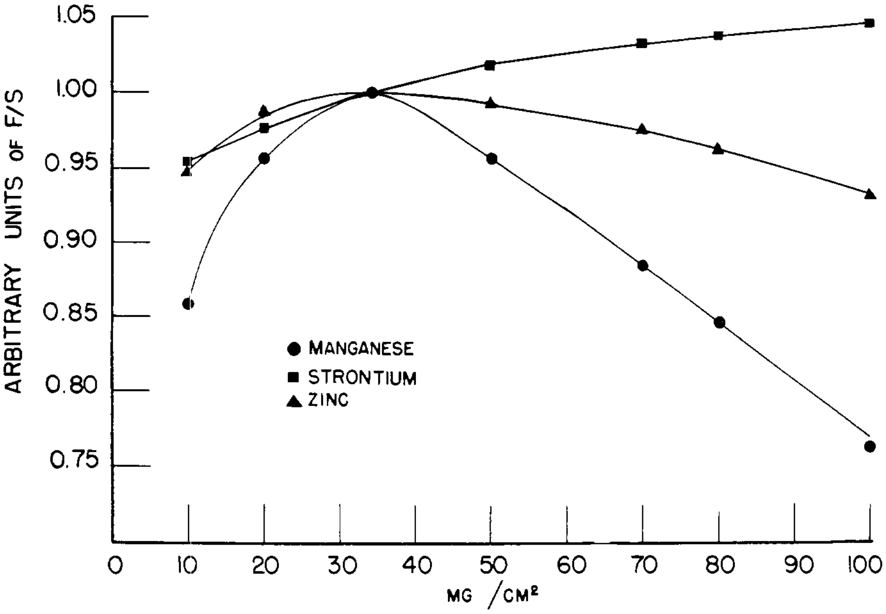

Figure 3. The ratios (in arbitrary units) of the $K\alpha$ radiation intensities to the scatter radiation intensities as a function of the weight of the sample

surface reflection phenomenon not affected by thickness. This hypothesis would explain the decrease in sensitivity with samples thinner than 35 mg. per sq. cm. To explain such a consistent point of inflection, such a surface reflection phenomenon would have to be rather independent of the variation in the nature of the sample.

On the basis of these, a 0.05-inch thick frame was chosen, which for most materials yields samples equivalent to 30 to 40 mg. per sq. cm. Figure 3 also indicates the range of sample weights over which the method is essentially constant permitting the use of a fixed frame thickness without weighing the sample.

This method appears to correct for many of the day-to-day variations in the instrument as well as the sample variables. However, a consistent program of daily reference standards is necessary to keep check on these instrument variables. For this purpose, aluminum alloy samples that give lines over the range of interest are recommended. The daily position of these lines on the goniometer scale and their intensity under standard conditions are logged and serve as a check on instrument stability.

The degree of deviation from the standard values which can be tolerated depends upon the accuracy desired. Generally the goniometer is not realigned until the peaks deviate about 0.1° 2θ. Correction factors are applied to the desired angles, however. Also realignment for intensity is not made unless deviations in excess of 5% of the standard rate are obtained. These same standards are used to realign to the theoretical angles and standard intensities. Any rugged standard material which will give lines in the area of interest that are measurable under the power settings required for the samples will suffice.

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FROZEN FOODS STORAGE EFFECTS

Effect of Storage Conditions on Nutrients in Frozen Green Beans, Peas, Orange Juice, and Strawberries

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PROPER QUICK FREEZING of foods is effective in preserving their nutritional value. To provide basic data on nutrients in commercially available frozen fruits, juices, and vegetables, an extensive survey was carried out on samples taken from regular production lines (*1*). Much information is available on the effects of maltreatment, during processing of frozen foods, on certain nutrients—particularly ascorbic acid (*6*). Although direct observations were not made in this regard, the nutrition survey data indicated that, in general, maltreatments which can cause serious loss of nutrients in harvesting and processing are avoided.

Frozen foods are known to remain well preserved at 0° F., but storage at higher temperatures can cause vitamin losses and marked effects on color and flavor, which results in lower quality at the retail level. Although surveys have suggested that less than 1% of frozen food products are subjected to serious temperature abuse during commercial handling (*5*), it is important to collect information on stability under various storage conditions. The Department of Agriculture is conducting a broad study on time-temperature effects (*7*). The nutrition survey samples referred to above were stored for an average of approximately 7 months at 0° F. before they were analyzed. The present report deals with nutritional values in frozen green sweet peas, French-style green beans, sliced strawberries, and orange juice concentrate after storage under less favorable conditions. Twenty-one nutrients were determined in samples stored for periods ranging up to 11 months at 0° F. with an additional 1-month exposure at 20° F. in each case.

Materials and Methods

Two cases of each product were obtained directly from packers. Each product sample was of uniform good quality and processed from the same lot of raw material. The strawberries, French-style green beans, and green sweet peas were packed during the 1954 season. Concentrated orange juice

Table I. Observed Nutrient Levels

Frozen Food Time: Treatment	Vitamins, Mg./100 Grams: Total ascorbic acid	β-Carotene	Folic acid	Niacin	Pantothenic acid	Riboflavin	Thiamine	Vitamin B_6	Proximate Composition: Solids, %	Ash, %	Ether extract, %	Protein, %	Carbohydrate, %: Crude fiber	Total by difference	Calories/100 g., Atwater system	Minerals, Mg./100 Grams: Calcium	Total iron	Phosphorus	Magnesium	Potassium	Sodium
Green beans, French style																					
0:0	12.5	0.30	0.018	0.27	0.188	0.11	0.08	0.081	5.8	0.50	0.15	1.59	0.81	3.58	18	40	1.3	39	24	182	0.3
3:Control	7.6	0.24	0.018	0.41	0.093	0.11	0.08	0.093	7.4	0.54	0.15	0.56	0.80	5.15	23	44	1.9	37	24	182	0.3
3:Variation	4.3	0.19	0.021	0.39	0.060	0.11	0.09	0.094	7.6	0.54	0.22	1.56	0.82	5.28	25	42	1.7	38	24	192	0.5
6:Control	7.0	0.14	0.023	0.34	0.158	0.10	0.09	0.071	7.7	0.55	0.11	1.74	0.88	5.33	24	43	1.2	38	24	187	0.4
6:Variation	3.9	0.13	0.007	0.41	0.158	0.11	0.07	0.063	7.2	0.51	0.09	1.67	0.82	4.89	22	41	1.2	39	24	187	0.4
12:Control	6.0	0.23	0.017	0.39	0.089	0.11	0.08	0.064	7.6	0.47	0.02	1.76	0.93	5.38	24	40	1.1	36	23	193	0.6
12:Variation	3.0	0.22	0.019	0.36	0.080	0.10	0.11	0.064	7.7	0.47	0.07	1.78	0.89	5.33	24	39	1.1	36	22	196	0.6
Orange juice[a]																					
0:0	58.5	0.009	0.005	0.33	0.164	0.011	0.088	0.026	11.2	0.34	0.05	0.61	0.07	10.20	44	10.2	0.12	13.5	10.4	178	0.3
3:Control	59.3	0.006	0.005	0.26	0.143	0.016	0.071	0.028	11.7	0.41	0.03	0.60	0.07	10.61	45	10.1	0.10	13.3	10.1	172	0.2
3:Variation	57.8	0.006	0.005	0.20	0.143	0.014	0.071	0.027	11.6	0.37	0.02	0.62	0.07	10.58	45	10.1	0.10	13.2	10.1	172	0.2
6:Control	56.9	Trace	0.005	0.30	0.130	0.017	0.065	0.030	10.8	0.36	0.01	0.63	0.07	9.84	41	10.1	0.10	13.4	9.7	169	0.3
6:Variation	55.4	Trace	0.005	0.30	0.130	0.019	0.068	0.029	11.0	0.39	0.01	0.64	0.07	9.93	41	9.8	0.14	13.4	9.8	169	0.3
12:Control	57.1	0.003	0.005	0.35	0.176	0.018	0.062	0.028	10.7	0.35	0.02	0.63	0.07	9.66	41	10.1	0.12	13.3	10.5	173	0.3
12:Variation	55.9	0.003	0.005	0.35	0.178	0.016	0.065	0.028	10.6	0.36	0.01	0.66	0.05	9.62	41	9.8	0.13	13.4	10.0	173	0.3
Peas, green sweet																					
0:0	22.5	0.48	0.016	2.08	0.343	0.12	0.33	0.141	18.1	0.85	0.26	5.43	1.58	11.59	68	17	2.0	89	23	156	170
3:Control	20.9	0.41	0.014	2.24	0.250	0.12	0.36	0.207	18.9	0.88	0.41	5.44	1.58	12.16	72	18	5.4	88	21	156	171
3:Variation	11.8	0.41	0.010	2.32	0.160	0.11	0.34	0.185	18.9	0.84	0.46	5.31	1.64	12.34	73	17	5.3	88	21	156	171
6:Control	19.8	0.41	0.008	2.55	0.405	0.11	0.35	0.128	18.9	0.97	0.24	5.36	1.70	12.38	71	17	1.5	89	21	160	171
6:Variation	10.7	0.40	0.004	2.50	0.330	0.10	0.38	0.150	18.3	1.07	0.26	5.38	1.73	11.62	68	17	1.4	88	21	159	172
12:Control	20.1	0.46	0.016	2.13	0.243	0.13	0.32	0.131	19.4	0.75	0.14	5.57	1.82	12.90	73	16	1.7	89	21	160	180
12:Variation	8.2	0.43	0.015	2.06	0.205	0.13	0.35	0.129	18.8	0.96	0.10	5.46	1.81	12.31	70	16	1.6	90	20	160	180
Strawberries, sliced																					
0:0	41.7	0.01	0.003	0.30	0.090	0.01	0.01	0.048	27.4	0.24	0.05	0.33	0.51	26.76	103	14	0.4	19	8.7	121	2.5
3:Control	36.2	0.01	0.008	0.38	0.135	0.06	0.02	0.073	28.9	0.31	0.18	0.31	0.71	28.11	109	14	0.7	19	7.9	119	2.5
3:Variation	35.2	0.01	0.009	0.43	0.133	0.06	0.02	0.074	29.2	0.29	0.22	0.36	0.75	28.37	111	13	0.7	20	7.9	122	2.7
6:Control	35.7	. .	0.014	0.49	0.158	0.06	0.02	0.039	28.6	0.25	0.17	0.52	0.71	27.70	108	14	0.5	20	7.8	124	2.4
6:Variation	32.6	. . .	0.013	0.47	0.153	0.06	0.02	0.044	29.8	0.27	0.12	0.55	0.75	28.82	112	15	0.5	20	7.7	128	3.0
12:Control	36.4	<0.01	0.007	0.52	0.118	0.05	0.01	0.040	29.8	0.18	0.08	0.56	0.82	28.97	112	14	0.6	19	8.8	122	2.8
12:Variation	33.4	<0.01	0.007	0.52	0.127	0.05	0.02	0.041	29.4	0.13	0.01	0.58	0.66	28.64	110	15	0.5	19	9.4	123	3.3

[a] Values reported are calculated for reconstituted orange juice.

was drawn from the 1954–55 season. The packages were of a conventional type currently used in packing for the retail trade: for the vegetables, paperboard boxes overwrapped with waxed paper; for the strawberries, metal-end paperboard containers, and for the orange juice concentrate, metal cans. One case of each product was used as a control. The control samples were held at 0° F. throughout the study. Variation samples were held in storage at 0° F. for 2, 5, and 11 months and were exposed to 20° F. for 1 month at the end of these periods. This degree of mishandling was chosen to simulate serious abuse which can cause marked changes in color and flavor of certain products (*2*, *4*, *7*).

Initial analysis of each product was made on a composite sample prepared from three random packages from the control case and three random packages from the variation case. Subsequently, at each time interval, composite samples made from six random packages of each product from control and variation samples, respectively, were tested.

The preparation of samples and the analytical methods employed have been described (*1*). The total ascorbic acid values represent reduced ascorbic acid plus dehydroascorbic acid.

Results and Discussion

Analytical values obtained are given in Table I. Table II shows per cent retention of the nutrients measured under various storage conditions.

As would be expected, the greatest effects of storage were observed in the case of ascorbic acid. In the green beans, the retention of ascorbic acid was as low as 25% in the variation samples and as low as 50% in the control. In the peas, there was no appreciable loss of ascorbic acid in the control samples but as low as 36% retention in the samples exposed to the higher temperature. Strawberries showed comparatively little loss of ascorbic acid. Considering the time-temperature conditions, these results on strawberries are in line with the report of Guadagni, Nimmo, and Jansen (*2*).

There was complete retention of ascorbic acid in the orange juice concentrate. The concentrate was packed in metal containers which were effective in promoting retention of ascorbic acid in frozen strawberries (*2*). If frozen orange juice or concentrate is properly packed, the ascorbic acid is stable even at temperatures somewhat above 0° F. (*3*, *4*). However, the importance of maintaining storage temperature at 0° F. or lower is emphasized by McColloch *et al.* (*4*) who report that "changes in cloud and flavor occurring in frozen concentrated orange juice may be initiated at all temperatures above 0° F."

The variation in β-carotene in the orange juice concentrate samples is of

Frozen French-style green beans, green sweet peas, sliced strawberries, and orange juice concentrate were stored at 0° F. for 2, 5, and 11 months and exposed to 20° F. for 1 month at the end of these periods (variation samples). Control samples were held at 0° F. throughout these storage periods. Twenty-one nutrients were determined. In general, there was good retention of vitamins, except for ascorbic acid in all samples of green beans and in the variation samples of peas.

doubtful significance considering the low level of the vitamin present. There appears to be a moderate loss of thiamine in both the control and variation orange juice concentrate samples. Otherwise there is not a consistent decrease in any of the other nutrients, although in some of the products, the results might indicate a variable decrease in folic acid, pantothenic acid, riboflavin, vitamin B_6, and some of the proximate and mineral values. In addition to normal experimental error, these variations may be due to package-to-package variation or to inadequacies of some of the vitamin methods employed at their present stage of development. As put by Krehl and Cowgill (*3*), "It must be remembered, that the assay methods for folic acid, pyridoxine, and inositol have not reached as high a degree of accuracy as certain other microbiological methods—i.e., niacin. This, of course, does not mean that the present methods cannot be

Table II. Adjusted Per Cent Retention of Vitamins

(Expressed as % retention in control: % retention in variation sample)

Frozen Food Stored, Months	Vitamins: Ascorbic acid	β-Carotene	Folic acid	Niacin	Pantothenic acid	Riboflavin	Thiamine	Vitamin B_6	Proximate Composition: Solids, %	Ash, %	Ether Extract, %
Green beans, French style											
3	61:34	80:63	100:100b	100a:100b	49:32	100:100	100:100b	100a:100b	100a:100b	100a:100b	100:100b
6	56:31	47:43	100a:39	100a:100b	84:84	91:100	100a:88	88:78	100a:100b	100a:102	73:60
12	48:24	77:73	94:100b	100a:100b	47:43	100:91	100:100b	79:79	100a:100b	94:94	13:47
Orange juice, concentrate											
3	101:99	67:67	100:100	79:61	87:87	100a:100b	81:81	100a:104	104:104	100a:100b	60:40
6	97:95	[a]	100:100	91:91	79:79	100a:100b	74:77	100a:100b	96:98	100a:100b	20:20
12	98:96	33:33	100:100	100a:100b	100a:100b	100a:100b	70:74	100a:100b	96:95	103:100b	40:20
Peas, green sweet											
3	83:52	85:85	88:63	100a:100b	73:47	100:92	100a:103	100a:100b	104:104	104:99	100a:100b
6	88:48	85:83	50:25	100a:100b	100a:96	92:83	100a:100b	91:100b	104:101	100a:100b	92:100
12	89:36	96:90	100:94	102:99	71:60	100a:100b	97:100b	93:91	100a:104	88:100b	54:38
Strawberries, sliced											
3	87:84	100:100	100a:100b	100a:100b	68:67	67:67	100a:100b	100a:100b	100a:100b	100a:100b	100a:100b
6	86:78	[a]	100a:100b	100a:100b	79:77	67:67	100a:100b	81:92	104:100b	104:100b	100a:100b
12	87:80	[a]	100a:100b	100a:100b	59:64	56:56	100:100b	83:85	100a:100b	75:54	100a:20

Frozen Food Stored, Months	Proximate Composition: Protein, %	Carbohydrate, %: Crude fiber	Carbohydrate, %: Total by difference	Calories/100 g. Atwater system	Minerals: Calcium	Total iron	Phosphorus	Magnesium	Potassium	Sodium
Green beans, French style										
3	98:98	99:101	100a:100b	100a:100b	100a:100b	100a:100b	95:97	100:100	100:100b	100:100b
6	100a:100b	100a:101	100a:100b	100a:100b	100a:103	91:91	97:100	100:100	103:103	100a:100b
12	100a:100b	100a:100b	100a:100b	100a:100b	100:98	82:82	92:92	96:92	100a:100b	100a:100b
Orange juice, concentrate										
3	98:102	100:100	104:104	103:103	99:99	83:83	99:98	97:97	97:97	67:67
6	103:100b	100:100	96:97	96:97	99:96	83:100b	99:99	93:94	95:95	100:100
12	103:100b	100:71	95:94	95:95	99:96	100:100b	99:99	101:96	97:97	100:100
Peas, green sweet										
3	100:98	100:104	100a:100b	100a:100b	100a:100	100a:100b	99:99	91:91	100:100	101:101
6	99:99	100a:100b	100a:100	104:100	100:100	67:60	100:99	91:91	103:102	101:102
12	103:101	100a:100b	100a:100b	100a:103	94:94	80:73	100:101	91:87	103:103	100a:100b
Strawberries, sliced										
3	94:100b	100a:100b	100a:100b	100a:100b	100:93	100a:100b	100:100b	91:91	98:101	100:100b
6	100a:100b	100a:100b	104:100b	100a:100b	100:100b	100a:100b	100a:100b	90:89	102:100b	96:100b
12	100a:100b	100a:100b	100a:100b	100a:100b	100:100b	100a:100b	100:100	101:100b	101:102	100a:100b

100a expresses retentions in control samples of more than 104%, while 100b does the same for variation samples. Per cent retentions greater than 100 for both control and variation samples are attributable to minute amounts of some test components, to errors in assaying minute quantities, and to package variability within a given case of samples processed from the same lot of raw material.

[a] Insufficient to measure.

conducted with reasonable accuracy and precision; it simply means that the stage of our knowledge concerning the determination of the particular vitamins studied has not advanced as far."

In general, except for ascorbic acid in green beans, there was good retention of vitamins on storage of the four products studied for 12 months at 0° F. This also held for samples stored for 11 months at 0° F., plus 1 month at 20° F., except for the 25 to 50% retention of ascorbic acid in green beans and green sweet peas.

There appears to be a critical break point in stability of frozen food products not far above 0° F. with marked adverse effects on color and flavor, in addition to losses of ascorbic acid (*2, 4, 7*). This makes it imperative to avoid temperatures above 0° F. Currently, government agencies and the frozen food industry are working toward this end.

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CORRESPONDENCE

Lysine Content of Cottonseed Meals

Sir: Cottonseed meal, the proteinaceous residue of cottonseed which has been processed for oil, varies widely in its value as a protein supplement for poultry rations. This variation in nutritive value is consistently encountered even when the toxic component, gossypol, is reduced to what generally is accepted as a tolerable level.

Numerous investigators have demonstrated that exposure to heat during processing impairs the nutritive value of proteins. Often the evidence indicates that lysine is affected in some manner.

The determination of lysine by ion exchange procedures shows that autoclaving for 2 hours reduces the lysine content of cottonseed protein approximately 35%, as reported by Conkerton, Martinez, Mann, and Frampton [J. Agr. Food Chem. **5,** 460–3 (1957)]. In chick feeding studies, a similar 2-hour autoclaving period reduced the nutritive value of the cottonseed meal approximately 70%. Additional confirmation of the sensitivity of lysine to destruction, even under normal processing conditions, is reported in this communication.

The commercially produced meals used in this experiment were selected on the basis of protein solubility in dilute aqueous alkali and other chemical tests, to be representative of the extremes in heat-damaged meals encountered in the normal processing of cottonseed in the entire cotton belt. Wherever practicable, cottonseed samples representative of 3- to 4-ton seed lots entering the dehulling machinery were obtained at the oil mills producing the selected meals.

The seed samples were dehulled by hand and extracted at room temperature to remove the oil. Cottonseed meal S 6, 76 was prepared on a pilot-plant scale from prime seed by extraction of the rolled meats at room temperature with petroleum ether, followed by butanone.

The quantitative determinations of lysine were carried out on the hydrochloric acid hydrolyzates of the meal and seed materials by a modification of the Moore and Stein ion exchange procedure.

The data obtained on the cottonseed meals and the method by which they were produced are recorded in Table I. There is a marked variation in the lysine content of commercially produced cottonseed meals. This is significant in the utilization of cottonseed meal as a protein supplement in feeds for nonruminant animals, where the ration is composed, in a large measure, of cereals. In such instances, one function of the protein supplement is to make up the deficiency of lysine. Therefore, a part of the variation in the growth response of animals fed cottonseed meal as a protein supplement may be accounted for on the basis of the variation in the lysine supplied in the rations.

Table I. Lysine Content of Cottonseed Meals

Cottonseed Meal, Lot Number	Lysine, G./16 G. of Nitrogen	Process
S 6, 76	4.3	Experimental meal
C.M. 6	4.0	Prepress-solvent
C.M. 21	3.9	Screw press
C.M. 45	3.9	Prepress-solvent
C.M. 19	3.9	Solvent extracted
C.M. 36	3.6	Screw press
C.M. 16	3.6	Hydraulic press
C.M. 10	3.4	Prepress-solvent
C.M. 13	3.4	Screw press
C.M. 440	2.3	Screw press

A major portion of the variation in the lysine content of cottonseed meal arises because of lysine destruction during the processing operations, as shown in Table II. Approximately one fifth of the lysine content of the seed may be destroyed in conventional commercial operation.

Cottonseed meals C.M. 13 and C.M. 21 are both screw press meals. C.M. 13 was produced with an oil-cooled screw press operated at maximum capacity during which extremes of temperature and pressure are encountered. The resulting meal was scorched and dark brown in color. Conversely, C.M. 21 was produced with a water-cooled screw press operated at reduced speed and low temperature and pressure. The data taken as a whole, indicate that the quantity of lysine destroyed is dependent upon the severity of the processing, and not upon the type of processing used.

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Table II. Lysine Content of Cottonseed and of Meals Produced Therefrom

Cottonseed	Lysine, G./16 G. Nitrogen	Source	Cottonseed Meal	Lysine, G./16 G. Nitrogen	Process
C.S. 21	4.1	Commercial seed	C.M. 21	3.9	Screw press
C.S. 19	4.1	Commercial seed	C.M. 19	3.9	Solvent extracted
C.S. 36	4.2	Commercial seed	C.M. 36	3.6	Screw press
C.S. 10	4.1	Commercial seed	C.M. 10	3.4	Prepress solvent
C.S. 13	4.2	Commercial seed	C.M. 13	3.4	Screw press