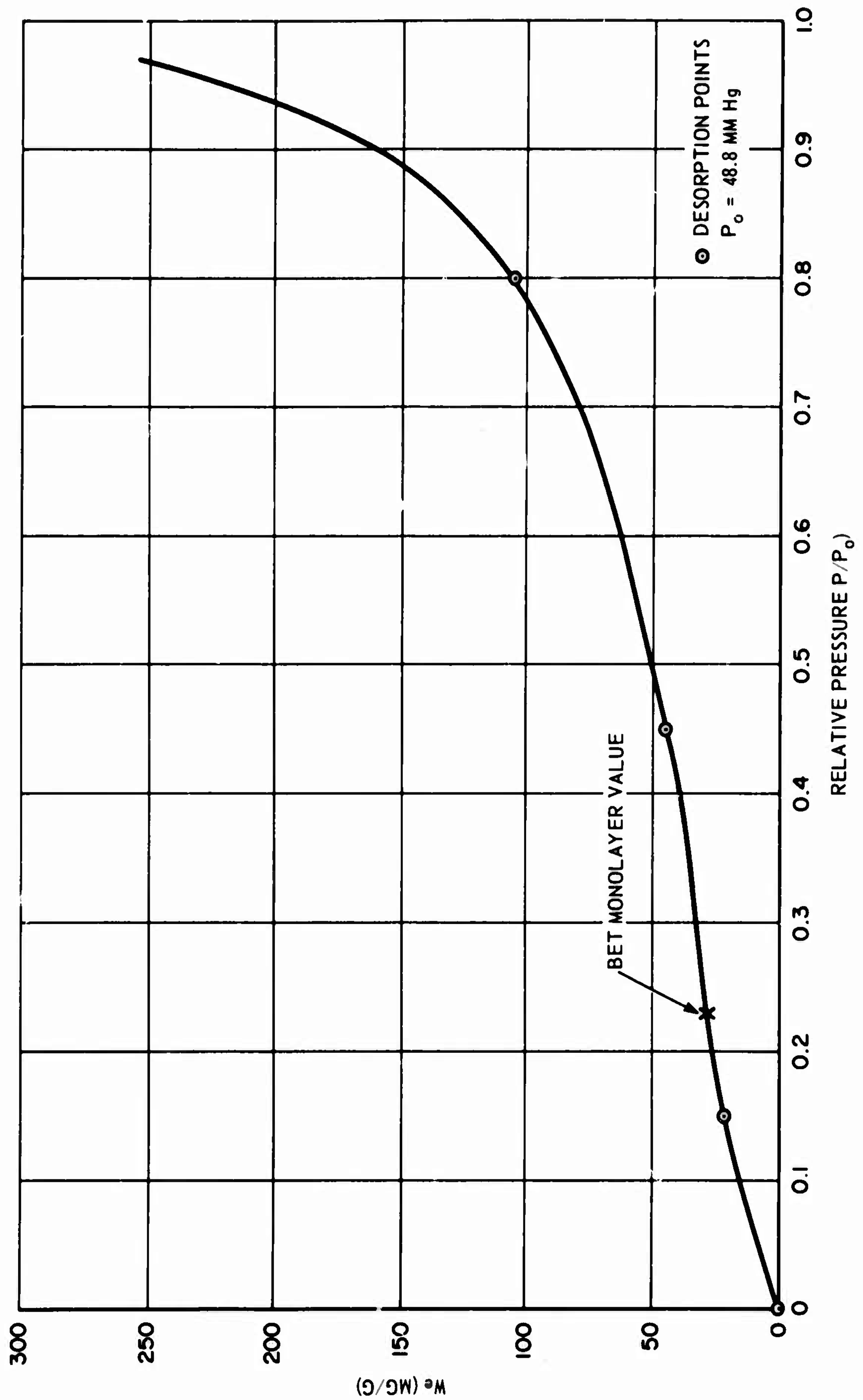

Figure 5. Water Sorption Isotherm for Dried Egg Yolk Powder at 37.77°C

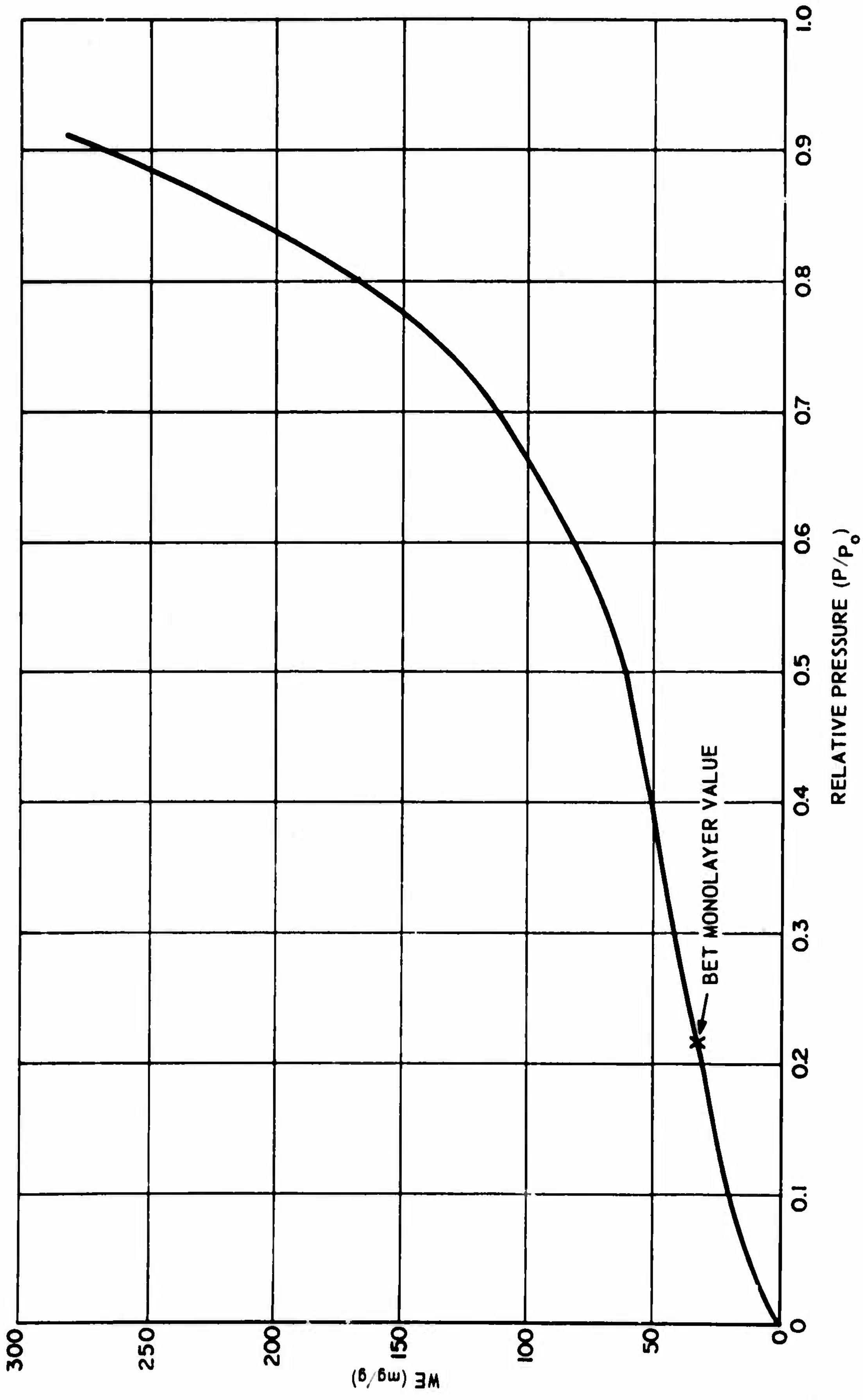

Figure 6. Water Sorption Isotherm for Oven-Dried Beef Powder at 37.77°C

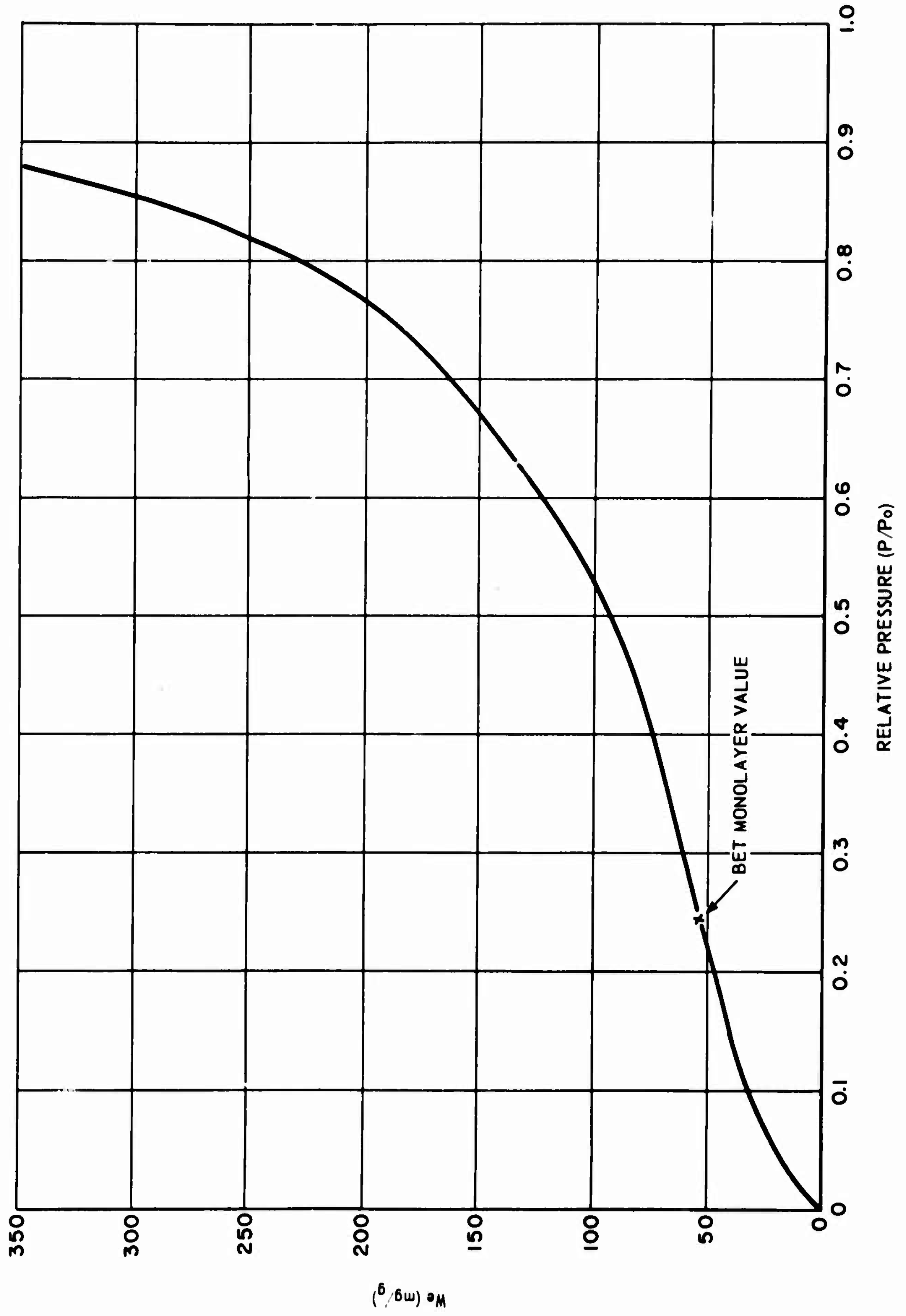

Figure 7. Water Sorption Isotherm for Freeze-Dried Shrimp Powder at 37.77°C

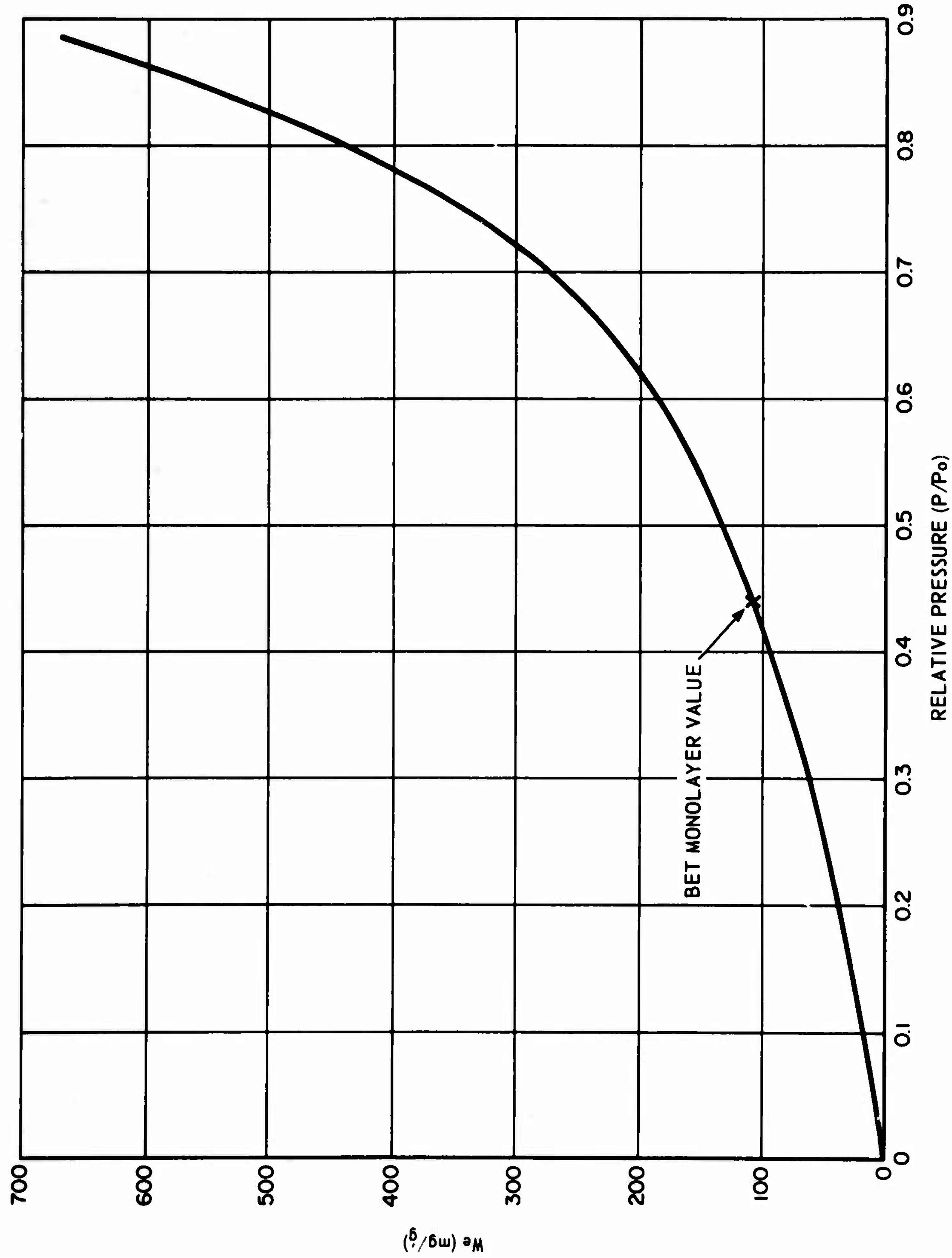

Figure 8. Water Sorption Isotherm for Freeze-Dried Apple Powder at 37.77°C

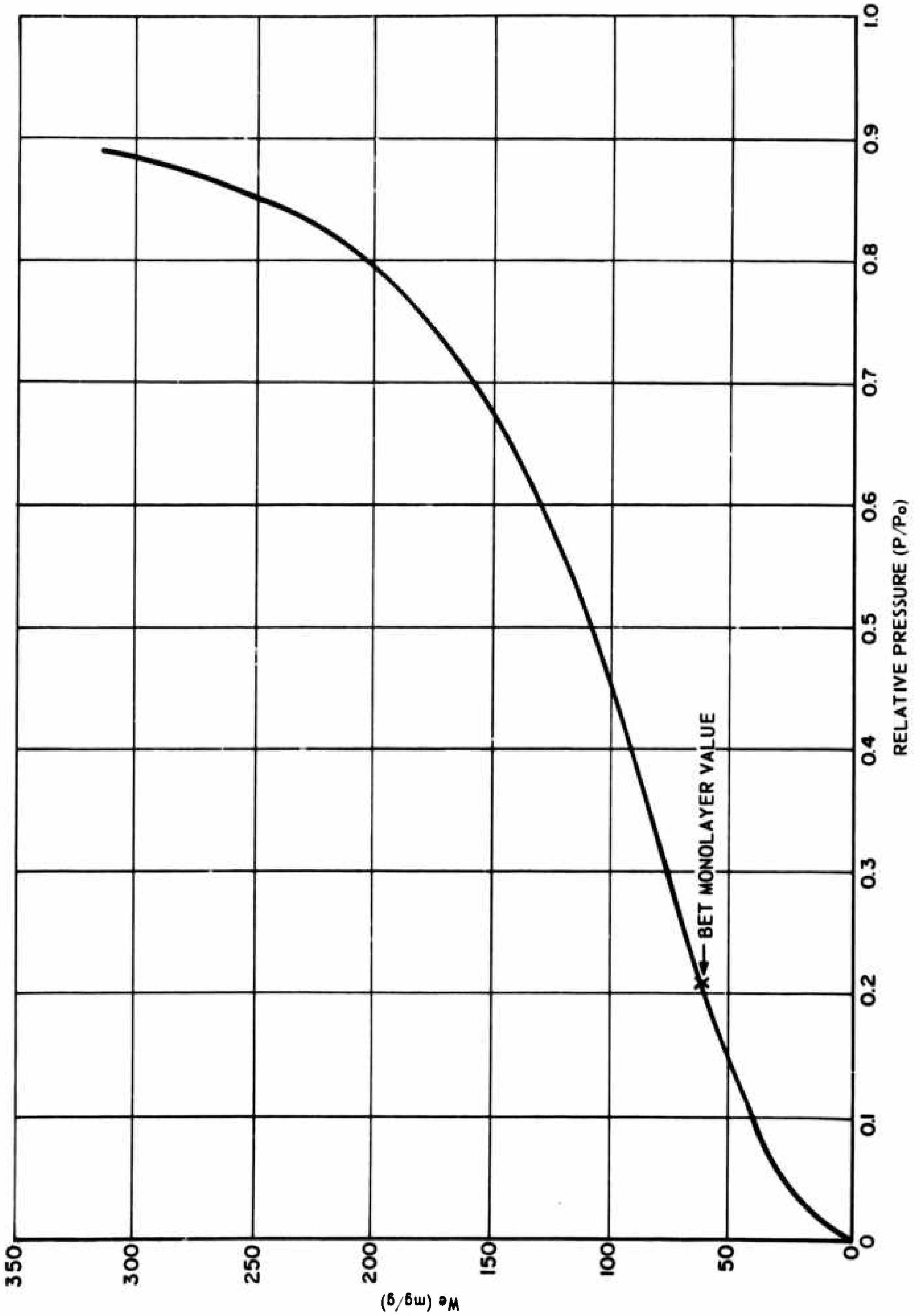

Figure 9. Water Sorption Isotherm for Freeze-Dried Cooked Potato Powder at 37.77°C

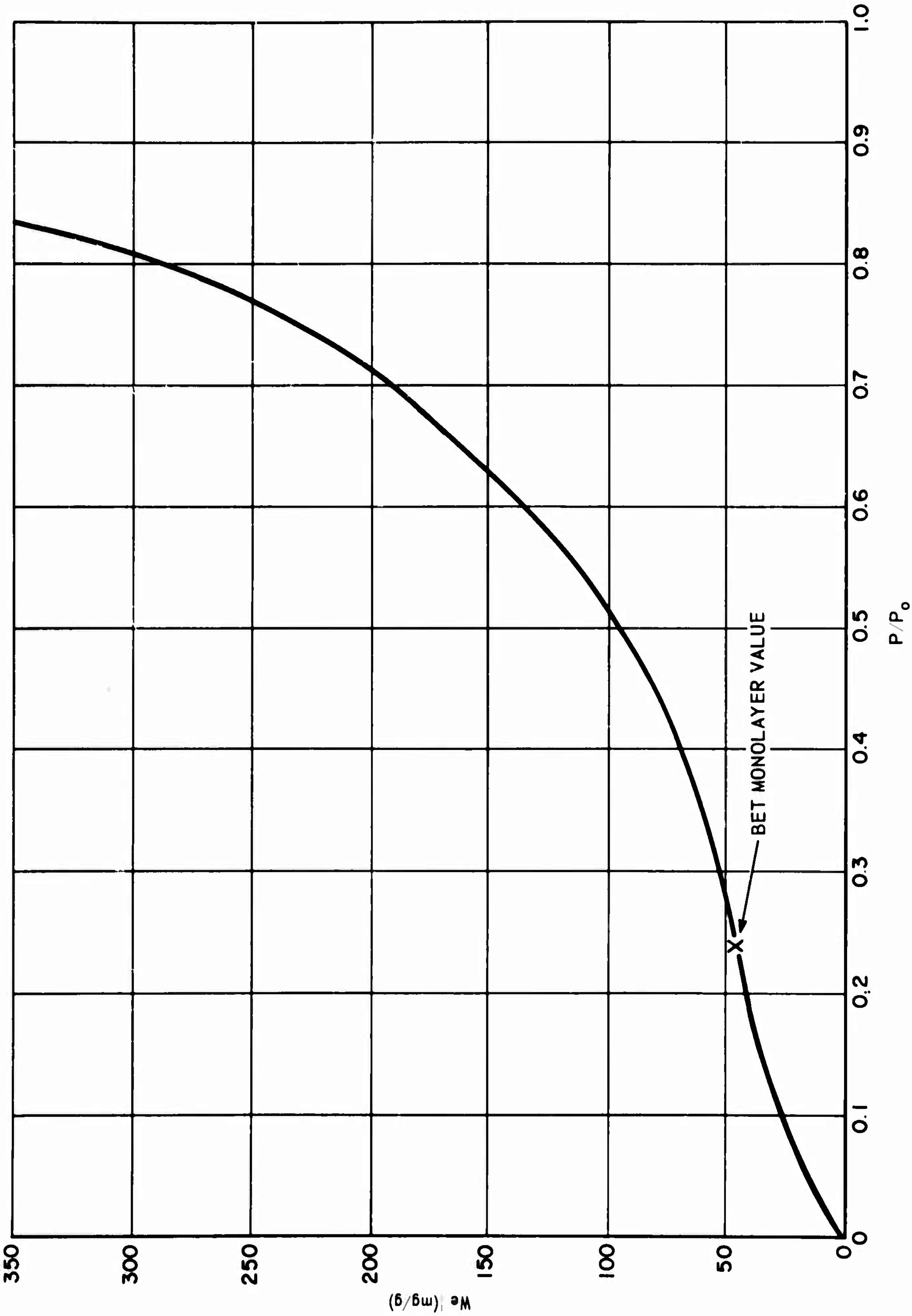

Figure 10. Water Sorption Isotherm for Freeze-Dried Cooked Sweet Potato Powder at 37.77°C

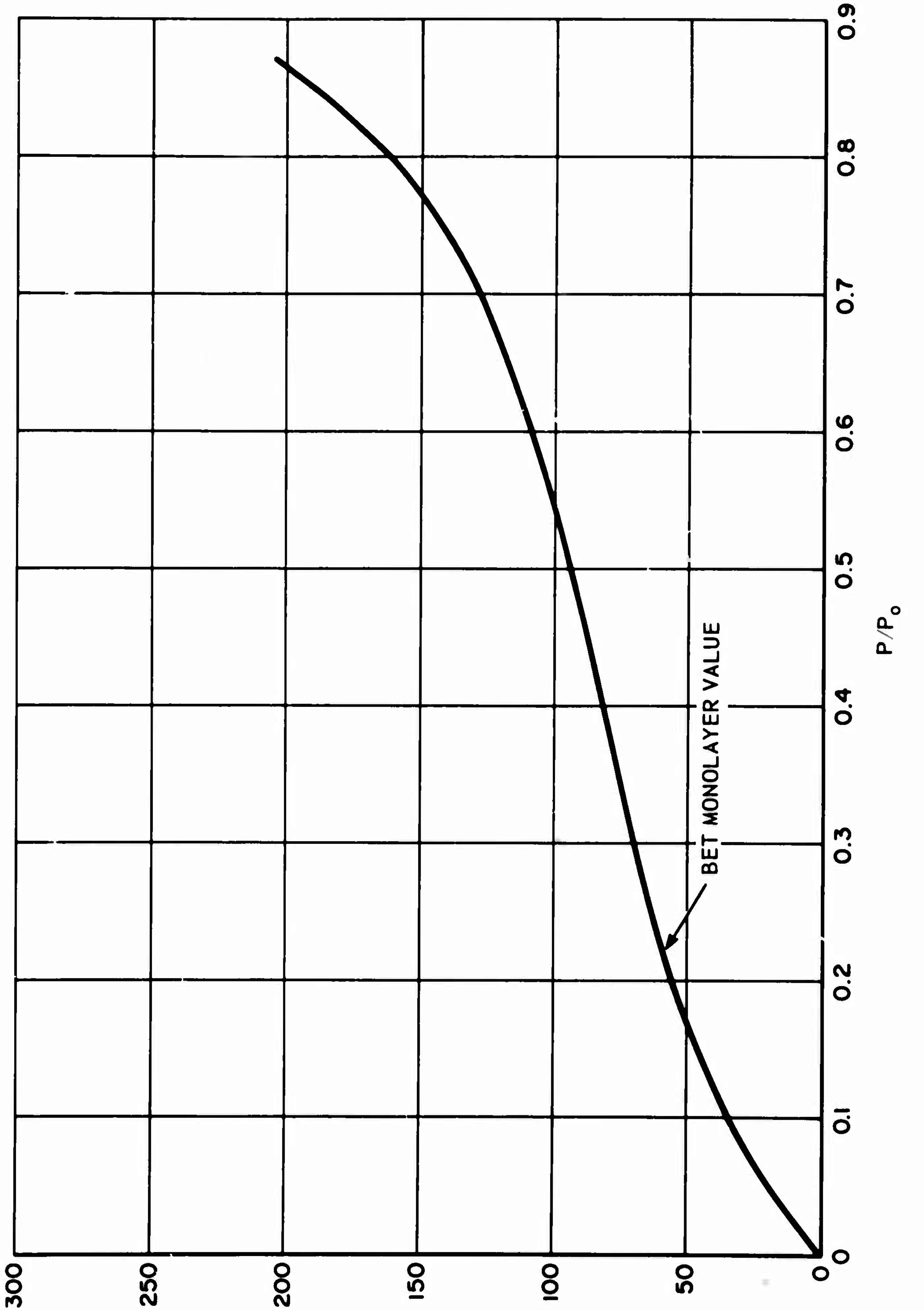

Figure 11. Water Moisture Sorption Isotherm for Rice Powder at 37.77°C

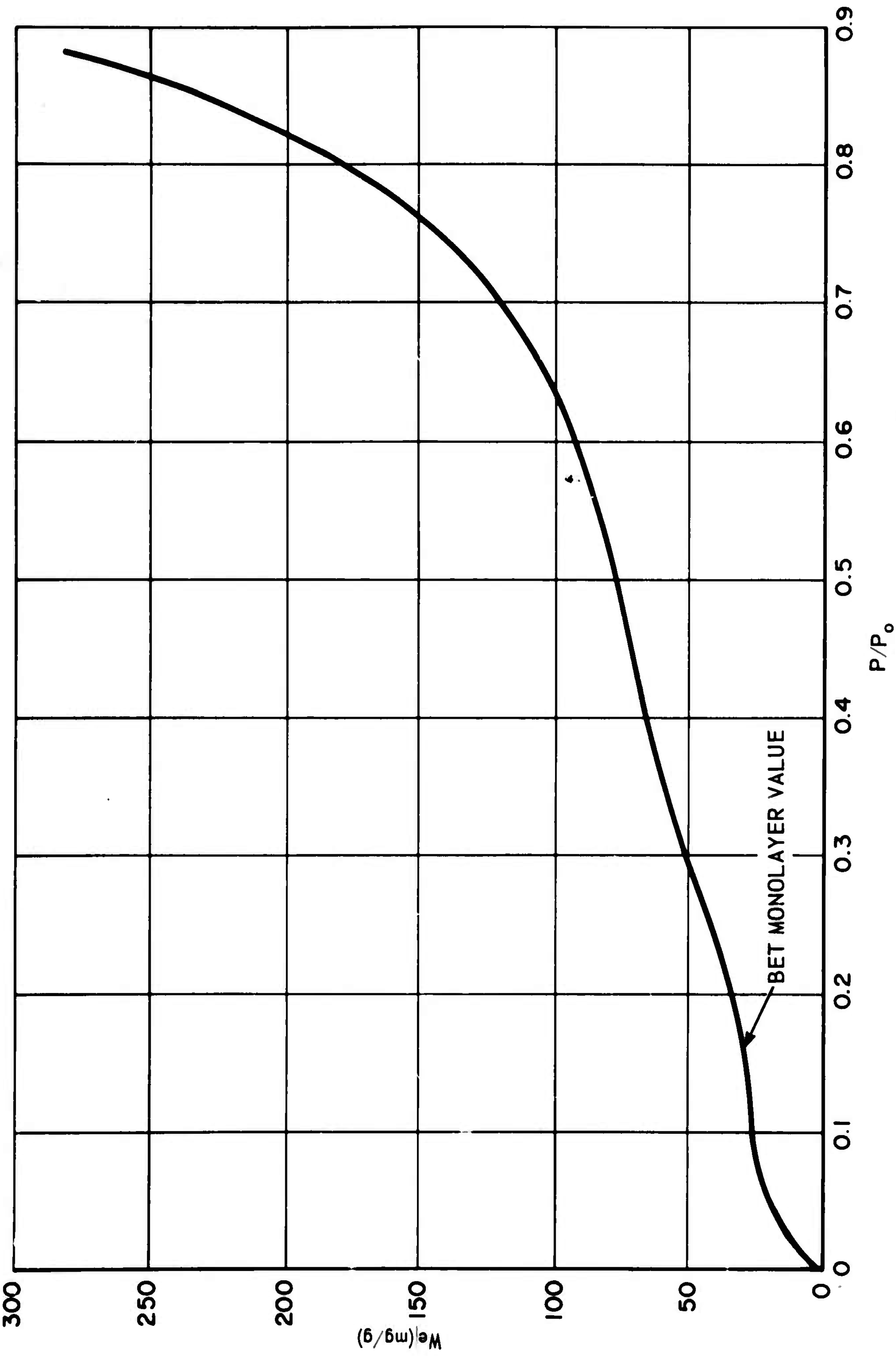

Figure 12. Water Moisture Sorption Isotherm for Freeze-Dried Milk Powder at 37.77°C

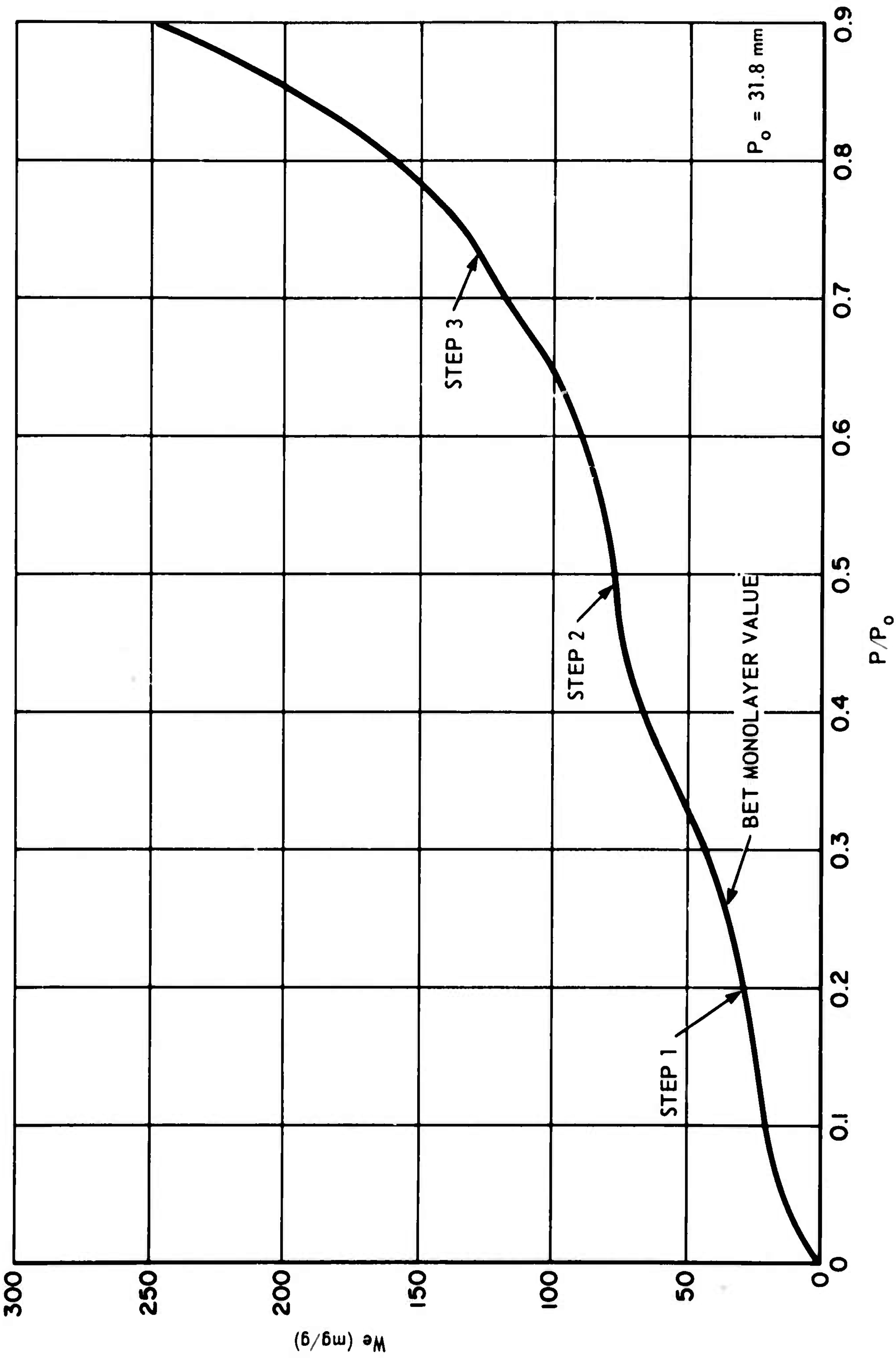

Figure 13. Water Moisture Sorption Isotherm for Freeze-Dried Milk Powder at 30°C

2.2.2 BET[2] Monolayer Values

The equilibrium sorption values given in Tables 1 through 9 were used to calculate BET monolayer values for the foods. The BET isotherm equation was used:

$$We = \frac{WmCP}{(P_o - P)\left[1 + (C-1)(P/P_o)\right]} \qquad (1)$$

where We = equilibrium sorption weight

Wm = weight sorbed at monolayer coverage

P = equilibrium vapor pressure

P_o = saturation pressure of vapor

$$C = k \exp. (E_A - E_L / RT) \qquad (2)$$

In this latter equation, k is a constant approximately equal to one, E_A is the heat of sorption and E_L is the heat of liquifaction of the vapor (10.4 k cal/mole at 25°C).

Although a monolayer value can be obtained from the BET equation, this equation has severe limitations; it is usually valid between values of 0.1 to 0.5 relative pressure. In addition, in the case of a swelling gel, the number of interior sorption sites may be so large as to make the number of exterior sites negligible.

The BET monolayer values were calculated by plotting $P/We\,(P_o - P)$ vs. P/P_o. This gave a straight line whose slope was equal to $(C-1)/WmC$ and intercept equal to $1/WmC$. The values of Wm calculated are shown in Table 19. They were calculated assuming a cross-sectional area of the water molecule of $10.8 A^2$[3]. Relative pressures at the monolayers are also shown in Table 19.

Table 19
Equilibrium Moisture Scrption Values at the
BET Monolayer for Dried Food Powder

Food	45°C Isotherm			40°C Isotherm			27.77°C Isotherm			35°C Isotherm			30°C Isotherm		
	Wt. of Water mg/g	Water Surface Area m2/g	P/Po	Wt. of Water mg/g	Water Surface Area m2/g	P/Po	Wt.of Water mg/g	Water Surface Area m2/g	P/Po	Wt.of Water mg/g	Water Surface Area m2/g	P/Po	Wt.of Water mg/g	Water Surface Area m2/g	P/Po
Banana	105.3	380.5	0.50	105.3	380.5	0.50	105.3	380.5	0.50	105.3	380.5	0.50	--	--	--
Egg Yolk	29.9	108.1	0.20	27.0	97.6	0.20	27.0	97.6	0.22	27.0	97.6	0.23	--	--	--
Beef	36.7	132.6	0.24	34.3	124.0	0.21	37.0	133.7	0.22	37.0	133.7	0.27	--	--	--
Shrimp	53.8	194.4	0.25	48.3	174.5	0.22	51.0	184.3	0.25	57.4	207.4	0.28	--	--	--
Apple	--	--	--	108.7	392.8	0.46	109.0	393.9	0.46	110.0	397.5	0.44	132.1	477.4	0.49
White Potato	--	--	--	61.0	220.4	0.21	61.0	220.4	0.21	61.0	220.4	0.21	67.4	243.6	0.22
Sweet Potato	--	--	--	45.5	164.4	0.26	46.0	166.2	0.24	50.4	182.1	0.25	48.7	176.0	0.27
Rice	--	--	--	53.6	193.7	0.23	61.4	221.9	0.23	71.2	257.3	0.32	75.2	271.8	0.26
Milk	--	--	--	33.1	119.6	0.20	33.3	120.3	0.18	33.7	121.8	0.16	36.6	132.3	0.26

The BET monolayer values at 37.77°C were used as the basis for determining storage conditions for the foods. Foods were stored at 37.77°C at two relative pressures below the monolayer value, a relative pressure equal to the monolayer value, and two relative pressures above the monolayer value. The food storage conditions are detailed more fully in Section 2.

2.2.3 Fugassi Isotherm Values

The following equation was derived by Fugassi, *et al.*,[4,5,6,7,8] to cover the case of vapor sorption onto swelling gels.

$$We = \frac{AKK_1P_oC}{1 + (K_1P_o - 1)C\,(1-C) + KK_1P_oC} \qquad (3)$$

where We = weight of material sorbed at equilibrium

- A = total amount of moisture sorbed, one sorbate molecule per site
- 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
- P_o = saturation vapor pressure
- C = relative pressure = P/P_o

This equation was found to fit the experimental data over the entire relative pressure region. A computer program was set up to calculate values of A, K and K_1. Essentially, this program summed nine values of We to obtain three groups of summation values -- one group for low relative pressures, one group for medium relative pressures, and one group for high relative pressures. The computer then solved the three resulting equations simultaneously to obtain the values of A, K and K_1. These values, for the various food powders at the different temperatures, are shown in Tables 20, 21, and 22. A sample calculation of these values is given in Appendix A.

This method of calculating the Fugassi equation places a heavy emphasis on the low relative pressure values where sorption on active surface sites is predominant. Further, the Fugassi equation assumes one sorbate molecule per sorption site. If activated sorption occurs, or if a sorbate molecule occupies more than one sorption site, then negative values of A, K, and K_1 can be obtained. It can be seen in Tables 20, 21, and 22 that there are some negative values. These negative values are meaningless and cannot be interpreted quantitatively. It is possible to obtain the correct values by kinetic studies or by extrapolation of the sorption isotherm. These are difficult to do accurately and are not in the scope of this study so the negative values were let stand for the sake of completeness and to show that the Fugassi isotherm equation is not always

TABLE 20

Values of the Fugassi "A" Constant for the Various Foods at Different Temperatures

Food	Fugassi "A'" Values (mg/g)				
	45°C	40°C	37.77°C	35°C	30°C
Banana	738	740	760	781	----
Egg Yolk	256	288	315	321	----
Beef	-4797	-7306	4855	1687	----
Shrimp	813.6	3137	1406	1667	----
Apple	----	2260	4135	11147	3413
White Potato	----	387	414	480	468
Sweet Potato	----	-1912	-1969	-2090	-1850
Rice	----	280	340	415	249
Milk	----	595	664	749	297

TABLE 21

Values of the Fugassi "K" Constant for the Various Foods at Different Temperatures

Foods	Fugassi "K" Values				
	45°C	40°C	37.77°C	35°C	30°C
Banana	0.20	0.22	0.22	0.27	----
Egg Yolk	0.15	0.12	0.10	0.10	----
Beef	-0.01	-0.01	0.01	0.02	----
Shrimp	0.08	0.02	0.04	0.04	----
Apple	----	0.05	0.03	0.01	0.04
White Potato	----	0.22	0.20	0.17	0.18
Sweet Potato	----	-0.03	-0.03	-0.03	-0.03
Rice	----	0.27	0.24	0.20	0.39
Milk	----	0.07	0.07	0.07	0.24

Table 22

Values of the Fugassi "K_1" Constants for the Various

Foods at Different Temperatures

	Fugassi "K_1" Values (mm^{-1})				
Food	45°C	40°C	37.77°C	35°C	30°C
Banana	0.01	0.01	0.01	0.01	----
Egg Yolk	0.11	0.16	0.19	0.21	----
Beef	-0.20	0.27	0.19	0.14	----
Shrimp	0.10	0.24	0.18	0.16	----
Apple	----	0.03	0.03	0.04	0.05
White Potato	----	0.14	0.16	0.22	0.30
Sweet Potato	----	0.10	0.06	0.02	0.22
Rice	----	0.13	0.19	0.26	0.16
Milk	----	0.35	0.26	0.16	0.09

accurate. In general, it can be stated that as the sorption temperature decreases both A and K increase indicating that more sorption occures at lower sorption temperatures.

An interesting calculation can be made using the Fugassi A and K values. 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. Also it is explicit in the Fugassi equation that

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

From these expressions a value for (H_2O) exterior and (H_2O) interior can be calculated. It is noteworthy that when K is very much less than one, the value for (H_2O) exterior is approximately the same as the BET monolayer value. These values of (H_2O) exterior are reported in Table 23 as monolayer values calculated by the Fugassi equation. It can be seen that when K is not very much less than one, these monolayer values differ from the BET values.

2.2.4 Heats of Sorption

Clausius-Clapeyron plots were constructed from the equilibrium sorption values; and the heat of sorption of moisture on the different foods was calculated. These plots are isosteres where ln P is plotted vs. 1/T in degrees Kelvin. The slope of the line is equal to $-\Delta H/R$ where R is the gas law constant and ΔH is the heat of sorption. For each food the heat of sorption of water vapor was constant over the temperature range studied. These heats of sorption are reported in Table 24.

TABLE 23

Monolayer Sorption Values Calculated
From the Fugassi Equation

Food	45°C We(mg/g)	40°C We(mg/g)	37.77°C We(mg/g)	35°C We(mg/g)	30°C We(mg/g)
Banana	124.0	133.0	137.0	167.0	----
Egg Yolk	34.0	31.0	28.0	29.0	----
Beef	31.0	26.0	40.0	37.0	----
Shrimp	60.0	49.0	52.0	58.0	----
Apple	----	110.0	120.0	110.0	119.0
White Potato	----	70.0	69.0	71.0	71.0
Sweet Potato	----	55.0	56.0	55.0	59.0
Rice	----	60.0	66.0	69.0	70.0
Milk	----	37.0	43.0	51.0	56.0

Table 24

Table 24

Heats of Sorption of Water Vapor on Dried Food Powders

Bananas 35-45°C		Egg Yolks 35-45°C		Beef 35-45°C		Shrimp 35-45°C	
Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole	Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole	Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole	Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole
50	9.2	20	5.0	25	9.0	25	9.0
100	9.8	27	7.5	37	9.0	51	9.4
200	10.8	40	9.3	50	9.1	100	9.2
300	11.2	60	9.0	100	10.2	150	10.0
400	11.4	80	9.8	150	10.8	200	11.2
		100	10.8	200	11.0	250	11.8

Apples 30-40°C		White Potatoes 30-40°C		Sweet Potatoes 30-40°C		Rice 30-40°C	
Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole	Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole	Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole	Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole
50	11.8	50	12.3	25	10.0	50	12.4
109	11.3	61	12.5	50.4	11.1	71.2	12.6
200	11.2	100	11.1	100	9.5	100	11.9
300	10.4	150	10.3	150	9.7	150	10.5
400	10.3	200	10.5	200	9.5	200	10.5

Milk 30-40°C	
Amount Water Sorbed mg/g	$-\Delta H$ kcal/mole
25	7.0
33.7	7.5
50	9.1
100	10.7
150	10.1

Commonly, at low relative pressures the heat of sorption is larger than the heat of liquifaction of the gas by a few hundred to a few thousand calories per mole because of the heat of interaction of the sorbate molecules with active sites on the sorbent surface. As more sorbate molecules are sorbed the heat of interaction approaches the heat of liquifaction of the vapor. For multilayer sorption, the heat of sorption of all layers above the first monolayer should equal the heat of liquifaction of the vapor since the range of most sorbate-sorbate interactions is assumed not to extend past the first layer. For water vapor, the heat of liquifaction is 10.4 k cal/mole at 25°C.

However, it is possible to have heats of sorption for the first monolayer which are equal to or less than the heat of liquifaction of the vapor.[9] In the former case a type III isotherm will result. The BET constant C will equal one, and the resultant isotherm will be a simple exponential curve. Graham[9] states that in order to have a heat of sorption less than the heat of liquifaction, the sorbate molecules must be isolated from each other.

The foods in Table 24 can be divided into three groups; those whose heat of sorption increased with increased sorption, those with constant heat of sorption, and those with a decrease in heat of sorption. In the first group are milk and egg yolk unambiguously. Banana, beef, and shrimp could belong to the first or second group depending on the accuracy of the measurements. Apples, white potatoes, and rice belong to the third group. Sweet potatoes probably belong to the second group.

2.3 Discussion of Results

These moisture sorption studies are primarily measurements of thermodynamic and other physical data. By themselves they can give no indication of the changes which take place in foods during storage. The results obtained in the moisture sorption studies must be correlated with chemical evaluation of the foods. However, the moisture sorption results are of basic interest by themselves.

2.3.1 Moisture Sorption Isotherms

Two types of moisture sorption isotherms were observed in this study; type II and type III. Apple powder and banana powder gave a type III isotherm, while the other foods gave a type II isotherm. Both types of isotherms are indicative of multilayer sorption. The moisture sorption isotherm of milk powder was unusual in that two or three distinct sorption steps appeared. These sorption steps were probably due to the formation of succeeding monolayers.

In general, the amount of moisture sorbed increased as the sorption temperature decreased, but there were some interesting variations of this rule. For some foods, at low relative pressures, the amount of sorption decreased with a decrease in sorption temperature, but increased with decreasing temperatures at high relative pressures. These foods were bananas, beef, and shrimp. Other foods, sweet potato, rice, and milk, appeared to increase to a maximum sorption and then decrease in sorption as temperature decreased. Egg yolk powder showed a continual decrease in sorption as the sorption temperature decreased. These anomalies are not easily explained. They may be due to the fact that all the foods had many more interior sorption sites than exterior sorption sites and at the lower temperatures and lower relative pressures, diffusion to these interior sites occurs slowly. Indeed for rice and milk, the Fugassi "K" constant increased sharply indicating a major decrease in the number of available interior sites.

All of the foods, with the exception of sweet potatoes, exhibited hysteresis loops. These hysteresis loops were probably due to capillary condensation. During the desorption phase, the equilibrium pressure for removal of capillary condensate is reduced according to the concept of the Kelvin equation.[10] Hence, the equilibrium weight for a given relative pressure is greater during the desorption phase than during the sorption phase, and the cycle exhibits a hysteresis loop. This should be mainly a physical phenomenon and not related to the chemical structure of the foods. Therefore, it is interesting that sweet potatoes did not show a hysteresis loop. All of the foods showed a slight amount of irreversible sorption indicating that some chemical reaction could have occurred. For this reason different samples of food were used for each isotherm.

2.3.2 BET Monolayer Values

The BET monolayer values varied considerably from food to food, but for each food they were relatively constant over the temperature range studied. The relative pressures at the

BET monolayers varied according to the type of isotherm. For the type III isotherm the relative pressure was about 0.5, while for the type II isotherms the relative pressure was about 0.2. The BET water surface areas for these foods were all large, especially in comparison to BET liquid nitrogen surface areas. Berlin and Pallansch[11] reported the BET liquid nitrogen surface areas of crushed turkey, shrimp, and spray-dried milk to be about 0.3 m^2/g, and for freeze-dried diced carrots to be 0.5 m^2g. If these values can be taken as typical values, then the water surface areas seem to be quite large. However, the diameter of the water molecule (10.8A) is about 50% that of the nitrogen molecule (15.8A)[3] so that the water molecule can enter pores and crevices which are not available to the nitrogen molecule. In addition, water is a polar molecule so that activated sorptions can occur, especially at the temperatures of this study. The variation in amount of moisture sorption at the monolayer with the food sample is a measure of the attraction of a particular food for water. A better comparison of attraction can be made at saturation pressure by comparing the Fugassi "A" values.

2.3.3 Fugassi Isotherm Equation Values

The Fugassi isotherm equation was derived to analyze the sorption of vapors onto swelling gels. The Fugassi equation is derived by equilibrating the rates of sorption and desorption on the surface and also by equilibrating the rates of migration of sorbed molecules from surface sites to interior sites and back again. As for the BET equation, no specific sorption sites are assumed, and the energy of sorption of the second and higher monolayers is assumed to be equal to the heat of liquifaction. Two special assumptions for the Fugassi equation are that the number of surface sites is small compared to the number of interior sites and that one molecule occupies one site. In the derivation of this equation K is defined as

$$K = \phi A/(1-\phi)A\sum_{n=1}^{\infty} n\theta_n \tag{6}$$

in which ϕ is the fraction of internal reaction, A is the total number of moles of reaction site per gram of sorbent, and $\sum n\theta_n$ is the average concentration of sorbate on the surface. In order to effect a solution of the equation, it is specified that

$$\sum_{n=1}^{\infty} n\theta_n = \theta_1/(1-C)^2 \quad , C<1 \tag{7}$$

in which θ_1 is the fraction of surface sites holding one molecule and C is the relative pressure; hence

$$K = \phi(1-C)^2/(1-\phi)\theta_1 \tag{8}$$

The quantity θ_1 is given by

$$\theta_1 = K_1P_oC(1-C)/\left[1 + (K_1P_o-1)C\right] \tag{9}$$

Since the number os surface sites (S_o) is small compared to the number of interior sites

$$We = A\phi \qquad , A \gg S_o \tag{10}$$

A combination of the last three equations yields the Fugassi equation. It can be seen from this discussion that

$$K \neq (H_2O)\text{ exterior}/(H_2O)\text{ interior}$$

so that none of the values reported in Table 23 are correct. Only when K is very small is this assumption approximately correct. It is interesting that these values approximate the BET monolayer value. This has been found to be true for the sorption of moisture onto bacterial surfaces[12] as well. An analysis of the Fugassi equation has not shown why this agreement exists.

The only clearly discernible trend which can be seen in the Fugassi A, K, and K_1 values is that A increased as the sorption temperature decreased, and there are many exceptions to this. The negative results are meaningless. They probably result from a breakdown of the assumptions of the Fugassi equation; that is, more than one molecule may be sorbed on a site or activated sorption at low relative pressures may be occurring. However, the Fugassi isotherm equation appears to offer a method of determining the relative proportion of surface and interior sorption sites as well as determining the amount of sorption at saturation pressure.

2.3.4 Heats of Sorption

The heat of sorption can be easily calculated by two different methods; from the Clausius-Clapeyron equation or from the BET "C" value. This latter value is valid, however, only in the relative pressure region 0.1 to 0.5 where a straight line plot is obtained. The values reported in Table 24 were calculated with the Clausius-Clapeyron equation. At sorption points above the BET monolayer level the heat of sorption of moisture onto all foods was essentially equal to the heat of liquifaction of water vapor. Below the monolayer levels, there was some variation in the heat of sorption. Those cases where the heat of sorption was less than the heat of condensation are indicative of sorption on isolated sites with little sorbate-sorbate interaction. Those cases where the heat of sorption was greater than the heat of liquifaction are indications of strong sorption on activated sites.

3. Banana Flakes

3.1. Experimental

3.1.1. Moisture Levels

The banana flakes as received contained 2.4% water. A portion was placed in a humidity cabinet saturated with water which raised the moisture to 23.2%. Blends of the 2 produced the 5 moisture levels.

3.1. Analytical Procedures

Extraction with 70% Ethanol

Sufficient bananas were weighed out so as to yield 10 gms of anhydrous food. Fifty cc of water was added and after soaking for 30 minutes the pH was taken. In Table 26 this pH is recorded as slurry pH. One hundred fifty cc of 95% alcohol was added and the mixture osterized for 1 minute. The slurry was filtered by means of Whatman No. 1 filter paper and the residue washed with 2-25 cc aliquots of 70% ethanol. The filtrate was adjusted to a final volume of 250 cc.

Formol Titration of Residue

The residue was washed from the filter paper with water and the pH increased to 8.5 with 0.1N NaOH. After the addition of 1 gtt of alkali the pH remained above 8.5 for 1 minute. Then 10 cc of formalin was added to each 100 cc of solution and after holding for 5 minutes returned to pH 8.5 in an identical manner.

Formol Titration of Filtrate

To 100 cc of the filtrate was added 100 cc of water and the pH of the solution brought to 8.5 with 0.1 N NaOH. Twenty cc of formalin was added and the pH returned to 8.5.

Absorption of the Filtrate at 280 mu, 390 mu, 420 mu and the Determination of Sugar as Dextrose.

An aliquot of the alcohol extract was diluted 1 to 400 (based on the weight of the banana flakes) with 75% ethanol for absorption at 280 mμ and 1 to 40 for absorption at 390 and 420 mμ.

For % sugar as dextrose the method of Folin and Wu for blood glucose was performed on 1 cc of the 1 to 400 solution.

3.1. Results and Discussion (Table 2)

Maximum browning occurred at a moisture content of 14.1% which corresponds to equilibration at 62.0% relative humidity. There was a maximum loss of sugar, amino nitrogen of the residue after alcohol extraction and alcohol extract. The titratable acidity of the residue and extract were enhanced. This may have been caused by the aldehyde groups of the sugars combining with the free amino acids and proteins. Likewise the pH of the aqueous slurring remaining after extraction with ethanol was the lowest of the storage samples. They may be due to the amino groups of the proteins combining with the aldehyde group of the sugars leaving the protein molecule more acidic.

3.1. Optimum Moisture Content

The banana flakes appeared to be the least stable when stored at a moisture content of 14.1% corresponding to equilibration at 62% relative humidity. Since water was not formed with time at the lowest moisture level which indicates the absence of reactions between polar groups with the formation of water, the preferred storage level may be 2.4% or lower. This was the amount of water they contained on arrival.

3.2 Egg Yolk Solids

3.2.1 Experimental

3.2.1.1 Procedure for Obtaining the Five Moisture Levels

The egg yolk as received contained 4.6% water. An aliquot was placed in the freeze dryer with the upper and lower

TABLE 25

BANANA FLAKES

	Initial % Moisture	Taste Test	Extraction with 70% Et OH/100 grams — % Sugar as Dextrose	1/400 280 Mμ	1/40 390 Mμ	1/40 420 Mμ	pN Slurry	IN NaOH to pN8.3	Alcohol Residue Mgs Amine Nit.	Extr… IN N… to p…
			17 Weeks - December 15, 1964							
rence	2.4	5.2	37.8	.134	.122	.08	4.9			
% R.H.	3.9	5.7	40.2	.112	.125	.083	4.85			
% R.H.	6.2	5.3	38.7	.112	.14	.09	4.85			
% R.H.	9.1	6.1	37.8	.115	.153	.098	4.83			
% R.H.	14.1	5.9	33.3	.105	.170	.11	4.7			
% R.H.	19.7	6.3	40.2	.105	.168	.11	4.75			
			22 Weeks - January 18, 1965							
rence		6.1	38.4	.106	.125	.095	5.0			
0% R.H.		6.8	40.4	.103	.13	.095	4.9			
5% R.H.		6.5	40.4	.114	.145	.095	4.9			
5% R.H.		6.6	38.4	.118	.17	.11	4.9			
0% R.H.		6.4	34.8	.118	.20	.13	4.8			
0% R.H.		5.8	44.0	.112	.185	.125	4.8			
rence	2.1	4.4	41.8	.095	.123	.092	4.95	4.6	23.5	28.
0% R.H.	3.6	4.8	43.6	.098	.13	.095	4.95	4.2	25.2	28.
5% R.H.	5.9	4.9	44.1	.108	.153	.098	4.9	4.1	24.1	28.
5% R.H.	8.8	2.8	40.4	.113	.175	.11	4.85	5.4	25.8	28.
0% R.H.	13.6	5.2	37.8	.115	.21	.135	4.75	5.2	19.0	31.
0% R.H.	19.9	4.7	45.0	.122	.21	.14	4.8	5.9	26.9	30.

e of Storage: August 18, 1964

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TABLE 25

NANA FLAKES

ol ue s Amine t.	Extract/100 grams Extract IN Na OH to pN 8.5	Mgs Amine Nit.	37.8°C	% Moisture	Taste Test	Extraction with 70% EtOH /100 grams % Sugar as Dextrose	1/400 280 Mμ	1/40 390 Mμ	1/40 420 Mμ	pH Slurry
						3 Weeks - September 8, 1964				
					5.0	41.4	.127	.130	.090	4.9
					5.4	44.8	.117	.133	.095	4.93
					6.1	42.6	.130	.172	.110	4.95
					5.3	41.4	.146	.200	.135	4.90
					5.3	34.0	.143	.210	.145	4.85
					6.2	44.4	.128	.180	.125	4.85
						7 Weeks - October 6, 1964				
					6.1	44.2	.125	.120	.09	4.9
					4.9	46.6	.133	.145	.10	4.9
					7.0	49.0	.165	.20	.125	4.9
					5.4	40.6	.190	.28	.185	4.9
					5.9	38.2	.213	.33	.215	4.7
					4.6	47.2	.243	.33	.21	4.7
						11 Weeks - November 3, 1964				
.5	28.0	74.2		2.4	6.2	46.5	.128	.13	.09	5.0
.2	28.2	46.2		3.0	5.8	45.0	.207	.17	.115	5.0
.1	28.9	50.4		5.3	6.7	43.5	.195	.24	.16	4.95
.8	28.5	47.6		8.3	5.8	42.0	.250	.37	.25	4.85
.0	31.2	43.4		13.3	5.8	37.5	.448	.47	.31	4.65
.9	30.0	60.2		21.8	-	48.8	.375	.49	.31	4.7

plates set at 100°F. In 3 hours both chambers had a vacuum of 15 microns so all the water was assumed out of the food. A moisture determination gave .52%. This egg yolk was blended manually with the 4.6% moisture material to obtain the 5 moisture levels for storage.

3.2.1.2 Analytical Methods

Extraction of the Fat with Ether

Sufficient egg yolk was weighed into a 250 cc beaker so that after the water was removed 10 grams of anhydrous food would be present. The water was removed by the freeze dryer in 3 1/2 hours. The egg yolk was mixed for 15 minutes with 100 cc of ether by means of a magnetic stirrer and then allowed to settle for 10 minutes. The supernatant was decanted on to a 15 cm sheet of Whatmen #40 filter paper. The extraction was continued with 50 cc aliquots of ether until a 250 cc volumetric flask was filled.

Percent Fat Extracted

A 50 ml aliquot of the ether extract was placed in a previously weighed 100 cc beaker and the ether removed by placing the beaker in the hood overnight. The last traces of solvent were removed by placing the beaker in a 100°C oven for 10 minutes. After cooling in a desiccator for 30 minutes the beakers were weighed and the amount of fat extracted calculated.

Free Fatty Acids as Oleic

To the above fat residue was added 75 cc of 95% ethyl alcohol and 3 cc of a 1% alcohol solution of phenolphthalein. The solution was heated to boiling and then titrated with 0.1N sodium hydroxide to a pink color.

Perioxide Number

A 40 cc aliquot of the ether extract was placed in a 500 cc Erlenmeyer flask, 50 cc of a solution of freshly prepared 60% acetic acid plus 40% chloroform added followed by 1 cc of a saturated potassium iodide solution. After shaking for exactly 1 minute, 5 cc of a 1% starch solution plus 95 cc of water was added. The blue color was titrated with .01N sodium thiosulfate. The results were expressed as millimoles peroxide per 1000 grams of fat.

Pancreatic Digestion of the Defatted Egg Protein

Two and one half grams of the protein remaining after extraction of the egg yolk with ether was suspended in 25 cc of water. After soaking for 30 minutes, the pH was taken and the pH adjusted to 8.5 with 0.1N NaOH. Then 1 cc of a solution of pancreatin (.3 gm pancreatin suspended in 7 cc water) was added, mixed and the sample placed in the incubator. At the end of 2 hours the pH was again adjusted to 8.5, the volume adjusted to 100 cc, the flask stoppered and placed back in the 37.8°C incubator for the night. The next morning a formol titration was performed on a 25 cc aliquot of the digest.

Digestion of the Egg Yolk with Pancreatic Lipase (13)

Sufficient egg yolk to give 2.5 grams of anhydrous solids was mixed with 20 cc of water, 2.5 cc phosphate buffer added (68 gms KH_2PO_4, plus 330 cc 1N KOH q.s. 1 l, pH 4.2) and 1 cc of the enzyme solution (.3 gm lipase plus 7.5 cc water). The mixture was stirred occassionally during the day to make sure the fat was completely broken up and then held overnight in the incubator. To the digestion solution was added 10 cc of water plus 7.5 grams KH_2PO_4. The mixture was washed into a separating funnel by means of 15 cc of water. Fifty cc of solvent was added (5 parts ethyl ether plus 1 part petroleum ether), the solution shaken for 30 seconds, another 50 cc of solvent added and the solution shaken again for 30 seconds. After 5 minutes the ether layer was decanted into a 250 cc volumetric flask. The digestion solution was extracted with 50 cc aliquots of solvent until the folumetric flask was filled.

Percent Fat Extracted and Free Fatty Acids as Oleic on the Egg Yolk Digested with Lypase

A 50 cc aliquot of the extract was placed in a tarred 100 cc beaker and the solvent removed by placing the beaker in the hood overnight. The beaker was reweighed and percent fat extracted calculated. To the fat residue was added 75 cc of 95% alcohol and 3 cc of a 1% alcohol solution of phenolphthalein. The solution was heated to boiling and titrated to a pink end point with 0.3N NaOH.

3.2.2 Results and Discussion

3.2.2.1 37.8°C Storage (Table 26)

There was an increase in the viscosity of an aqueous slurry of the egg yolk. This was first observed after 7 weeks of storage at 37.8°C in the egg yolk with an initial moisture content of 3.5%. When an omelet was prepared for the taste test the water slurry of the reference was water thin while this sample was approximately twice as viscous. After 11 weeks

EGG YOLK SOLIDS STABILIZED, HENNING

37.8°C	Initial % Moisture	Taste Test	Headspace Gas Analysis % CO_2	% O_2	pH Slurry	Pancreatic Digestion/100 grams IN NaOH to pH 8.5	pH Slurry after Digestion	IN NaOH Prior to Formalin	Gms Ami Nit.
						3 Weeks September 10, 1964			
Reference	4.6	7.4			6.1	15.6	7.05	97.8	1.27
4.0% R.H.	.52	7.2	.033	19.3	6.1	19.6	6.95	108.3	1.37
10.0% R.H.	1.58	6.3	.043	19.4	6.15	16.8	7.0	107.2	1.40
21.5% R.H.	2.61	7.0	.038	19.2	6.15	16.2	7.1	90.3	1.29
27.5% R.H.	2.95	6.7	.053	18.7	6.15	16.8	7.1	95.5	1.50
35.0% R.H.	3.53	6.4	.063	18.6	6.15	16.8	6.9	104.5	1.74
						7 Weeks October 8, 1964			
Reference		7.6	0.11	19.7	5.95	17.4	7.15	105.6	1.48
4.0% R.H.		5.6	0.13	18.8	6.0	17.8	7.2	104.8	1.42
10.0% R.H.		7.3	0.17	17.9	6.0	18.0	7.25	102.8	1.40
21.5% R.H.		6.2	0.25	17.0	6.0	18.0		106.4	1.54
27.5% R.H.		6.5	0.28	16.9	6.0	18.0	7.2	105.8	1.44
35.0% R.H.		6.6	0.31	16.9	6.0	18.2	7.15	109.4	1.37
						11 Weeks November 5, 1964			
Reference	4.55	7.8	0.25	20.0	6.1	16.6	7.35	96.2	1.33
4.0% R.H.	1.41	6.9	0.28	18.5	6.0	18.4	7.45	95.6	1.22
10.0% R.H.	1.95	5.8	0.23	19.7	6.0	18.0	7.4	95.6	1.20
21.5% R.H.	2.57	6.2	0.40	16.1	5.95	18.4	7.45	96.0	1.20
27.5% R.H.	3.00	6.6	0.35	18.0	5.95	18.8	7.45	95.0	1.18
35.0% R.H.	3.42	6.4	0.23	17.1	6.0	18.8	7.45	96.2	1.21

Date of Storage: August 20, 1964

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, HENNINGSEN FOODS, INC.

ms	Fat Analysis/100 grams				Lipase Digestion/100 grams	
H to in	Gms Amino Nit.	% Fat Extd.	% FFA as Oleic	Peroxide No.	% Fat Extd.	% FFA as Oleic
964						
	1.27	48.7	1.07	0		
	1.37	48.3	.97	0		
	1.40	48.0	.78	0		
	1.29	48.3	.97	0		
	1.50	48.4	1.07	0		
	1.74	48.9	1.07	0		
	1.48	47.3	1.2	0		20.3
	1.42	46.7	1.2	0		19.8
	1.40	47.0	1.4	0		26.6
	1.54	47.4	1.4	0		24.9
	1.44	47.9	1.4	0		19.8
	1.37	47.9	1.5	0		17.0
54						
	1.338	48.8	1.13	0	34.2	24.6
	1.226	46.7	1.13	0	34.7	22.0
	1.204	46.7	1.13	0	36.3	24.4
	1.204	47.3	1.36	0	33.5	24.0
	1.187	46.6	1.47	0	33.3	22.9
	1.210	47.1	1.58	0	38.8	27.4

B

all the samples produced an aqueous slurry so viscous that it was difficult to pour into the skillet. The lipoproteins may have unfolded or the very reactive groups of the phospholipids have combined producing large molecular weight aggregates. Possibly both are occurring simultaneously plus a multitude of other physical and chemical phenomena. Further evidence that the egg particles are larger was seen when the fat was extracted with ether. A small amount of fines passed through the filter paper in the case of the reference but were not observed in any of the storage samples.

The development of a bad odor and taste did not occur. Data on the foods which spoiled (shrimp, nonfat dry milk solids, and beef, (Table 39) indicates that this occurs when the food is stored at a moisture level corresponding to 61% relative humidity.

3.2.2.2 25°C Storage (Table 27)

The protein quality of flame-dried fish meals which are subjected to high temperature are inferior to steam-dried meals and vacuum dried meals are superior to both of these (14). Biological values of 70 ± 1.4 were obtained for flame-dried menhaden fish meal and 76 ± 1.9 for vacuum-dried meal (8% increase).

After 17 weeks of storage the egg yolk differed in its digestibility by pancreatin and lipase. The sample stored at a moisture content of 0.52% had the greatest reduction. The phospholipids of the egg yolk have changed so that they no longer are as readily digested. Mere reduction of the moisture to 0.53% does not seem detrimental because the reference which was kept frozen had this moisture content.

TABLE 27

EGG YOLK SOLIDS STABILIZED, HENNINGSEN F

25°C	Initial % Moisture	Taste Test	Headspace C+gs. Analysis % CO_2	% O_2	pH Slurry	Pancreatic Digestion/100 grams IN NaOH to pH 8.5	pH Slurry after Digestion	IN NaOH Prior to Formalin	Gms Amino Nit.
						17 Weeks	December 17, 1964		
Reference	0.53	7.7	.06	20.0	6.1	18.0	6.9	105.2	1.288
4.0% R.H.	2.4	7.7	.03	20.0	6.05	18.4	7.55	84.2	1.293
10.0% R.H.	2.9	7.4	.03	20.0	6.1	18.4	6.95	100.4	1.299
21.5% R.H.	3.2	7.0	.03	20.0	6.05	18.4	6.9	102.8	1.271
27.5% R.H.	3.6	7.4	.03	20.0	6.1	18.4	7.2	93.2	1.221
35.0% R.H.	3.9	7.4	.03	20.0	6.1	18.0	7.5	85.6	1.232
						25 Weeks	February 11, 1965		
Reference		7.6	.12	19.98	5.85	11.4	7.8	124.2	1.053
4.0% R.H.		7.5	.075	20.03	6.0	10.4	7.6	129.6	.930
10.0% R.H.		7.0	.07	20.03	6.0	11.0	7.6	130.2	.930
21.5% R.H.		7.3	.07	20.03	6.0	11.0	7.75	128.8	.963
27.5% R.H.		7.3	.07	20.03	5.95	10.8	7.5	140.4	.963
35.0% R.H.		7.3	.075	20.03	6.0	12.0	7.8	133.6	.952
						33 Weeks	April 8, 1965		
Reference	4.6	7.7	.133	20.0	6.02	19.4	7.22	108.0	1.622
4.0% R.H.	2.6	7.3	.083	18.5	6.0	19.8	7.22	105.2	1.613
10.0% R.H.	2.8	7.2	.082	19.7	6.0	18.9	7.3	105.2	1.653
21.5% R.H.	3.1	7.5	.081	16.1	5.95	19.7	7.22	109.2	1.555
27.5% R.H.	3.4	7.5	.083	18.0	6.0	19.4	7.38	100.6	1.431
35.0% R.H.	3.4	7.3	.082	17.1	6.0	19.8	7.4	101.6	1.467

Date of Storage: August 20, 1964

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ABLE 27

IZED, HENNINGSEN FOODS, INC.

grams		Fat Analysis/100 grams			Lipase Digestion/100 grams	
NaOH ·or to ·malin	Gms Amino Nit.	% Fat Extd.	% FFA as Oleic	Perioxide No.	% Fat Extd.	% FFA as Oleic
.964						
.2	1.288		1.13	0	37.9	28.33
.2	1.293	48.0	1.19	0	36.5	22.11
.4	1.299	48.6	1.24	0	38.3	24.87
.8	1.271	48.4	1.36	0	35.9	24.87
.2	1.221	48.14	1.41	0	38.7	25.56
.6	1.232	49.79	1.47	0	37.0	24.87
55						
2	1.053	48.54	1.24	0	38.5	27.6
6	.930	47.26	1.41	0	34.8	23.3
2	.930	48.12	1.36	0	37.4	27.6
8	.963	47.62	1.47	0	35.4	24.2
4	.963	48.38	1.70	0	36.8	27.6
6	.952	48.91	1.75	0	36.7	27.6
[illegible]	1.622	47.6	1.24	0	35.6	25.9
[illegible]	1.613	46.5	1.22	0	36.2	23.1
[illegible]	1.653	47.6	1.38	0	37.2	24.9
[illegible]	1.555	48.8	1.52	0	35.6	24.2
[illegible]	1.431	49.9	1.64	0	38.6	27.6
[illegible]	1.467	47.4	1.62	0	38.3	26.3

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The proteins of egg yolk have been poorly characterized. Two lipoprotein fractions, lipovitellin and lipovitellinin, have been prepared from egg yolk. Phospholipids, almost entirely lecithins, are found in these two proteins. The properties and compositions of these lipoproteins are apparently dependent upon the method of preparation (15).

The data for pancreatic digestion at 25 weeks differs because instead of using defatted protein the egg yolk as such was digested with pancreatin.

The moisture content of the sample stored at 0.5% water increased to 2.4% after 17 weeks presumably due to phospholipid interaction. The moisture in this sample only increased to 1.4% after 11 weeks at 37.8°C. Other reactions must be occurring simultaneously, as fat hydrolyzes which uses up water. The formation of water must have some connection with the diminished digestion by lipase. Water was formed in all the samples and all were digested less by the lipase than the reference. These linkages between the polar groups of lipids which result in the formation of water, the lipase apparently cannot hydrolyze. It would be interesting to again remove the water formed on storage and place the egg yolk back in storage. If this were repeated enough times it might be possible to make this food completely indigestible.

The odor of the solution after digestion with lipase was very disagreeable. The ether-petroleum ether extract after the removal of the solvent in the hood could not be placed in an oven to drive off the last traces of solvent. The odor drove everyone out of the room and clung to the oven for weeks.

3.2.2.3 Optimum Moisture Content

None of the moisture levels used for storage is the optimum level because water is being formed on storage. This indicates that chemical reactions are occurring with the formation of water. The compounds involved may be lipoprotein and the linkages so formed are not split by lipase. The egg yolk as purchased contained 4.6% water. Further work would be required to establish is this is the optimum moisture level for storage.

3.3 Dehydrated Cooked Ground Beef

3.3.1 Experimental

3.3.1.1 Moisture Levels

The beef as received contained 5.5% water. The moisture content was reduced to 1.3% by holding the beef overnight in the freeze dryer at 100°F. The moisture content was raised to 12.6% by placing the beef in a chamber overnight which was saturated with water vapor. The intermediate moisture levels were made by blending appropriate amounts of these two together. The beef was stored in glass jars with the covers taped shut so that air or water could not enter or leave.

3.3.1.2 Analytical Procedure

Extraction of Fat

Enough beef was weighed out so that after removal of the water 30 grams of anhydrous food would remain. The moisture was reduced to a low level by placing the beef in the freeze dryer for 4 hours with the upper and lower plates at 100°F. By means of a magnetic-stirrer the beef was extracted 5 times with

50 cc of ether. The beef and ether were mixed for 15 minutes and then allowed to settle for 10 minutes. The supernatant was decanted on a 15 cm #4 filter paper and the filtrate collected in a 200 cc volumetric flask.

Percent Fat Extracted

A 50 ml aliquot of the ether extract was placed in a previously weighed 100 cc beaker and the ether removed by placing the beaker in a 100°C oven for 10 minutes. After cooling in a desiccator for 30 minutes the beakers were weighed and the amount of fat extracted calculated.

Free Fatty Acids as Oleic

To the above fat residue was added 75 cc of 95% ethyl alcohol and 3 cc of a 1% alcohol solution of phenolphthalein. The solution was heated to boiling and then titrated with 0.1N sodium hydroxide to a pink color.

Perioxide Number

A 40 cc aliquot of the ether extract was placed in a 500 cc Erlenmeyer flask, 50 cc of a solution of freshly prepared 60% acetic acid plus 40% chloroform added followed by 1 cc of a saturated potassium iodide solution. After shaking for exactly 1 minute, 5 cc of a 1% starch solution plus 95 cc of water was added. The blue color was titrated with .01N sodium thiosulfate. The results were expressed as millimoles peroxide per 1000 grams of fat.

Extraction of the Defatted Beef with Water

Ten grams of the beef was mixed with 100 cc of water and allowed to soak for 30 minutes. The sulurry was mixed for 15 minutes with a magnetic stirrer and then allowed to stand for 10 minutes. The supernatant was decanted on to a sheet of 12.5 cm #2 filter paper followed by a 15 cm #40 filter paper. The residue was extracted 4 more times with 50 cc of water and the filtrate collected in a 200 cc volumetric flask.

Formol Titration of the Extracted Residue

The pH of the slurry was raised to 8.5 with 0.1N sodium hydroxide. When after 1 drop of alkali the pH remained at or above 8.5 for 1 minute the formalin was added (1 cc for each 20 cc of slurry). The formaldehyde was allowed to react for 5 minutes and then the pH was brought back to 8.5 in the same manner.

Formol Titration of the Aqueous Extract

A 40 cc aliquot of the extract was brought to a pH value of 8.5 with 0.1N sodium hydroxide. Two cc of formalin was added and the pH again brought to 8.5. The results were expressed as milligrams amino nitrogen per 100 grams of the defatted beef.

Reaction of the Aqueous Extract with Nitrous Acid

To 50 cc of the aqueous extract was added 0.8 cc 4 N hydrochloric acid and 20 cc 0.1N sodium nitrite. The reaction was allowed to proceed for exactly 5 minutes, 0.1N sodium hydroxide ran in rapidly to within 1 cc of the end point and then continuously until pH 8.5 was reached. The results were expressed as milligrams alpha amino nitrogen per 100 grams of beef protein.

Hematin Absorbance at 470 Millimicrons

The aqueous extract was centrifuged for 15 minutes at 10,000 rpm and the optical density recorded at 470 millimicrons.

Thiobarbituric Acid Test on Beef

Five grams of beef, 97.5 cc of water, 2.5 cc 4N HCL and a drop of Dowfoam A were steam distilled at a rate where 50 cc of distillate was collected in 10 minutes. Five cc of the distillate plus 5 cc of .02 M 2-thiobarbituric acid were immersed in a boiling water bath for 35 minutes. The tubes were cooled in tap water for 10 minutes and read at 538 millimicrons. Simultaneously a malonaldehyde standard curve was prepared.

3.3.2 Results and Discussion

3.3.2.1 37.8°C Storage (Table 28)

According to the taste panel, the beef stored with a moisture content of 7.8% spoiled after 7 weeks of storage. It had a decayed putrid odor. It differed from the reference and the other samples in storage in that the defatted protein as well as an aqueous extract of the defatted protein contained less amino nitrogen and the hematin pigments of the aqueous extract were enhanced. The reduction in amino nitrogen accounts for the increased titratable acidity of the beef slurry as well as its low pH. The protein is more acidic.

TABLE 28

WILSON'S DEHYDRATED COOKE

7.8°C	Initial % Moisture	Taste Test	Headspace Gas Analysis % CO_2	% O_2	% Fat Extd	Fat Analysis/100 gms Fat FFA as Oleic	Millimoles Peroxide pr 1000 gms Fat	Extr. of Defa Formal titn of pH Slurry	CCN NaOH pH 8.	Ext Form pH Slurr
							3 Weeks	September 18, 1		Septe
eference	5.5	7.0	.011	20.6	22.5	1.1	0	6.05	12.0	6.05
.0% R.H.	1.2	7.1	.011	18.7	10.3	1.1	0	5.75	12.6	5.75
9.0% R.H.	3.7	6.8	.035	18.8	21.8	1.1	0	5.7	15.2	5.7
7.0% R.H.	5.3	7.1	.810	18.2	22.2	1.3	0	5.8	15.8	5.8
0.5% R.H.	7.8	6.8	1.02	16.1	22.2	1.5	0	5.75	15.2	5.75
6.0% R.H.	12.6	6.7	1.0	15.7	20.6	2.2	0	5.5	15.8	5.5
							7 Weeks	October 16, 196		Octob
eference		7.5	.13	20.4	17.8	1.2	0	5.7	15.6	5.7
.0% R.H.		6.3	.27	18.8	15.2	1.1	0	5.45	16.5	5.45
9.0% R.H.		6.8	.40	19.1	19.0	1.4	0	5.6	15.5	5.6
7.0% R.H.		6.3	1.16	14.5	19.4	1.3	0	5.6	16.8	5.6
0.5% R.H.		4.8	1.17	16.9	20.7	2.1	0	5.45	16.5	5.45
6.0% R.H.		6.3	1.21	17.1	21.7	3.5	0	5.4	17.	5.4
							11 Weeks	November 13, 1		Nove
eference	5.2	6.8	.14	20.0	21.0	1.1	3.2	5.7	16.	5.7
.0% R.H.	1.7	6.8	.26	18.4	22.2	1.1	67	5.3	18.	5.3
9.0% R.H.	3.6	6.2	.36	18.7	22.5	1.5	2250	5.6	18.	5.6
7.0% R.H.	4.3	6.2	.94	15.5	21.2	1.7	2220	555	20.	555
0.5% R.H.	5.8	4.2	.95	17.1	22.0	2.4	1200	5.5	18.	5.5
6.0% R.H.	11.1	6.8	1.08	16.9	23.8	4.1	2.8	5.45	21.	5.45

ate of Storage: August 28, 1964.

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;OOKED GROUND BEEF

Defatted Beef with Water

;n of residue 100 gms   Aqueous Ext/100 gms

;CN NaOH to pH 8.5	Mgs Amine Nit.	pH Slurry	CCN NaOH to pH 8.5	Formal Titn	HNO_2 Titn	Hematin Abspn 470 Mμ	Mgs TEP Equiv/ 100 gms
l8, 1964							
12.0	336	5.7	22.0	137		.048	.34
12.6	305	5.8	20.4	137		.048	.34
15.2	414	5.75	23.6	149		.05	.34
15.8	363	5.85	22.6	153		.06	.34
15.2	353	5.85	22.0	145		.06	.20
15.8	372	5.75	21.2	151		.048	.15
, 1964							
15.6	445	5.8	24.0	162	90	.058	.38
16.5	412	5.7	23.8	151	93	.052	.35
15.5	400	5.7	24.4	151	62	.053	.34
16.8	413	5.65	[illegible]5.2	146	129	.068	.27
16.5	333	5.7	23.2	140	134	.07	.20
17.4	361	5.65	23.2	148	78	.058	.15
.3, 1964							
16.8	434	5.75	22.8	151	46	.048	.35
18.6	428	5.8	22.8	147	14	.047	.24
18.3	430	5.75	22.8	140	11	.049	.18
20.4	437	5.8	22.0	140	33	.057	.17
18.3	433	5.75	22.5	133	14	.06	.10
21.2	442	5.7	22.8	140	-25	.045	.06

B

Changes occurred in the physical properties of the beef during storage. When the fat was extracted from the beef with ether the beef particles stored at moisture levels above the monolayer value settled very slowly in the ether and the beef residue occupied a larger volume than the reference. Filtration was very rapid though. The two beef samples stored at moisture levels below the monolayer value settled very rapidly to a compact mass but filtration was very slow because beef fines clogged the pores in the filter paper. The sample with the monolayer moisture level resembled the reference in these respects.

Above the monolayer moisture level there is an unfolding of the beef particles which probably is due to hydrolysis of lipids that causes an increase in the size of the beef particles. The fines disappear so filtration is very rapid. The increase in free fatty acids and decrease in moisture is evidence that the fat is being hydrolyzed. The beef stored at a moisture content of 1.2% had more fines than the reference and other samples with filtration very slow. The beef particles have become smaller and more compact. Polar groups of phospholipids presumably have reacted with other lipids as well as protein producing a more compact beef particle. Such reactions lower the amino nitrogen of the beef protein and account for the increase in moisture on storage.

The ether extract of the beef with an initial moisture content of 1.2% was lighter in color than the reference. This may be due to the oxidation of ether soluble pigments. The B-Carotene of sweet potatoes rapidly disappeared at this moisture level.

After storage for 11 weeks at 37.8°C none of the samples resemble the reference upon extraction of the fat with ether. The reference particles after mixing with ether settled rapidly and filtered slowly. In all the storage samples, the beef particles settled slowly with filtration very rapid. After this length of storage at this high a temperature the beef has become greatly altered physically from lipoprotein hydrolysis and hydrolysis of the amide groups from glutamine. The linkages formed through reactions of lipoprotein which caused the enhancement of fines presumably have been broken. The data supports these assumptions. There is an increase in free fatty acids and a loss of water. The molecule is more acidic as shown by the lower pH of the water slurry and the increased amount of alkali required to raise the pH to 8.5. However, the amino nitrogen remains stable which indicates that peptide bonds have not been hydrolyzed. Flavor wise the optimum moisture content for maximum deterioration was 7.8%.

3.3.2.2 <u>25°C Storage (Table 29)</u>

Upon extraction of the fat from the beef with ether after 12 and 18 weeks of storage in the samples with an initial moisture content of 1.3 and 3.7% there appeared to be an excessive amount of greasy material which adhered to the upper edge of the filter paper. The 1.2% moisture sample also contained an excessive amount of fines which refused to settle and a portion of these small particles passed through the filter paper. The reference only had a few such particles small enough to pass

TABLE

WILSON'S DEHYDRATEI

25°C	Initial % Moisture	Taste Test	Headspace Gas Analysis % CO_2	Headspace Gas Analysis % O_2	% Fat Extd	Fat Analysis/100 gms Fat: FFA as Oleic	Fat Analysis/100 gms Fat: Millimoles Peroxide pr 1000 gms Fat	Extr. Formal Ti: pH Slurry
							12 Weeks	November
Reference	5.4	6.1	.219	19.9	21.0	1.2	0	5.65
7.0% R.H.	1.6	6.4	.139	18.9	20.3	1.2	0	5.5
29.0% R.H.	4.3	6.2	.187	19.4	20.4	1.2	0	5.6
47.0% R.H.	5.3	5.9	.145	21.1	21.7	1.4	0	5.6
60.5% R.H.	7.6	6.1	.167	20.0	21.1	2.0	0	5.5
76.0% R.H.	12.9	5.9	2.48	15.8	22.2	3.0	0	5.3
							18 Weeks	January
Reference		7.5	.44	19.7	21.6	1.5	0	5.6
7.0% R.H.		6.8	.15	20.0	20.6	1.2	0	5.7
29.0% R.H.		71	.21	19.9	21.2	1.6	0	5.5
47.0% R.H.		7.3	.18	19.9	21.5	1.5	0	5.6
60.5% R.H.		7.0	.14	20.0	23.0	2.3	0	5.6
76.0% R.H.		7.2	14.4	5.7	23.1	3.5	0	5.4
							24 Weeks	February
Reference	5.8	6.6	.625	19.48	18.9	1.6	0	5.6
7.0% R.H.	1.4	7.1	.165	19.94	18.6	1.8	1.3	5.45
29.0% R.H.	3.6	7.3	.165	19.94	19.7	1.5	16.9	5.5
47.0% R.H.	5.3	6.6	.13	19.8	19.3	1.7	13.7	5.45
60.5% R.H.	7.3	5.4	.155	19.95	21.7	2.6	10.8	5.4
76.0% R.H.	12.4	5.8	15.0	5.1	20.9	24.3	18.8	5.1

Date of Storage: August 28, 1964

A

TABLE 29

HYDRATED COOKED GROUND BEEF

Extr. of Defatted Beef with Water 'ormal Titn of residue/100 gms			Aqueous Ext/100 gms					
H lurry	CCN NaOH to pH 8.5	Mgs Amine Nit.	pH Slurry	CCN NaOH to pH 8.5	Formal Titn	HNO_2 Titn	Hematin Abspn 470 M	Mgs TEP Equiv/ 100 gms
November 24, 1964								
5.65	17.4	455	5.75	23.0	140	63	.04	0.3
5.5	16.8	375	5.7	23.0	133	35	.048	0.3
5.6	16.2	417	5.8	23.0	126	49	.038	0.3
5.6	16.0	389	5.85	22.5	140	28	.053	0.3
5.5	19.3	402	5.8	23.0	119	35	.055	0.24
5.3	18.2	409	5.65	22.5	126	21	.03	0.18
January 6, 1965								
5.6	17.7	413	5.8	21.0	140	70	.038	
5.7	18.4	448	5.73	21.5	133	7	.034	
5.5	23.2	448	5.7	22.0	126	14	.033	
5.6	18.2	454	5.7	22.0	126	28	.034	
5.6	16.0	361	5.7	22.0	126	14	.039	
5.4	19.4	410	5.65	21.5	119	14	.027	
'ebruary 17, 1965								
5.6	15.6	417	5.8	23.0	147		.05	
5.45	17.0	358	5.7	24.5	133		.045	
5.5	16.9	400	5.7	30.0	182		.046	
5.45	16.7	398	5.7	24.3	140		.05	
5.4	18.6	420	5.55	23.5	140		.055	
5.1	20.6	400	5.5	30.5	168		.063	

B

through the filter paper. The 2 samples with a moisture content of 7.8 and 12.6% did not have any fines nor the greasy material. The reference and monolayer moisture sample were approximately the same in this respect.

In Table 30 may be found the approximate weight of the greasy material, the weight of the defatted residue and the total of the two. Also the grams of fat extracted are listed. The total weight of beef insoluble in ether is the same for the reference and storage samples. The greasy substance, excessive fines in the low moisture sample, and their absence at high moisture levels must be physical phenomena. As explained previously when discussing the storage data at 37.8°C the phospholipid moiety of the lipoproteins are thought to be involved. The slight decrease in the amount of fat extracted at the low moisture levels is believed to be caused by the formation of water through chemical reactions of polar groups in ether soluble phospholipids. Likewise the slight increase in the amount of fat extracted above the monolayer is due to hydrolysis of ether soluble lipids. The weight increase is the weight of water taken up through hydrolysis. The reference and sample stored at the monolayer moisture level are very nearly the same. This may indicate fat stability or that a balance exists between reactions involving the formation and uptake of water.

The outstanding characteristic of the lecithins is their high chemical reactivity: they are easily oxidized or

TABLE 30

WILSON'S DEHYDRATED COOKED

	Initial % H_2O	% H_2O	Wt. of Defatted Residue	Wt. of Lipiton Filter Paper	Total	Gms Fat Extd
			12 Weeks 25°C			
Reference	5.5	5.4	23.2	1.3	24.3	6.3
7.0% R.H.	1.2	1.6	22.7	1.8	24.5	6.0
29.0% R.H.	3.7	4.3	22.8	1.6	24.4	6.1
47.0% R.H.	5.5	5.3	23.3	1.1	24.4	6.4
60.5% R.H.	7.8	7.6	23.5	1.1	24.6	6.3
76.0% R.H.	12.6	12.9	23.7	0.9	24.6	6.6
			18 Weeks 25°C			
Reference			23.6	2.0	25.6	6.4
7.0% R.H.			23.0	1.7	24.7	6.1
29.0% R.H.			23.2	1.3	24.5	6.3
47.0% R.H.			23.5	1.3	24.8	6.4
60.5% R.H.			23.9	0.7	24.6	6.8
76.0% R.H.			24.1	0.6	24.7	6.9
			24 Weeks 25°C			
Reference		5.8	23.6			5.6
7.0% R.H.		1.4	22.8			5.5
29.0% R.H.		3.6	23.25			5.8
47.0% R.H.		5.3	23.5			5.7
60.5% R.H.		7.3	23.2			6.3
76.0% R.H.		12.4	23.7			6.[illegible]

TABLE 30

YDRATED COOKED GROUND BEEF

Total	Gms Fat Extd	% H_2O	Wt.of Defatted Residue	Wt.of Filter Paper	Total	Gms Fat Extd
			3 Weeks 37.8°C			
24.3	6.30					6.76
24.5	6.08					3.10
24.4	6.11					6.54
24.4	6.49					6.67
24.6	6.32					6.66
24.6	6.68					6.18
			7 Weeks 37.8°C			
25.6	6.48					5.35
24.7	6.18					4.55
24.5	6.36					5.69
24.8	6.44					5.82
24.6	6.89					6.21
24.7	6.94					6.51
		R.H. 28%	11 Weeks 37.8°C			
	5.68	5.2	22.9			6.31
	5.57	1.7	22.9			6.68
	5.89	3.6	22.9			6.74
	5.78	4.3	23.3			6.36
	6.37	5.8	23.8			6.61
	6.27	11.1	23.3			7.13

hydrolyzed, and easily combined with a number of other substances such as proteins and carbohydrates. They are found in all plant and animal tissue, are subject to rapid deterioration in air, are readily hydrolyzed by relatively mild acid or alkaline conditions with cleavage of both the phosphate and fatty acid ester linkages. Naturally occurring lecithin fractions ordinarily are highly unsaturated and exhibit iodine numbers of 100 or greater. Lecithin rapidly autooxidizes to a brown material having the typical tallowy odor of oxidized lipides. This odor is presumably due to unsaturated carbonyl compounds (16).

Table 31 contains the composition of the foods studied in this contract (17). The milk, beef and shrimp are the only foods that developed bad odors and flavors. The egg yolk would have spoiled if the moisture had been a little higher. The one outstanding difference in these foods from the foods that did not spoil is the phosphorus content. Of the storage fat in muscle the phospholipids predominate with mammalian muscles on the average containing 4.5% and .25% cholestrol. Future analytical work on this type of food will be directed at the changes which occur in this group of compounds.

3.3.2.3 Optimum Moisture Level

The data indicates that the lipids of the beef are more stable when stored at the monolayer moisture content. However at this moisture level headspace gas analysis shows that oxygen uptake by the beef is at a maximum so the beef should be packed under nitrogen or carbon dioxide. The beef as purchased, contained the monolayer amount of moisture.

TABLE 31

COMPOSITION OF FOODS, 100 G., EDIB

	% Water	Cal Food Energy	G Protein	G Fat	Carbohydrate Total	Carbohydrate Fiber	A
Milk, nonfat (skim)	90.5	36	3.5	.1	5.1	0	.
Beef, medium fat carcass	63	240	18.2	18	0	0	.
Shrimp, canned	66.2	127	26.8	1.4	-	-	5.
Potatoes, raw	77.8	83	2.0	0.1	19.1	.4	1.
Sweet potatoes, boiled	68.5	123	1.8	0.7	27.9	1.0	1.
Eggs, yolk	49.4	361	16.3	31.9	.7	0	1.
Apples, raw	84.1	58	.3	.4	14.9	1.0	.
Bananas, raw	74.8	88	1.2	.2	23	.6	.
Rice, cooked	70.5	119	2.5	.1	26.2	.1	.

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LE 31

100 G., EDIBLE PORTION (2)

ydrate Fiber	G Ash	Mg Calcium	Mg Phos-phorous	Mg Iron	I.U. V.t A	Mg Thiamine	Mg Ribo-flavin	Mg Niacin	Mg Ascorbic Acid
0	.8	123	97	.1	Trace	.04	.18	.1	1
0	.9	11	161	2.7	0	.08	.16	4.4	0
-	5.8	115	263	3.1	60	.01	.03	2.2	0
.4	1.0	11	56	.7	20	.11	.04	1.2	17
1.0	1.1	30	49	.7	7.700	.09	.05	.6	20
0	1.7	147	586	7.2	3.210	.27	.35	Trace	0
1.0	.3	6	10	.3	90	.04	.03	12	5
.6	.8	8	28	.6	430	.04	.05	17	10
.1	.7	8	45	.3	0	.01	.01	.4	0

B

3.4 Shrimp

3.4.1 Experimental

3.4.1.1 Method for Obtaining Moisture Levels

The shrimp was placed in a cabinet saturated with water vapor and equipped with a fan. The following periods of time in the chamber increased the moisture content of the shrimp as follows: 0 hours, 1.6%; 1/2 hour, 5.4%; 1 hour, 6.6%; 4 hours, 10.1%; and 10 hours, 18.0%.

3.4.1.2 Analytical Methods

Volatile Amine Determination

Enough shrimp was weighed out so as to yield 25 grams of anhydrous food. The shrimp was ground to a fine powder and placed in a flask containing 250 cc of water, 10 gms of sodium citrate and a smidgeon of Dowfoam A. The receiver was connected to a graduated cylinder containing 1.5 cc 4 N HCl and 23.5 cc of water. The rate of distillation was regulated so as to require about 30 minutes to collect 65 cc of distillate.

Amines Which Neutralize HCl

A 25 cc aliquot of the distillate was brought to a pH of 8.5 with 0.1N NaOH. The difference in titration between the blank and the unknown gave the milligrams amine nitrogen which reacted with the HCl.

Amines Which Reacted with Nitrous Acid

To a 25 cc aliquot of the steam distillate was added 10 cc of 0.1N Nitrous Acid and then held for exactly 10 minutes. The pH was rapidly brought up to 8.5. The difference in alkali required between the blank and unknown gave the amines which reacted with nitrous acid.

Amines Which Reacted with Formalin

A 25 cc aliquot was brought to pH 8.5 with 0.1N Na OH, 1 cc formalin added and the pH returned to 8.5. The difference between the blank and unknown gave the milligrams amine nitrogen which reacted with the formalin.

Digestion with Pancreatin

For each moisture level enough shrimp was ground with a mortar and pestle to yield 2.5 gms of anhydrous food. The shrimp was soaked in 50 cc of water for 30 minutes and the slurry pH taken. With a magnetic stirrer - pH meter assembly the pH was increased to 8.5 with 0.1N NaOH. Then 1 cc of a suspension of pancreatin (0.3 gm pancreatin plus 1 cc of water) was mixed with the shrimp and then held for 2 hours at 37.8°C whereupon the pH was again adjusted to 8.5. The process was repeated in 4 hours, the volume adjusted to 200 cc, the flask stoppered and placed in the incubator overnight. The next morning a formal titration was performed on a 50 cc aliquot.

Digestion with Pepin

The shrimp, after grinding to a fine powder, was weighed out so as to yield 2.5 gms of anhydrous food. The digestion solution was composed of 2.5 gm pepsin, 15.25 cc concentrated HCl and enough water to make a total volume of 2500 cc. The shrimp was mixed with 400 cc of the digestion solution, shaken several times during the day and then held overnight at 37.8°C. A formal titration was performed on a 50 cc aliquot.

3.4.2 Results and Discussion

3.4.2.1 25°C Storage (Table 32)

The shrimp deteriorated very rapidly. Because so many reactions were going on so fast and simultaneously, interpretation of the data is more difficult than for the other foods. For example, volatile amines were continuously being formed as well as consumed. The shrimp differs in composition from the rice, bananas and apples which the taste panel judged as satisfactory after storage at 25°C and 37.8°C, by having more protein, fat, ash, calcium, phosphorous and iron and having no fiber, starch or sugar. (Table 28).

The shrimp was purchased in tin cans packed under nitrogen and did not require refrigeration. When exposed to the air and water added for the 5 storage conditions, rapid deterioration began resulting in a foul, amine-like odor. The shrimp with an initial moisture content of 10.1 and 18.0% became tough and difficult to chew. When ground with a mortar and pestle, the tissue was broken down into tough fibers that resembled soft wood and which were almost impossible to reduce to a fine powder. The reference and shrimp with a moisture content below the monolayer value were easily and quickly pulverized to an extremely light, fluffy powder. The rich red color of the shrimp faded to a pink and the 2 high moisture samples browned a little.

Digestion with pepsin and pancreatin did not indicate any great impairment in digestion of the protein. Formol

TABLE 32

WILSON'S FULLY COOKED FREEZE

25°C	% Moisture	Taste Test	Steam Distillate Mgs Amine N/100 gms Anhydrous Shrimp: Amines which Neut. Hcl	Amines React with HNO_2	Amines React Formalin	Pancreatic Digestion/1 Anhydrous Shrimp: pH Slurry	IN NaOH to pH 8.5
						6 Weeks October 23, 1964	
Reference		6.6	50.4	56	67.2	7.45	
4.8% R.H.		6.1	11.2	11	61.6	7.35	
28.5% R.H.		6.2	28.0	27	56.0	7.45	
35.5% R.H.		6.0	22.4	17	50.4	7.65	
59.5% R.H.		4.2	16.8	17	61.6	7.5	
78.5% R.H.		3.6	0	0	61.6	7.35	
						12 Weeks December 4, 1964	
Reference	1.7	5.6	16.8	11.2	50.4	7.5	13.8
4.8% R.H.	2.6	5.3	11.2	5.6	77.2	7.5	15.4
28.5% R.H.	6.8	5.2	72.8	56.0	50.4	7.5	15.4
35.5% R.H.	7.1	4.9	56.0	39.2	77.2	7.45	14.4
59.5% R.H.	10.5	Bad Odor	77.2	44.8	77.2	7.6	11.8
78.5% R.H.	15.3	Bad Odor	61.6	16.8	77.2	7.45	14.2
						18 Weeks January 15, 1964	
Reference	1.7		22.4	28.0	50.4	7.55	12.0
4.8% R.H.	2.9	Bad Odor	39.2	44.8	44.8	7.4	12.8
28.5% R.H.	6.2	Bad Odor	50.4	56.0	72.8	7.55	13.0
35.5% R.H.	6.8	Bad Odor	22.4	16.8	44.8	7.7	11.2
59.5% R.H.	12.4	Bad Odor	56.0	84.0	72.8	7.55	11.4
78.5% R.H.	16.2	Bad Odor	44.8	50.4	16.8	7.25	20.0

Date of Storage: September 11, 1964

A

TABLE 32

LY COOKED FREEZE DRIED SHRIMP

eatic Digestion/100 gms
rous Shrimp

Pepsin Digestion

ɾ	IN NaOH to pH 8.5	IN NaOH Prior to Formalin	Gms Amine Nit.	N NaOH Prior to Formalin	Gms Amine Nit.
ɔctober 23, 1964					
		116	1.83	115	1.52
		123	1.90	115	1.34
		123	1.74	128	1.52
		119	1.75	115	1.25
		122	1.48	122	1.39
		120	1.58	102	1.52
December 4, 1964					
	13.8	113	3.09	74	1.34
	15.4	114	3.18	70	1.43
	15.4	119	2.76	90	1.43
	14.4	121	2.89	70	1.48
	11.8	109	2.78	74	1.43
	14.2	127	2.76	74	1.43
January 15, 1964					
	12.0	179	3.83	77	1.12
	12.8	181	3.54	99	1.17
	13.0	178	3.81	112	1.21
	11.2	177	3.41	123	1.21
	11.4	172	3.79	102	1.17
	20.0	181	3.58	119	1.30

titration of the digest of the stored samples showed a lower amino nitrogen than the reference. The amount of alkali required to raise the pH of the digest to 8.5 prior to the addition of formalin was the same for the reference and storage samples. The disappearance of amino nitrogen probably occurred through the reaction of polar groups with components as phospholipids. The digestive disturbances might be due to the formation of toxic compounds which upset the bacterial flora of the intestine and enzymes with concomitant flatulence, pain, malaise, etc.

Future analytical work on foods of this type will consist of finding out the changes the phospholipids undergo. How well are they digested by enzymes and the extent they react with themselves as well as with other food constituents. Phospholipids are composed of extremely reactive compounds. Lecithin turns brown on exposure to air because of the oxidation of the unsaturated acids present. One of the products of the hydrolysis of lecithin, accounting for about 15% of the molecule is choline which is a moderately strong base. In combination with acetic acid the compound acetylcholine is formed which is of significance in nerve activity. An enzyme in cobra venom splits off the unsaturated fatty acid radical producing a compound which has a strong hemolytic action on red blood cells (18). From the active part that water plays on the storage behavior of these foods there must be all types of hydrolytic reactions going on. Linkages are split and then reformed with some other molecule.

Another very active constituent of lecithin is phosphoic acid. As little as one mole of phosphate per mole of pepsin may serve in the stabilization of the 3 dimensional protein structure. A single phosphate group in pepsin forms a cluster linking 2 sites of the polypeptide chain to form a cyclic loop (19). The thickening of the egg yolk aqueous slurry and the toughening of the shrimp with storage maybe caused by reactions involving these very active groups of phospholipids.

The instability of the shrimp was exemplified in the data on distillation of amines. The volatile amines which neutralized HCl of the reference which was kept frozen dropped from 50.4 to 16.8 mgs after 11 weeks and then started to increase. The volatile basic nitrogen includes mainly ammonia, trimethylamine and dimethylamine (20). Trimethylamine is found in the conjugated form and results from the degradation of the tissue. It is a strong base. Dimethylamine is very soluble in water forming a very strong alkaline solution. It may be irritating to the skin and mucous membranes.

After 6 weeks of storage the amines have declined at all moisture levels but the greatest loss occurred at the lowest and highest moisture levels. After 12 weeks the amines which neutralized HCl dropped from 50.4 to 16.8 mgs for the reference while amines were on the upswing for the stored shrimp.

The overall maximum instability of this food occurred at a moisture content of 10.1% which corresponds to equilibration at 59.5% relative humidity. There was a maximum amount of amines formed which neutralized HCl, reacted with nitrous acid and formalin. The presence of these amines are in evidence when the shrimp was digested with pancreatin. At 10.1% water the shrimp slurry had the highest pH and required the least amount of alkali to adjust the slurry to 8.5. It was digested less readily than the reference and other samples as is seen by the amount of alkali required after digestion to raise the pH to 8.5 and had the lowest amino nitrogen content on formol titration. At 18 weeks this same trend holds.

3.4.2.2 37.8°C Storage (Table 33)

Only the data for 3 weeks storage seems to be of significance since at the other 2 storage intervals the shrimp had deteriorated too far. Maximum amine production occurred at 10.1% moisture. This is also borne out upon digestion with pancreatin where the pH of the aqueous slurry was 7.7 and only required 11.6 cc of 1N NaOH to raise the pH to 8.5 prior to the addition of pancreatin.

3.4.3 Optimum Moisture Level

The shrimp stored at the monolayer moisture level seems to be the most stable. The moisture content was more constant indicating that hydrolytic reactions and chemical reactions which form water are less.

3.5 Apples

3.5.1 Experimental

3.5.1.1 Moisture Levels

The apples were trayed and placed in a humidity cabinet which was saturated with water vapor and equipped with an efficient

TABLE 33

WILSON'S FULLY COOKED FRE

37.8°C	Initial % Moisture	Taste Test	Steam Distillate Mgs Amine Net/100 gms Anhyd. Shrimp: Amines which Neut. Hcl	Amines React with HNO_2	Amines React Formalin	Pancreatic Di Anhydrou: pH Slurry	CCN NaOH to pH 8.5
						3 Weeks October 2, 1964	
Reference	1.6	7.4	43.1	9.6	71.9	7.4	15.4
4.8% R.H.	1.6	7.7	24.0	33.6	81.5	7.4	13.8
28.5% R.H.	5.4	6.3	81.5	28.7	62.3	7.7	16.0
35.5% R.H.	6.6	6.5	52.7	67.1	57.5	7.5	14.4
59.5% R.H.	10.1	6.5	86.3	57.5	81.5	7.7	11.6
78.5% R.H.	18.0	3.9	52.7	47.9	91.1	7.5	14.4
						7 Weeks October 30, 196	
Reference			33.6	45	39.2	7.45	15.6
4.8% R.H.		Bad Odor	16.8	34	33.6	7.35	17.4
28.5% R.H.		Bad Odor	61.6	56	61.6	7.43	17.8
35.5% R.H.		Bad Odor	100.8	78	39.2	7.65	12.4
59.5% R.H.		Bad Odor	78.4	67	61.6	7.5	14.2
78.5% R.H.		Bad Odor	56.0	50	67.2	7.35	16.6
						11 Weeks November 27, 1	
Reference	1.7		16.8	11.2	41.4	7.45	14.2
4.8% R.H.	3.1	Bad Odor	33.6	-5.6	39.2	7.45	14.0
28.5% R.H.	4.7	Bad Odor	72.8	72.8	84.0	7.45	14.2
35.5% R.H.	5.3	Bad Odor	50.4	5.6	61.6	7.65	12.6
59.5% R.H.	7.1	Bad Odor	56.0	67.2	77.2	7.6	11.8
78.5% R.H.	13.0	Bad Odor	61.6	39.2	89.6	7.45	14.4

Date of Storage: September 11, 1964

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ABLE 33

OOKED FREEZE DRIED SHRIMP

creatic Digestion/100 gms Anhydrous Shrimp			Pepsin Digestion		Permanganate Oxidation NO.
CCN NaOH to pH 8.5	N NaOH Prior to Formalin	Gms Amine Nit.	N NaOH Prior to Formalin	Gms Amine Nit.	No.
er 2, 1964					
15.4	106	1.83	113	1.68	40
13.8	109	1.82	146	1.49	10
16.0	107	2.09	141	1.59	0
14.4	108	1.93	136	1.53	30
11.6	104	1.92	136	1.60	30
14.4	115	1.94	154	1.64	10
er 30, 1964					
15.6	94	1.48	86	1.48	
17.4	97	1.52	96	1.52	
17.8	95	1.52	90	1.52	
12.4	92	1.61	90	1.61	
14.2	89	1.57	93	1.57	
16.6	105	1.61	106	1.61	
mber 27, 1964					
14.2	141	3.09	153	1.29	
14.0	135	3.09	144	1.434	
14.2	141	2.91	154	1.344	
12.6	148	2.73	164	1.344	
11.8	145	2.98	151	1.344	
14.4	146	2.93	161	1.478	

B

fan. The apples as received had a moisture content of 2.0%. Upon removal from the humidity chamber the apples were equilibriated overnight in a polyethylene bag before being placed in storage. The apples were stored in glass jars with the lids taped so that water or air could not leave or enter.

3.5.1.2 <u>Analytical Procedures</u>

<u>Extraction with 63% Ethyl Alcohol</u>

Twenty grams of apples on an anhydrous basis were soaked in 100 cc of water for 1 hour. One hundred cc of 95% alcohol was added and the mixture osterized for 1 minute at high speed. The suspension was poured on a 15 cm sheet of No. 40 filter paper and the residue washed with 4-25 cc aliquots of 95% alcohol. The filtrate was adjusted to a volume of 300 cc.

The residue was washed from the filter paper with a wash bottle and a formol titration performed. For each 20 cc of slurry 1 cc of formalin was added.

Similarly a 25 cc aliquot of the filtrate was diluted with 25 cc of water and a formol titration executed.

The Folin Wu method for blood glucose was used for the determination of sugar.

An aliquot of the filtrate was used for absorption at 390 and 420 mμ with the Spectronic 20 and a Beckman DU Spectrophotometer for carbonyl absorption at 280 mμ.

3.5.2 Results and Discussion (Tables 34 and 35)

At both temperatures none of the samples failed the taste test. Maximum browning occurred at the monolayer moisture level where loss of free acids, sugar, amino nitrogen and absorption at 280, 390 and 420 mu was maximum. According to Table 32 apples are very low in protein, fat, ash, phosphorus and iron which probably accounts for why they did not spoil. The browning of a food apparently is not detrimental to the taste. The apples were stored at a high enough moisture content for spoilage to take place.

The apples at an initial moisture content of 2.0 increased in water which was typical of the other foods studied. The apples stored at an initial moisture content of 6.6 and 11.2% were quite stable as regards moisture change. Reactions which lead to formation and those which use up water must have balanced each other out or at the monolayer these reactions are at a minimum. The lipids of the sweet potatoes and beef were more stable to hydrolysis at the monolayer. When the beef, shrimp and nonfat dry milk solids spoiled it was the samples stored at a moisture content corresponding to equilibration at 61% relative humidity. The apples at this moisture content increased in water 4.0% at 25°C and 2.0% at 37.8°C. Then at the next moisture level, 19.1%, the water decreased 3.1% at 25°C and 4.6% at 37.8°C.

3.5.3 Optimum Moisture Content

Even though the apples browned the most at the monolayer moisture level this probably is the preferred level for storage because the food seems to be more stable in regard to moisture change.

TABLE 34

FREEZE DRIED APPLES WE[ED

Extraction with 63% [...]tio

25°C	% Moisture	Taste Test	Residue/100 gms pH Slurry	Residue/100 gms IN NaOH to pH 8.5	Residue/100 gms Mgs Amine Nit.	Filtrate/100 gms pH Slurry	Filtrate/100 gms IN NaOH to pH 8.5	Filtrate/100 gms Mgs Amine Nit.	[...]rat IN N to pH 8
						7 Weeks	January 26, 196[5]		Janu
Reference		4.5		4.75	7.0		105.0		10[9]
11.5% R.H.		4.4		4.4	3.3		106.3		10[6]
33.5% R.H.		5.1		4.6	4.3		98.8		98
49.2% R.H.		5.2		4.5	3.5		93.8		9[1]
61.8% R.H.		5.1		4.0	4.3		95.0		9[5]
67.3% R.H.		4.8		5.3	3.5		101.3		10[1]
						12 Weeks	March 2, 1965		Mar
Reference	2.6	5.0	4.9	4.1	4.2	3.82	107.5	43.8	10[7]
11.5% R.H.	3.6	4.7	4.8	4.1	2.8	3.7	105.4	29.8	10[5]
33.5% R.H.	6.9	5.3	4.9	3.8	6.3	3.9	99.7	43.8	9[9]
49.2% R.H.	10.4	4.8	4.9	4.3	3.5	3.9	93.4	30.6	9[1]
61.8% R.H.	18.7	5.4	4.75	4.3	3.5	3.9	94.7	26.3	9[4]
67.3% R.H.	15.8	5.6	4.8	5.0	4.2	3.85	101.6	33.1	10[1]
						16 Weeks	March 30, 1965		Mar
Reference	3.2	5.1	4.9	3.5	4.6	3.82	110.0	52.8	11[0]
11.5% R.H.	4.8	4.9	5.1	3.8	2.8	3.9	104.4	44.8	10[4]
33.5% R.H.	6.8	5.3	5.05	3.6	3.5	3.95	94.7	38.9	9[4]
49.2% R.H.	11.3	5.4	4.75	3.5	3.5	3.9	91.4	29.1	9[1]
61.8% R.H.	20.1	5.8	4.4	4.5	3.5	3.8	94.7	31.3	9[4]
67.3% R.H.	16.0	5.6	4.5	4.4	2.8	3.8	99.7	42.1	9[9]

Placed in Storage: December 8, 1964

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34

3 WEALTHY VARIETY

1 63% Ethanol

gms Mgs Amine Nit.	1/625 280 Mμ	1/625 390 Mμ	1/62.5 420 Mμ	% Sugar as Dextrose
5, 1965				
	.045	.14	.11	46.2
	.092	.145	.1	48.7
	.160	.165	.11	48.7
	.195	.205	.135	47.5
	.145	.16	.11	46.2
	.210	.175	.12	50.0
1965				
43.8	.088	.125	.095	48.4
29.8	.105	.122	.09	49.3
43.8	.212	.21	.14	48.3
30.6	.230	.22	.148	49.1
26.3	.177	.143	.105	51.0
33.1	.230	.236	.165	54.7
1965				
52.8	.067	.14	.105	51.6
44.8	.118	.138	.098	49.5
38.9	.187	.198	.13	51.6
29.1	.218	.205	.15	51.0
31.3	.180	.152	.112	52.6
42.1	.194	.16	.115	54.2

B

TABLE 35

FREEZE DRIED APPLES WE[illegible]

Extraction with 63% [illegible]

37.8°C	Initial % H_2O	Taste Test	Residue/100 gms pH Slurry	Residue/100 gms IN NaOH to pH 8.5	Residue/100 gms Mgs Amine Nit.	Filtrate/100 gms pH Slurry	Filtrate/100 gms IN NaOH to pH 8.5	Filtrate/100 gms Mgs Amine Nit.
						3 Weeks	December 29, 19[illegible]	
Reference	2.0	3.7	4.75	3.25			107.5	
11.5% R.H.	2.0	3.7	4.75	3.25			103.8	
33.5% R.H.	6.6	4.0	4.7	3.63			97.5	
49.2% R.H.	11.2	4.4	4.9	4.0			92.3	
61.8% R.H.	16.1	5.2	4.45	4.25			95.0	
67.3% R.H.	19.1	5.1	4.6	4.25			100.0	
						7 Weeks	January 26, 196[illegible]	
Reference		5.0	4.4	6.13	5.3		102.5	
11.5% R.H.		4.8	4.4	5.88	5.3		92.5	
33.5% R.H.		5.9	4.75	5.0	5.3		85.0	
49.2% R.H.		4.2	4.6	5.88	3.5		85.0	
61.8% R.H.		4.3	4.3	6.5	3.5		90.0	
67.3% R.H.		4.3	4.4	5.63	3.5		91.3	
						12 Weeks	March 2, 1965	
Reference	1.9	4.7	4.9	4.1	4.2	3.82	107.5	
11.5% R.H.	2.9	4.7	5.1	3.8	3.5	3.9	92.2	
33.5% R.H.	5.6	5.1	5.05	4.5	3.5	3.95	82.8	
49.2% R.H.	10.2	4.4	4.75	5.7	0.7	3.9	85.3	
61.8% R.H.	18.1	4.5	4.4	6.4	3.5	3.8	91.4	
67.3% R.H.	14.5	3.9	4.5	6.4	3.5	3.8	93.1	

Placed in Storage: December 8, 1964

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TABLE 35

D APPLES WEALTHY VARIETY

ion with 63% Ethanol

ate/100 gms		1/625	1/625	1/625	
NaOH o 8.5	Mgs Amine Nit.	280 Mμ	390 Mμ	420 Mμ	% Sugar as Dextrose
ember 29, 1964					
7.5		.122	.135	.09	57.0
3.8		.17	.14	.10	52.3
7.5		.28	.21	.135	55.5
2.3		.305	.24	.18	55.5
5.0		.24	.185	.135	58.0
0.0		.275	.21	.15	58.0
uary 26, 1965					
2.5		.163	.155	.11	49.5
2.5		.275	.167	.115	52.0
5.0		.475	.31	.215	50.8
.0		.463	.37	.205	52.0
.0		.400	.25	.17	59.0
.3		.443	.31	.22	58.0
rch 2, 1965					
.5		.088	.125	.095	48.4
.2		.365	.244	.162	47.7
.8		.468	.41	.29	48.6
.3		.630	.49	.35	54.0
.4		.610	.44	.295	56.8
.1		.650	.48	.32	59.3

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3.6. White Potatoes

3.6.1. Experimental

Moisture Levels

The potatoes as received had a moisture content of 1.2% and were used for one of the storage levels. Traying and holding the potatoes in a humidity cabinet saturated with water vapor raised the moisture as follows: 1 hour, 4.3%; 1 1/2 hours, 5.7%; 2 hours, 6.7%; and 3 hours, 7.7%. The potatoes were then equilibrated overnight in a polyethylene bag, placed in glass jars and the caps taped shut.

3.6.2. Analytical Procedures

Extraction with 71% Alcohol

Sufficient potatoes were ground with a mortar and pestle to give 5 gms of anhydrous food. The potatoes were soaked in 25 cc of water for 30 minutes. Seventy-five cc of 95% alcohol was added, the slurry stirred for 15 minutes with a magnetic stirrer and then allowed to settle for 10 minutes. The supernatant was decanted on to a sheet of Whatman No. 40 filter paper. The potatoes were extracted 3 additional times with 30 cc of 71% alcohol.

Formol Titration of the Residue

The residue was washed from the filter paper with a wash bottle. The pH was raised to 8.5 with 0.1N NaOH and before the addition of the formalin 1 gtt of alkali wouldhold the pH at or above 8.5 for 1 minute. The formalin was allowed to react for 5 minutes and the pH again brought to 8.5 by the same procedure.

Formol Titration of Filtrate

One hundred cc of filtrate was diluted with 100 cc of water. Ten cc of formalin was used. The reason the pH was so much lower than the residue was because alcohol depresses the pH.

3.6.3 Results and Discussion (Tables 36 and 37)

The potatoes did not deteriorate. Very little, if any, change in moisture occurred.

3.7 Sweet Potatoes

3.7.1 Experimental

3.7.1.1 Method for Obtaining the Five Moisture Levels

The sweet potatoes (moisture content 2.3%) were trayed and placed in a humidity cabinet where the temperature was 90°F and the relative humidity 80%. This food took up water very rapidly. The following periods of time in the humidity cabinet increased the moisture content as follows: 10 minutes, 3.15%; 30 minutes, 6.0%; 1 hour, 7.8%; 2 1/2 hours, 11.5%.

3.7.1.2 Analytical Procedures

Extraction and Determination of B-Carotene (21)

Sufficient sweet potatoes were ground with a mortar and pestle so as to yield 5 grams of anhydrous food. The water was removed by holding the pulverized sweet potatoes in a vacuum oven overnight at 40°C and 27 inches of vacuum. With a magnetic stirrer the potatoes were mixed with 50 cc of a acetona-hexane solution (3+7) for 15 minutes and then allowed to settle for 10 minutes. The supernatant was decanted on to No. 40 filter

Freeze Dried White Potatoes — Date of Storage: December 29, 1964

Extraction with 70% Ethanol

25^{o} C.	% H_2O	Taste Test	Residue/100 gms. pH Slurry	Residue/100 gms. .N NaOH to pH 8.5	Residue/100 gms. Mgs. Amine N	Filtrate/100 gms. pH Slurry	Filtrate/100 gms. 1N NaOH to pH 8.5
						5 Weeks	February
Reference		6.7		5.0	88		11.6
2.0% R.H.		6.7		5.6	90		10.0
12.0% R.H.		7.7		54	83		10.8
21.5% R.H.		6.2		5.5	85		10.8
27.0% R.H.		7.0		5.5	81		11.6
34.0% R.H.		6.1		5.9	90		10.8
						11 Weeks	March 1
Reference	2.3	7.1	7.2	3.0	51	6.4	11.8
2.0% R.H.	1,7	6.5	7.15	3.0	46	6.3	12.0
12.0% R.H.	4.5	5.5	7.1	3.1	50	6.3	13.5
21.5% R.H.	5.9	7.2	7.15	2.9	52	6.3	11.7
27.0% R.H.	6.9	6.9	7.15	3.2	55	6.3	12.3
34.0%	8.1	6.9	7.15	3.3	53	6.3	12.0
						15 Weeks	April 1
Reference	1.6	6.1	7.15	3.2	50	6.4	12.0
2.0% R.H.	1.5	5.8	7.15	3.2	48	6.3	12.3
12.0% R.H.	4.3	5.8	7.2	3.3	47	6.3	13.0
21.5% R.H.	6.0	6.1	7.25	3.1	47	6.3	11.7
27.0% R.H.	6.6	5.7	7.25	3.5	48	6.3	12.1
34.0% R.H.	7.7	7.7	7.3	2.9	50	6.3	12.6

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...ms. ...H ...	Mgs. Amine N	Per 100 gms. Anhyd. Pot. Total in NaOH to pH 8.5	Total Mgs. Amine N	% Sugar as Dextrose	1/400 280 mμ	1/40 390 mμ	1/40 420 mμ
...ary 2, 1965							
5	151	16.6	239	.37	.193	.05	.024
)	151	15.6	241	.34	.178	.042	.02
3	157	16.2	240	.37	.180	.048	.02
3	157	16.3	242	.36	.173	.045	.02
5	157	17.1	238	.39	,175	.048	.022
3	151	16.7	241	.37	.170	.05	.028
...ch 16, 1965					1/500	1/50	1/50
3	199	14,8	250	,28	.155	.05	.022
)	185	15.0	231	.33	.044	.022	.058
5	193	16.6	243	.27	.05	.028	.048
7	185	14.6	237	.34	.042	.023	.06
3	186	15.5	241	.27	.045	.023	.048
)	193	15.3	246	.34	.048	.025	.06
...il 13, 1965					1/400	1/40	1/40
)	189	15.2	239	.29	.217	.052	.03
3	187	15.7	235	.31	.087	.049	.025
)	193	16.3	240	.31	.080	.052	.028
7	192	14.8	239	.28	.075	.052	.03
1	183	15.6	231	.28	.052	.055	.03
5	188	15.5	238	.29	.048	.062	.033

B

FREEZE DRIED WHITE POTATOES　　　　DATE OF STORAGE: DECEMBER 29, 1964　2

EXTRACTION WITH 70% ETHANOL

			Residue /100 gms.			Filtrate /100 ga	
37.8°C	Initial % H_2O	Taste Test	pH Slurry	.N NaOH to pH 8.5	Mgs. Amine N	pH Slurry	In NaO to pH 8.5
						3 Weeks	Jan
Reference	1.2	7.3		4.0	67		10.4
2.0% R.H.	1.2	8.0		5.3	77		9.6
12.0% R.H.	4.3	7.2		4.3	66		10.8
21.5% R.H.	5.7	7.4		4.6	62		10.0
27.0% R.H.	6.7	7.7		5.3	83		9.6
34.0% R.H.	7.7	7.7		5.6	83		10.4
						7 Weeks	Feb
Reference		7.5	7.05	3.8	64	6.3	9.6
2.0% R.H.		7.3	6.95	4.3	63	6.25	9.6
12.0% R.H.		6.8	6.9	4.6	67	6.2	9.6
21.5% R.H.		7.0	7.0	4.2	62	6.2	9.8
27.0% R.H.		6.1	7.0	4.1	50	6.2	10.0
34.0% R.H.		6.5	6.9	4.6	69	6.2	10.8
						11 Weeks	Ma
Reference	1.5	7.1	7.2	3.8	43	6.4	11.4
2.0% R.H.	1.9	7.2	7.15	4.0	59	6.3	11.4
12.0% R.H.	4.4	7.1	7.1	3.5	57	6.3	13.2
21.5% R.H.	5.8	7.1	7.15	3.6	53	6.3	12.5
27.0% R.H.	6.5	7.1	7.15	4.3	56	6.3	11.4
34% R.H.	7.5	6.9	7.15	4.1	56	6.3	12.2

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TABLE #37

...0 GMS. ...AOH ...5	Mgs. Amine N	Per 100 gms. Anhyd. Pot. Total in NaOH to pH 8.5	Total Mgs. Amine N	% Sugar as Dextrose	1/400 280 mμ	1/40 390 mμ	1/40 420 mμ
...anuary 19, 1965							
	185	14.4	252	.42	.203	.048	.028
	157	14.9	234	.42	.170	.049	.020
	185	15.1	251	.38	.182	.048	..022
	174	14.6	236	.37	.175	.050	.028
	157	14.9	240	.37	.157	,052	.028
	157	16.0	240	.42	.157	.058	.032
...ebruary 16, 1965							
	174	13.4	238	.61	.33	.082	.042
	.74	13.9	238	.32	.33	.058	.028
	183	14.2	250	,32	.33	.05	.025
	179	14.0	241	.28	.33	.055	.028
	190	14.1	250	.30	.338	.06	.032
	174	15.4	233	.29	.337	.067	.034
March 16, 1965							
	189	15.2	242	.22	.223	.048	.025
	189	15.4	248	.23	.210	.043	.022
	185	16.7	242	.22	.200	.048	.02[illegible]
	181	16.1	234	.21	.195	.047	.023
	180	15.7	236	.21	.187	.05	.023
	189	16.3	235	.18	.185	.059	.03

B

paper and the filtrate collected in a 250 cc volumetric flask. Extraction was continued until the flask was filled.

The optical density of the extract was read by means of a Evelyn Spectronic 20 at 436 mu. The B-Carotene was calculated by means of the following equation:

$$\frac{(-\log T) \times V \times 100}{196 \times L \times C} = \text{mg. carotene}$$

per 100 gms. sample where (-log T) is the optical density, V the volume of extract, L the cell depth in cm., and C the weight of the original sample. The formula was checked with a solution of B-Carotene of known concentration and agreed closely.

Milligrams Fat Extracted

An aliquot of the extract was evaporated in the hood overnight, the last traces of solvent removed by placing in an oven at 110°C for 15 minutes and then weighed.

Extraction with 71% Ethanol

Enough food was weighed out to yield 10 grams of anhydrous material. Fifty cc of water was added, allowed to soak 30 minutes and then ground in a mortar. The potatoes with an initial water content of 11.5% were so tough and rubbery that they could not be dry ground effectively. One hundred fifty cc of 95% ethanol was added and the slurry allowed to soak for 2 hours with occasional agitation. The slurry was poured onto No. 1 filter paper and the residue washed with 2-50 cc aliquots of 71% ethanol. The final volume of the filtrate was 300 cc.

Formol Titration of Residue

The residue was washed from the filter paper with a wash bottle and a formol titration performed.

Formol Titration of Filtrate

A 200 cc aliquot of the filtrate was diluted with 200 cc of water and a formol titration executed using 20 cc of formalin.

Analysis of Sugar

The Folin Wu method was used on an aliquot of the filtrate.

3.7.2 Results and Discussion

3.7.2.1 37.8°C Storage (Table 38)

The taste panel rated the sweet potatoes satisfactory after 11 weeks of storage. The 11.5% moisture sample changed in texture. It became tough and rubbery. Prior to extraction with alcohol since it could not be dry ground satisfactorily with a mortar and pestle it was wet ground. Filtration of this sample though was extremely rapid. When the carotene was extracted, samples with a moisture content of 2.3 and 3.15% filtered extremely slowly and finally the 2.3% moisture sample completely clogged the pores of the filter paper. The samples above the monolayer value filteres rapidly. The reference was intermediate between these two extremes.

The 11.5% moisture sample differs from the reference in that less fat was extracted, there was a decrease in the amino nitrogen of the residue and the alcohol extract and the residue and extract required more alkali to raise the pH to 8.5. The

Freeze Dried Sweet Potatoes

Date of Storage: January 6, 1965 .965

.8° C	Initial & H_2O	Taste Test	Acetone-Hexare Extn./100 gms Mgs. B Carotene	Mgs. Fat Extd.	70% Ethi Residue /100 pH Slurry	N NaOH to pH 8.5	70% …due / N Na… T… pH 8…
					3 Weeks January 27		…anuar…
ference	2.3	5.3	35.7	480	7.85	.4	.4
.0% R.H.	2.3	5.9	5.3	440	7.85	.3	.3
.0% R.H.	3.15	6.3	7.6	469	7.8	.4	.4
.0% R.H.	6.0	6.8	10.2	432	7.8	.3	.3
.5% R.H.	7.8	6.3	11.4	488	7.6	.5	.5
.8% R.H.	11.5	6.9	7.1	373	7.5	.6	.6
					7 Weeks February 24		…ebrua…
ference		4.7	32.7	618	7.4	.7	.7
.0% R.H.		5.1	1.7	351	7.45	.5	.5
.0% R.H.		5,1	4.4	570	7.55	.5	.5
.0% R.H.		4.9	6.5	648	7.3	.8	.8
.5% R.H.		5.1	5.9	633	7.2	.9	.9
.8% R.H.		5.4	7.0	498	7.1	1.0	1.0
					11 Weeks March 24, 1		…arch
erence	0.9	4.8	28.0	408	7.6	.7	.7
0% R.H.	2.3	5.7	1.9	388	7.5	.6	.6
0% R.H.	2.7	5.2	2.7	353	7.35	.8	.8
0% R.H.	5.6	5.8	4.8	458	7.1	1.3	1.3
5% R.H.	7.1	5.8	5.0	410		1.2	1.2
8% R.H.	10.9	5.9	3.7	320	6.8	1.4	1.4

A

TABLE #38

NOL EXTRACTION

MS		EXTRACT /100 GMS				
MGS. AMINE N	pH SLURRY	N NAOH TO pH 8.5	MGS. AMINE N	TOTAL N NAOH TO pH8.5	TOTAL MGS. AMINE N	% SUGAR AS DEXTROSE
1965						
18	6.5	7.2	55	7.6	73	16.9
14	6.4	6.3	55	6.6	69	12.0
15	6.4	6.3	42	6.7	57	12.7
14	6.35	7,2	42	7.5	56	13.0
14	6.35	7.5	34	8.0	48	12.3
13	6.3	8.4	34	9.0	46	12.7
, 1965						
20	6.45	7.6	59	8.3	78	15.3
20	6.35	6.0	50	6.5	70	13.1
18	6.35	6.3	50	6.8	69	13.7
17	6.3	7.8	38	7.6	55	13.1
17	6.2	8.1	29	9.0	46	13.1
13	6.1	9.6	34	10.6	46	13.1
965						
23	6.45	7.4	61	8.1	84	17.0
20	6.3	6.3	51	6.9	71	14.2
19	6.2	6.8	48	7.6	67	13.8
17	6.15	7.3	34	8.6	50	14.0
20	6.2	8.9	32	10.1	52	14.1
19	6.0	10.4	27	11.8	46	14.1

B

titratable acidity of the alcohol extract of the two low moisture samples is lower than the references. The monolayer sample resembles the reference very closely as to the amount of fat extracted and titratable acidity of the alcohol extract.

Hydrolysis of lipids as phospholipids with the formation of fatty acids, phosphoric acid and bases as choline could account for these changes. The loss in water in samples stored at moisture levels above the monolayer value shows that hydrolytic reactions are occurring.

The decrease in the amount of fat extracted and amino nitrogen are very likely related to the changes in texture. Phospholipids are readily hydrolyzed by relatively mild acid or alkaline conditions. The hydrolytic products as phosphoric acid, fatty acids and bases such as choline could combine with the free amino groups of amino acids and protein. The unhydrolyzed phospholipid is also a very reactive molecule. The acetone-hexane extract should have been analyzed for phosphorus thereby assisting in finding out what is happening on storage of this food.

According to Table 39 when a foul odor and taste appeared in a food, the food was stored at a moisture content corresponding to equilibration at 61% relative humidity. The foods which did not spoil were either stored below this moisture level or according to Table 31 are low in lipid and phosphorus. The lipids are capable of playing an active part in the production of foul odors in foods.

Freeze Dried Sweet Potatoes

	Monolayer Values		37.8° C Storage: Putrid Odor and Bad Taste		37.8° C Storage: Maximum Browning	
	% H_2O	Rel. Humidity	% H_2O	Rel. Humidity	% H_2O	Rel. Humidity
n Fat Dry lk Solids	4.1	24.0	8.0	61.0	8.0	61.0
ef	3.5	28.0	7.8	61.0	8.0	61.0
nanas	9.5	50.0	None		14.1	62.5
ples	10.0	45.5	None		11.2	49.2
eet tatoes	4.2 6.2	25.5 46.0	None		11.3	59.8
rimp	5.1	28.5	10.1	59.5	10.1	59.5

A

25° C Storage

Table # 39

ium ing Rel. Humidity	% Moisture Change	Putrid Odor and Bad Taste % H_2O	Putrid Odor and Bad Taste Rel. Humidity	Maximum Browning % H_2O	Maximum Browning Rel. Humidity	Moisture Change	% Water as Rec'd.
61.0	-0	8.0	61.0	8.0	61.0	+0.2	5.8
61.0	-2.0	7.8	61.0	7.8	61.0	-0.5	5.5
62.5	-0.8	None		14.1	62.5	-.05	1.6
49.2	+2.0	None		11.2	49.2	+ 4.0	2.0
59.8	-0.6	None		11.3	59.8	-0.3	2.3
59.5	-3.0	10.1	59.5	10.1	59.5	-2.3	1.6

B

3.7.2.2 25°C Storage (Table 40)

The carotene rapidly disappeared with its loss not occurring quite as rapidly at the monolayer moisture level. All the color could not be extracted from the sweet potatoes by the acetone-hexane solution in the two highest moisture samples.

None of the samples changed very much in moisture on storage. Less fat was extracted from the sample with a moisture content of 11.5% and the amino nitrogen of the alcohol extract was about 40% lower than the reference. Interaction could have occurred between these constituents so that the fat was no longer soluble in the acetone-hexane solution. The titratable acidity of this sample was less than the reference while at 37.8°C it was greater than the reference. These lipids must be taking part in a multitude of chemical reactions which vary with the moisture content and temperature.

3.7.2.3 Optimum Moisture Content

The monolayer moisture level was the optimum level because the lipids were more stable.

3.8 Rice

3.8.1 Experimental

3.8.1.1 Moisture Levels (Tables 41,42)

The rice had a moisture content of 7.6% and was used as such for one of the storage conditions. The rice was reduced to a moisture content of 2.55% by holding it overnight in the vacuum oven at 50°C and 27 inches of vacuum. One and one half

FREEZE DRIED SWEET POTATOES   DATE OF STORAGE: JANUARY 6, 1965

70% ETH

25° C	% H_2O	TASTE TEST	ACETONE-HEXANE EXTN./ 100 GMS MGS B-CAROTENE	MGS FAT EXTD.	RESIDUE/ 100 GMS pH SLURRY	NNaOH TO pH 8.5	MGS AMINE N
							4 WE
REFERENCE		6.4	36.7	597	7.3	.9	24
10.0% R.H.		5.6	7.6	525	7.5	.5	20
17.0% R.H.		6.3	10.6	549	7.55	.5	17
40.0% R.H.		7.2	16.7	570	7.5	.6	20
47.5% R.H.		7.3	20.0		7.4	.6	20
59.8% R.H.		7.0	11.8	428	7.3	.8	18
							9 WE
REFERENCE		5.0	30.6	490	7.5	.8	22
10.0% R.H.		4.8	2.6	423	7.35	.8	21
17.0% R.H.		4.8	5.2	453	7.45	.8	21
40.0% R.H.		5.7	8.2	475	7.4	.9	21
47.5% R.H.		6.0	11.7	523	7.5	.6	20
59.8% R.H.		5.5	7.5	360	7.2	.9	21
							13 WE
REFERENCE	0.9	6.1	24.2	375	7.68	.68	19
10.0% R.H.	2.6	6.4	2.0	393	7.72	.48	19
17.0% R.H.	3.6	6.1	4.0	355	7.7	.54	17
40.0% R.H.	6.0	6.9	7.0	400	7.68	.58	17
47.5% R.H.	7.7	6.8	8.4	420	7.6	.56	17
59.8% R.H.	11.2	7.1	4.9	275	7.5	.74	18

A

1965

TABLE #40

% ETHANOL EXTRACTION

	Extract /100 gms.			Per 100 gms Anhyd. Pot.		
	pH Slurry	NNaOH to pH 8.5	Mgs Amine N	Total N NaOH to pH 8.5	Total Mgs Amine N	% Sugar as Dextrose
4 Weeks February 3, 1965						
	6.25	7.5	59	8.4	83	14.7
	6.15	6.0	59	6.5	79	12.0
	6,25	5.7	55	6.2	71	13.3
	6.2	6.0	46	6.6	66	12.3
	6.2	6.0	50	6.6	70	12.1
	6.05	6.9	46	7.7	64	12.0
9 Weeks March 10, 1965						
	6.35	7.5	63	8.3	85	15.5
	6.35	6.2	51	7.0	73	12.5
	6.3	6.2	53	7.0	73	12.0
	6.35	5.8	51	6.7	72	12.5
	6.4	6.1	55	6.7	75	12.4
	6.25	6.5	43	7.4	64	12.3
.3 Weeks April 7, 1965						
	6.4	7.3	64	8.0	83	16.2
	6.4	6.2	48	6.7	66	13.8
	6.33	6.1	50	6.6	68	13.9
	6.4	7.3	48	7.9	65	14.3
	6.4	6.2	61	6.8	78	14.3
	6.32	6.8	41	7.5	59	13.9

B

ENRICHED PRE-COOKED LONG GRAIN MINUTE RICE BRAND

DATE OF STORAGE: JANUARY 14, 19

ALCOHOL EXTRACT /100 GRA

25° C	% H_2O	TASTE TEST	pH SLURRY	INNaOH TO pH 8.5	MGS AMINE N	pH SLURRY	INNaOH TO pH 8.5
						7 WEEKS MARCH	
REFERENCE		8.0				5.4	.52
7.5% R.H.		7.7				5.45	.54
14.0% R.H.		7.8				5.5	.52
25.0% R.H.		7.8				5.6	.52
37.5% R.H.		8.1				5.55	.52
44.0% R.H.		8.0				5.45	.56
						11 WEEKS APRIL	
REFERENCE		7.9	6.3	1.3	19.9	5.35	.38
7.5% R.H.		8.2	6.15	1.5	22.1	5.25	.42
14.0% R.H.		7.3	6.1	1.47	23.5	5.3	.26
25.0% R.H.		8.3	6.2	1.35	19.6	5.4	.39
37.5% R.H.		8.0	6.05	1.51	19.7		
44.0% R.H.		8.1	6.25	1.23	14.8	5.35	.38
						15 WEEKS MAY 2	
REFERENCE	7.4	7.9	6.35	..88	23.4	5.5	.50
7.5 R.H.	1.9	7.6	6.3	1.1	19.3	5.6	.47
14.0%R.H.	4.1	7.7	6.18	.9	21.7	5.4	.50
25.0%R.H.	5.3	7.7	6.18	.97	24.1	5.6	.50
37.5%R.H.	7.0	7.4	6.25	1.08	28.2	5.4	.50
44.0%R.H.	7.7	7.7	6.25	.93	25.3	5.28	.55

A

, 1965

TABLE #41

GRAMS ANHYDROUS RICE

OH O .5	MGS. AMINE N	TOTAL IN NAOH TO PH 8.5	TOTAL MGS AMINE N	% SUGAR TO DEXTROSE	1/12.5 280 $m\mu$	1/12.5 420 $m\mu$
ARCH 3, 1965						
	2.2			.04	.198	.001
	5.6			.029	.210	.002
	4.6			.03	.207	.002
	2.3			.029	.215	.002
	2.8			.03	.213	.002
	3.4			.029	.215	.001
PRIL 4, 1965						
	.0	1.68	19.9	.054	.100	.002
	.09	1.92	22.2	.051	.100	.002
	0	1.73	23.5	.046	.097	.002
	.61	1.74	20.2	.043	.104	.002
			19.7	.043	.107	.002
	0	1.61	14.8	.041	.112	.002
AY 2, 1965						
	1.8	1.38	25.2	.28	.19	.002
	1.5	1,57	20.8	.3	.2	.002
	1.5	1.4	23.5	.26	.18	.002
	1.5	1.47	24.6	.22	.19	.002
	1.1	1,58	29.3	.32	.12	.002
	1.5	1,48	26.8	.24	.2	.002

B

ALCOHOL EXTRAC

ENRICHED PRE-COOKED LONG GRAIN WHITE MINUTE RICE BRAND — DATE OF STORAGE: JA

37.8°C	INITIAL % H_2O	TASTE TEST	RESIDUE FROM ETHANOL EXTRACTION /100 GRAMS: PH SLURRY	IN NAOH TO PH 8.5	MGS. AMINE N	PH SLURRY	INNAO TO PH 8.
						5 WEEKS	FE
REFERENCE	7.6	5.6				6.1	5.0
7.5% R.H.	2.55	6.0				6.1	6.25
14.0% R.H.	3.8	5.8				6.0	5.0
25.0% R.H.	5.5	5.5				5.95	.50
37.5% R.H.	6.9	5.1				6.05	5.0
44.0% R.H.	7.6	5.7				6.0	5.0
						9 WEEKS	MA
REFERENCE		7.4				5.45	.6
7.5% R.H.		7.1				5.5	.56
14.0% R.H.		7.4				5.4	.6
25.0% R.H.		7.5				5.5	.56
37.5% R.H.		7.3				5.6	.56
44.0% R.H.		7.4				5.6	.6
						13 WEEKS	AP
REFERENCE	7.4	7.8	6.5	.94	14.2	5.55	.36
7.5% R.H.	2.3	7.7	6.05	1.4	22.8	5.45	.36
14.0% R.H.	3.9	7.8	6.35	1.3	17.9	5.7	.36
25.0% R.H.	5.5	7.8	6.45	1.05	17.4	5.75	.34
37.5% R.H.	7.0	7.8	6.4	1.16	15.0	5.8	.32
44.0% R.H.	7.5	7.9	6.4	.99	16 2	5.45	.34

A

ACT / 100 GRAMS [illegible]YDROUS RICE

JANUARY 14, 1965 TABLE # 42

…OH … …8.5	MGS. AMINE N	TOTAL IN NAOH TO pH 8.5	TOTAL MGS AMINE N	% SUGAR AS DEXTROSE	1/12.5 280 mμ	1/12.5 420 mμ
FEBRUARY 18, 1965						
	1.8			.043	.168	.01
5	0			.044	.215	.01
	1.1			.044	.163	.008
	0			.05	.174	.008
	0			.051	.175	.01
	1.1			.046	.188	.008
MARCH 18, 1965						
	.4			.06	.173	0
5	.9			.05	.187	0
	.4			.06	.185	0
	.4			.064	.190	0
	0			.055	.185	0
	-			.06	.193	0
APRIL 15, 1965						
	.26	1.30	14.5	.033	.31	.01
	0.[illegible]	1.76	22.8	.030	.29	.01
	.18	1.66	18.1	.033	.28	.01
	.26	1.34	17.7	.036	.312	.012
	.44	1.48	15.4	.031	.312	.008
	.61	1.33	16.8	.031	.313	.01

B

hours in the freeze dryer at 100°F lowered the moisture to 5.5% and 6 hours under the same conditions gave 3.8% water.

3.8.1.2 Analytical Procedures

Extraction with Ethanol

Enough rice was weighed out to represent 20 grams of anhydrous food. It was ground to a fine powder and then wet ground in 50% alcohol adding the alcohol in small aliquots so that the optimum amount of fluid was present for grinding. The slurry was transferred to a 250 cc volumetric flask. After placing a funnel in the neck of the flask it was heated in a boiling water bath for 1 hour and then allowed to stand overnight. The next morning the slurry was diluted to 250 cc with 95% alcohol and allowed to settle. The supernatant was decanted on to a sheet of Whatman No. 1 filter paper and a sheet of What No. 40 filter paper. The residue was rinsed with 2-25 cc aliquots of 71% ethanol.

Formol Titration of the Residue

The ground rice was washed from the filter paper and 250 cc volumetric flask with a wash bottle. With a magnetic stirrer and pH meter, the pH was raised to 8.5 with 0.1N NaOH and when 1 gtt of alkali maintained the pH above 8.5 for 1 minute the formalin was added (1 cc for each 20 cc of slurry). The formalin was allowed to react for 5 minutes and then the pH was returned to 8.5.

Formol Titration of the Filtrate

Two hundred cc of the filtrate was diluted with 200 cc of water and a formol titration performed.

3.8.2 Results and Discussion

This food apparently did not have any tendency to deteriorate. Little, if any, change in moisture occurred.

3.9 Nonfat Dry Milk Solids

3.9.1 Experimental

3.9.1.1 Methods for Attaining the Five Moisture Levels

The milk powder as received contained 5.8% water and was used as such for one storage condition. The lowest moisture level, 1.8%, was achieved by holding the milk powder overnight in a vacuum oven at 65°C. Heating the milk powder in a vacuum oven for 2 hours at 50°C gave the 3.2% moisture level. Holding the milk powder in the vacuum oven overnight at 25°C without any airflow lowered the moisture to 4.5%. The moisture was increased to 8.0% by placing the milk powder for 1 hour in a humidity cabinet saturated with water vapor and equipped with a good fan.

3.9.1.2 Analytical Methods

Precipitation of the Casein

Five grams of milk powder (anhydrous basis) was dissolved in 100 cc of water and allowed to soak for 30 minutes. With a magnetic stirrer-pH combination the pH was lowered to 4.7 by slowly adding 0.1N HCl. The mixture was held at 37.8°C for 1 hour and then the supernatant decanted on to Whatman No. 40 filter paper. By means of a magnetic stirrer the casein was washed for 10 minutes with 2-25 cc aliquots of water.

Formol Titration of the Casein

The casein was washed from the filter paper by means of a wash bottle. Sufficient alkali was added to dissolve the casein which with agitation required 30 minutes. The pH was then increased to 8.5. As the end point was approached, after adding a couple drops of 0.1N NaOH, a waiting period of 30 seconds was instituted so that the operator was certain that the needle of the pH meter was stable at pH 8.5. Ten cc of formalin was added and the pH returned to 8.5 in the same manner.

Formol Titration of the Filtrate and Percent Sugar as Dextrose

The filtrate was adjusted to a volume of 200 cc. One half cc was diluted to 20 cc with water and 1 cc used for the determination of sugar by the Folin - Wu method. A formol titration was performed on the remainder of the filtrate.

3.9.2 Results and Discussion

3.9.2.1 25°C Storage (Table 43)

The only sample rated down by the taste panel was the one containing 8% water. It didn't brown, but had a bad flavor and odor. The off flavor was picked up at 5 weeks and was not enhanced on further storage. This sample had a reduction in amino nitrogen of the casein, an increase in amino nitrogen of the filtrate and a fall in percent sugar as dextrose. These data might be interpretated as a gradual oxidative disintegration of milk proteins catalyzed by metal ions with water soluble fragments resulting.

BORDEN'S STARLAC INSTANT NONFAT DRY MILK SOLIDS PLACED IN STO

			Precipitation of Casein		Formal Titration of Casein		Formal Titratio of Filtr
5°C	% H_2O	Taste Test	pH Slurry	NI' Cl to pH4.7	N NaOH to pH 8.5	Mgs Amine N	NNaOH to pH 8.5
							5 Weeks Feb
EFERENCE		5.9	7.0	68.2	29.0	221	47.6
7.0% R.H.		5.8	7.0	66.4	28.8	213	46.4
6.0% R.H.		5.0	7.0	66.4	28.8	227	46.4
8.0% R.H.		6.2	6.95	67.6	29.2	218	47.2
5.0% R.H.		5.3	6.95	67.6	29.2	227	47.2
1.0% R.H.		3.8	7.0	66.8	28.6	216	46.8
							9 Weeks March
EFERENCE		5.8	6.7	65.4	28.9	244	46.3
7.0% R.H.		5.1	6.68	65.1	29.4	229	46.9
6.0% R.H.		5.8	6.7	65.7	29.4	242	46.3
8.0% R.H.		6.4	6.68	66.0	29.3	237	46.8
5.0% R.H.		5.9	6.7	66.6	29.5	232	46.9
1.0% R.H.		4.0	6.72	65.6	29.5	232	46.8
							13 Weeks Apri
EFERENCE	5.6	6.3	6.71	66.4	29.9	239	46.2
7.0% R.H.	2.7	6.3	6.7	66.7	31.0	240	47.2
6.0% R.H.	4.2	6.2	6.73	66.9	30.6	242	46.4
8.0% R.H.	4.3	6.3	6.7	67.2	30.7	243	47.1
5.0% R.H.	5.2	6.3	6.72	66.8	30.4	237	46.8
1.0% R.H.	8.2	4.3	6.68	65.6	29.9	235	46.0

PLACED IN STORAGE: JANUARY 21, 1965 TABLE #43

Formal Titration of Casein: N NaOH to pH 8.5	Formal Titration of Casein: Mgs Amine N	Formal Titration of Filtrate: N NaOH to pH 8.5	Formal Titration of Filtrate: Mgs Amine N	Mgs Total Amine N	% Sugar as Dextrose
5 Weeks February 25, 1965					
29.0	221	47.6	39	260	30.8
28.8	213	46.4	39	252	23.4
28.8	227	46.4	34	261	43.8
29.2	218	47.2	45	263	34.3
29.2	227	47.2	34	261	45.3
28.6	216	46.8	45	261	23.2
9 Weeks March 25, 1965					
28.9	244	46.3	41	285	29.7
29.4	229	46.9	50	280	30.1
29.4	242	46.3	45	287	30.8
29.3	237	46.8	59	297	30.8
29.5	232	46.9	44	276	31.6
29.5	232	46.8	48	270	30.8
13 Weeks April 22, 1965					
29.9	239	46.2	53	292	24.4
31.0	240	47.2	44	284	24.4
30.6	242	46.4	50	292	24.4
30.7	243	47.1	50	293	24.7
30.4	237	46.8	49	286	24.4
29.9	235	46.0	47	282	21.4

B

Moisture increased in the 1.8 and 3.2% storage levels. This is typical and presumably arises from interactions of sugars and phospholipids with amino acids and proteins.

3.9.2.2 37.8°C Storage (Table 44)

The 8.0% moisture milk powder failed the taste test after 3 weeks storage. The pH value of the reconstituted milk dropped, the sugar concentration decreased and there was a reduction in water soluble and casein amino nitrogen. The decline in pH could be due to sugar reacting with the amino groups of the casein making the molecule more acidic. Free amino acids and sugars taking part in the browning reaction account for their reduction.

Upon reconstitution of the milk powder with water the milk particles did not stay suspended but settled resulting,in a clear brown layer. Reactions of compounds taking part in the browning reaction probably had a part in this phenomenon. Also the unfolding and unwinding of milk proteins, lipoproteins and the hydrolysis of lipids had a part.

The decline in flavor cannot with certainty be attributable to the browning reactions. Potato granules have been placed in storage with the aid of a preservative,which browned badly but did not deteriorate in taste. The bad flavor and odor probably resulted from the hydrolysis of the small amount of lipoprotein present.

Borden's Starlac Instant Nonfat Dry Milk Solids | Placed in Storage: Jan

37.8°C	Initial % H_2O	Taste Test	pH Slurry	Precipitation of Casein: N HCl to pH 4.7	Formal Titration of Casein: N NaOH to pH 8.5	Formal Titration of Casein: Mgs Amine N	Fo Ti Fi: N N T pH
							3 Wee
Reference	5.8	5.7	7.0	67.4	29.2	227	46
7.0% R.H.	1.8	5.5	6.9	66.8	29.6	221	47
16.0% R.H.	3.2	5.5	6.95	66.0	28.4	216	45
28.0% R.H.	4.5	5.6	6.9	71.6	29.2	227	47
45.0% R.H.	5.8	6.0	6.95	68.0	29.2	227	47
61.0% R.H.	8.0	2.8	6.85	66.4	29.2	227	48
							7 Wee
Reference		4.1	6.7	64.9	29.5	243	45
7.0% R.H.		5.1	6.65	63.8	29.6	231	45
16.0% R.H.		4.9	6.75	64.6	29.7	239	45
28.0% R.H.		5.4	6.72	66.0	30.4	248	46
45.0% R.H.		4.5	6.7	66.6	30.0	242	47
61.0% R.H.	Brown and Bad Rej.	Flavor	6.5	62.4	30.0	210	49
							11 Weel
Reference	4.8	6.9	6.7	64.0	28.9	240	46
7.0% R.H.	1.3	6.0	6.62	64.1	29.5	223	47
16.0% R.H.	3.5	6.7	6.7	66.5	30.8	240	46
28.0% R.H.	3.8	6.1	6.7	64.0	29.7	237	46
45.0% R.H.	3.9	6.2	6.7	63.7	28.7	232	46
61.0% R.H.	7.0	Brown and Putrid Rej. Odor	6.35	58.9	30.2	198	46

A

ACED IN STORAGE: JANUARY 21, 1965

TABLE #44

L TION SEIN H 5	Mgs Amine N	Formal Titration of Fihrate: N NaOH to pH 8.5	Formal Titration of Fihrate: Mgs Amine N	Total Mgs Amine N	% Sugar as Dextrose
		3 Weeks February 11, 1965			
	227	46	39	266	26.2
	221	47	34	255	26.9
	216	45	39	255	25.6
	227	47	28	255	26.5
	227	47	39	266	26.5
	227	48	34	261	25.2
		7 Weeks March 11, 1965			
	243	45	43	286	26.5
	231	45	43	274	25.9
	239	45	41	280	25.7
	248	46	43	291	26.9
	242	47	43	287	25.7
	210	49	41	251	25.2
		11 Weeks April 8, 1965			
	240	46	45	285	27.6
	223	47	47	270	28.0
	240	46	45	285	24.1
	237	46	47	284	27.4
	232	46	47	279	28.4
	198	46	41	239	26.0

B

On further storage the 8.0% moisture powder had acquire such a putrid odor and had browned so badly that the taste panel was not exposed to it. There was also a continued fall in pH of the milk, in casein amino nitrogen, in the amino nitrogen of the filtrate and % of sugar as dextrose.

At both storage temperatures at the 1.8% moisture level the amino nitrogen of the casein is lower than the reference. This reduction is thought to be due to reactions of polar groups which occurred when the milk powder was held overnight at 63°C in order to reduce the moisture to this low a level.

Further analytical work on milk powder would be to study the changes that occur in the lipoproteins on storage. For example the phospholipids could be extracted by means of the Roese-Gottlieb Method Changes in the amount extracted, an increase or decrease in phosphorus or presence of cleavage products might be of assistance in finding out where the bad odor and flavor of the milk was coming from.

3.9.2.3 Optimum Moisture Level

The monolayer moisture level, 4.5%, appears to be the most stable on storage. The milk powder as purchased had a moisture content of 5.8%.

4. INTERRELATIONSHIPS BETWEEN THE SORPTION MEASUREMENTS AND STORAGE STABILITY

At the monolayer moisture level, generally speaking, the foods were more stable to moisture change and were less prone to take on bad odor and tastes. The polar groups are protected by a coating of water. Below the monolayer the polar groups react with the formation of water. Above the monolayer there is extra water available for deteriorative reactions to take place. These reactions are at a maximum at a moisture level corresponding to equilibration at 61% relative humidity.

In the determination of the moisture sorption isotherms surface areas in the neighborhood of 0.3 to 0.5 m^2/g were involved. When the foods were placed in storage surface areas a great deal larger were studies. If a 1 cm cube is subdivided into cubes with 10 mu edges, the surface area is increased from 6 sq. sm to 600 sq. m. The surface of silica gel is 560 m^2/g. Despite this tremendous difference in the areas involved (1400 times larger),there was found to be a definite relationship between the monolayer value of the foods and their storage stability.

Because the foods were stored and sealed under atmospheric conditions, oxygen may have been the most important factor in their deterioration. If Phase 1 were repeated with the foods stored in the absence of oxygen, would the bad odors and textural again occur? Would the changes in moisture observed again occur? All the oxygen adsorbed on and in the food and in the package would have to be removed. Such questions and more shall be answered upon completion of Phase 2 of this contract.

The precision of the analytical data varies. Such analyses as moisture, percent fat extracted, free fatty acids as oleic, peroxide number, hematin absorbance, thiobarbituric acid test, B-carotene, sugar, absorbance at 390 and 420 mu are accurate and readily reproducible. Data which necessitated the use of the pH meter as the formol titration of a protein residue and its extract, pH of a solution, titratable acidity and volatile amines are not as precise. The pH meter was left on 24 hours a day to minimize drift. Test Equipment said the variation was not the fault of the pH meter but was caused by small changes in the solution being examined such as, for example, small differences in temperature. They did not think a voltage regulator would help any. Absorbance at 280 mu was highly variable because the Beckman DU spectrometer did not function properly at times. This has now been corrected.

The reference foods showed more variation than they should have. They were stored in the freezer in quart glass jars and thus were opened a total of 6 times for the 6 storage intervals. At this time fresh air entered the jar along with a little water. In the future the reference shall be placed in small glass jars with just enough food for each storage interval.

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APPENDIX A

EXAMPLE CALCULATION OF THE FUGASSI ISOTHERM EQUATION VALUES

The following calculation is for moisture sorption onto milk at 30°C. The original data points, C and We, were read off the moisture sorption isotherm.

TABLE 25

VALUES FOR THE SOLUTION OF THE FUGASSI ISOTHERM EQUATION

Point No.	C	We (mg/g)	C/We$\times 10^3$	$\sum_1^3 Ci/iWe_i$	$\sum_1^3 C_i$	$\sum_1^3 C_i^2$	$\sum_4^6 C_i - \sum_1^3 C_i$	$\sum_4^6 C_i^2 - \sum_1^3 C_i^2$
1	0.1	22.0	4.545	18.090×10^{-3}	0.6	0.14	0.9	0.63
2	0.2	30.7	6.515					
3	0.3	42.7	7.030	B	F	J		
4	0.4	66.2	6.042	$\sum_4^6 Ci/We_i$	$\sum_4^6 C_i$	$\sum_4^6 C_i^2$	M	S
5	0.5	76.9	6.500	19.262×10^3	1.5	0.77	$\sum_7^9 C_i - \sum_4^6 C_i$	$\sum_7^9 C_i^2 - \sum_4^6 C_i^2$
6	0.6	89.3	6.720				0.85	1.08
7	0.7	119.0	5.880	D	G	K		
8	0.8	158.5	5.050	$\sum_7^9 Ci/We_i$	$\sum_7^9 C_i$	$\sum_7^9 C_i$		
9	0.85	197.5	4.310	15.240×10^{-3}	2.35	1.85		
							N	T
Po = 31.8 mm				E	H	L		

The letters in the boxes in Table 25 are merely for convenience in writing the following equations. The first three equations give the values which are used in the last three equations to calculate the Fugassi constants. No attempt has been made here to derive any of these equations, this is merely an illustration of a method of solving the Fugassi isotherm equation.

$$R = \frac{(D-B)\ (N) - (E-D)\ (M)}{(T)\ (M) - (S)\ (N)} = 9.410 \times 10^{-3}$$

$$Q = \frac{(D-B) + (R)\ (S)}{M} = 7.891 \times 10^{-3}$$

$$P = 1/3\ D - Q(G) + R(K) = 4.884 \times 10^{-3}$$

$$A = \frac{1}{P - R + Q} = 297.0 \text{ mg/g}$$

$$K = \frac{P - R + Q}{P + R} = 0.235$$

$$K_1 = \frac{R/P + 1}{P_o} = 0.092/\text{mm}$$