Effect of Fluctuating Storage Temperatures on Quality of Frozen Foods

WILLIS A. GORTNER, FAITH FENTON, FRANCES E. VOLZ, AND ELLA GLEIM
Cornell University, Ithaca, N. Y.

Frozen pork roasts, strawberries, snap beans, and peas were stored for 12 months at 0° F., at 10° F., and in a freezer alternately fluctuating between 0° and 20° F. in a repeating, 6-day cycle. Palatability tests, vitamin analyses, and determinations of other criteria of food quality were made periodically. Fat rancidity was evident after 4 months at temperatures above zero, but did not occur during 1 year at 0° F. Ascorbic acid losses during storage at 10° F. or at fluctuating temperatures ranged from half of the total in peas to two thirds of that present in strawberries and four fifths of that in snap beans. At 0° F., all of this vitamin was retained in the peas, and only one third was lost from the other products during a 1-year storage period. Palatability changes in general paralleled these chemical changes. The thiamine content of pork was not affected by the storage temperatures or storage period. • Exposure of frozen food used in this study to temperatures fluctuating between 0° and 20° F. resulted in quality changes similar to those occurring in food stored at 10° F. The quality of the food stored under both of these conditions was definitely inferior to that stored at 0° F. This suggests that exposure of frozen foods to temperatures above 0° F. rather than merely temperature fluctuation may be a major factor influencing deterioration of food quality.

FLUCTUATING temperatures due to many different conditions may be encountered in the storage of frozen foods. Temporary breakdown of facilities or equipment, improper response of temperature controlling devices, excessive loads during freezing, lack of a separate compartment for freezing, and frequent opening of the cabinet (particularly a side-opening box) all may lead to a temporary rise in the temperature of the stored food.

Statements in the literature (*1, 3, 10*) stress the point that temperature fluctuations should be minimized, either because excessive desiccation may result or because fluctuations may lead to excessive growth of ice crystals in the food. None of these statements is accompanied by data to prove that undue desiccation or crystal growth necessarily is encountered in such cases, or to prove that the food quality is appreciably impaired.

Work at Purdue (*9*) indicated that temperature fluctuation over the range −5° to −15° F. was not deleterious to frozen pork. Workers at Minnesota (*4*) stated that for frozen fruits and vegetables, constant storage temperature is not important if the storage temperature is 5° F. or less. In their study, the temperature fluctuated from 0° to −20° F. with no impairment in food quality in a 6-month period.

DuBois and Colvin (*2*) studied ascorbic acid in frozen peaches subjected to small temperature fluctuation three or four times daily, and reported that, as a result of fluctuating temperatures between 5° and −5° F., 50% of the originally added ascorbic acid was lost in 1 year, whereas like packs stored at a relatively constant temperature of 0° F. lost but 32% of the vitamin C during storage. Their data, however, dispute the significance of this statement.

If undesirable effects from varying temperatures are to be expected, they will be found in the freezing range above 0° F., the usually recommended storage temperature. Furthermore, this is the range most likely to be encountered in freezer cabinets or lockers subject to temperature fluctuation. No data are at hand to evaluate the effects on frozen food of temperature variations in the range of 0° to 20° F.

PROCEDURES

STORAGE TEMPERATURES. Three conditions of storage were maintained. A standard temperature of 0° ± 2° F. and a control of 10° ± 2° F. were maintained as representing constant temperature conditions. A third freezer, equipped with two thermostatic control switches, was carried through a temperature cycle between 0° and 20° F. For 36 hours out of every 6-day period, the control was set at 20° F.; for the remainder of the period (108 hours), the other control, at 0° F., was in operation. This cycle was such that the food temperature rose from 0° to 20° F. and remained there for several hours before again being lowered to 0° F. The 6-day cycle was automatically repeated during the entire course of the 1-year storage period. The cycle represents a magnitude and frequency of fluctuations that may be encountered during frozen food storage without actual exposure to thawing.

FOODS. Pork roasts weighing 3.5 to 4 pounds, cut at the rib end of the loin to avoid the tenderloin, were trimmed to approximately 0.5 inch of fat. Two 1-inch chops were cut adjacent to each roast in order to provide material for chemical analyses, one before and one after storage. Paired roasts from the same carcass were obtained to provide a better comparison between the storage temperature extremes. The meats were wrapped in moisture-vapor-resistant cellophane and covered with stockinette.

Commercially frozen Birds Eye brand strawberries (4 parts berries to 1 part sugar), snap beans, and peas of the 1944 pack as well as peas of the 1945 pack were included. The replicate samples of each product were from a single lot.

Those packages subjected to fluctuating temperatures were separated from each other by means of 1-inch dunnage strips, thus enabling free circulation of air around each individual package of frozen food.

DESICCATION. The packaged product was weighed as it entered frozen storage, and was weighed back as it was removed from storage. The meat was weighed to the nearest gram; other products were weighed to the nearest 0.1 gram. Weighing was carried out in a room maintained at 0° F.

COLOR. The color of the thawed strawberries and of the vegetables before and after cooking was matched with plates found in the Paul and Maerz Dictionary of Color.

THIAMINE. Thiamine was determined in the raw pork. The entire edible portion of the chop was thoroughly ground and a sample taken for moisture (Association of Agricultural Chemists method). A 20-gram portion was mixed with 10 grams of sodium sulfate, dried 24 to 48 hours in a vacuum desiccator over sulfuric

Table I. Effect of Storage under Various Temperature Conditions on Quality of Frozen Pork Loin Roasts[a]

Time of Storage, Months	Condition of Storage, ° F.	Thiamine Content, γ per G. Fat-Free Dry Wt.: Before storage	Thiamine Content: Loss (−) or gain(+) during storage	Peroxide Value of Fat, Me. per Kg.	Palatability Score[b]: Flavor of fat	Palatability Score: Aroma	Palatability Score: Total score	Palatability Score: % of maximum rating	Moisture Loss, G./Pkg.
0	...	...	...	3.9[c]	6.7	6.7	44.3	90	...
4	0	25.0	−2.3	5.6	5.7	6.7	35.7	73	2.0
	10	28.5	−3.2	12.1	5.1	4.8	33.3	68	2.0
	Fluctuating, 0 to 20	28.4	−2.8	15.5	4.3	4.9	30.7	63	2.5
8	0	23.4	+1.6	5.1	5.1	5.8	38.0	78	7.0
	10	22.9	+1.2	31.0	4.3	5.2	31.2	64	11.5
	Fluctuating, 0 to 20	28.3	−5.2	19.5	4.3	4.9	32.5	66	6.0
12	0	23.6	+1.0	7.9	4.8	4.8	31.4	64	9.7
	10	26.2	−3.6	48.8	1.2	2.4	20.5	42	14.7
	Fluctuating, 0 to 20	25.4	−0.9	40.6	1.9	3.3	23.2	47	9.3

[a] Values are averages of 2 replications, except 12-month samples which had 3 replications on all but palatability scoring.
[b] Total possible score was 49; 7 points were allowed for each factor.
[c] Average of 13 samples. Eight samples excluded on basis of faulty high peroxide value resulting from improper handling during extraction.

acid, and exhaustively extracted in an all-glass Soxhlet apparatus with low boiling petroleum ether. The dry extracted solids were weighed, powdered in a micro-Wiley mill, and samples were taken for thiamine extraction. An extract was prepared with 0.1 *N* sulfuric acid and thiamine determined by the Research Corporation method (*8*). Thiamine was calculated on a fat-free, dry weight basis.

Peroxides. These were also determined on the raw meat. For some initial analyses the petroleum ether extract obtained incidental to the preparation of the meat for thiamine assay was used. Aliquots were taken for a gravimetric determination of total fat and for the determination of peroxides by a micromodification of the procedure of Kokatnur and Jelling (*5*). In subsequent analyses, peroxidation during extraction was minimized by mixing the ground meat sample with an equal quantity of sodium sulfate and covering with petroleum ether. After mixing and standing 1 hour, the extract was filtered off and the residue was washed several times with fresh solvent. The combined filtrates were clarified by shaking with sodium sulfate, refiltered, and diluted to a suitable volume for analysis.

Ascorbic Acid. Ascorbic acid was determined on the strawberries as ready for serving, and on the peas and beans both before and after cooking. The assay procedure was the indophenol-xylene method, as modified by Nelson and Somers (*7*), using 3% metaphosphoric acid extracts. For the raw products, the frozen foods were allowed to reach 32° F. and mixed, and samples were then taken for moisture and ascorbic acid assay.

Considerable sample-to-sample variation was evident in the vitamin content. The average deviation from the mean of the varying ascorbic acid content for the different products was generally in the neighborhood of 3 to 9 mg. per 100 grams of dry weight, but in some samplings was as high as 15 mg.

Cooking. For peas, 300 grams of the vegetable were dropped into 20 grams of boiling water in a Pyrex-covered 1.5-quart enamel saucepan. The water was returned to the boil and the peas were simmered for 4 minutes. They were stirred once after 2 minutes of simmering.

For green beans, 250-gram portions were dropped into 50 grams of boiling water and simmered for 8 minutes.

The pork roasts were allowed to stand at room temperature until an internal temperature of 32° F. was registered, and were then placed fat side up on a rack in an open pan. They were roasted at 350° F. in a Despatch oven with a rotating hearth until the center of the meat reached 185° F.

Palatability. Judging was done by a panel of from 5 to 10 experienced food judges. The tests were all made shortly before a meal. Scoring was based on the following:

For pork, a total of 49 points possible: 7 each for odor, flavor of fat, flavor of lean, texture, tenderness, quality of drippings, and quantity of drippings.

Table II. Effect of Storage under Various Temperature Conditions on Quality of Commercially Frozen Strawberries[a]

Time of Storage, Months	Condition of Storage, ° F.	Ascorbic Acid in Product[b], Mg./100 G. Fresh Weight	Ascorbic Acid Distribution, %[b]: Berries	Ascorbic Acid Distribution, %: Liquid	Palatability Score[c]: Total	Palatability Score: % of maximum rating	Moisture Loss, G./Pkg.
0	...	63	84	16	15.4	86	...
4	0	49	51	49	16.9	94	2.5
	10	14	49	51	10.4	58	12.3
	Fluctuating, 0 to 20	20	46	54	13.0	72	8.1
8	0	47	46	54	16.4	91	4.4
	10	22	45	55	16.1	89	10.9
	Fluctuating, 0 to 20	22	49	51	13.1	73	6.5
12	0	46	50	50	13.2	73	5.9
	10	21	49	51	10.2	57	14.0
	Fluctuating, 0 to 20	18	47	53	12.2	68	9.8

[a] Values are averages of 3 replications.
[b] Product thawed to 37° F.
[c] Total possible score for palatability was 18.

Table III. Effect of Storage under Various Temperature Conditions on Quality of Commercially Frozen Snap Beans[a]

Time of Storage, Months	Condition of Storage, ° F.	Total Solids, %	Ascorbic Acid in Raw Beans, Mg./100 G. Dry Wt.	Ascorbic Acid Retention, %[b]: Cooked beans	Ascorbic Acid Retention, %: Liquid	Palatability Score[c]: Total	Palatability Score: % of maximum rating	Moisture Loss, G./Pkg.
0	...	9	70	55	17	23.2	97	...
4	0	8	79	57	25	21.6	90	2.0
	10	8	31	60	41	15.4	64	4.8
	Fluctuating, 0 to 20	8	23	73	37	15.6	65	3.3
8	0	8	61	68	19	21.0	88	3.1
	10	9	12	94	36	17.4	72	6.1
	Fluctuating, 0 to 20	8	19	72	24	17.4	72	3.1
12	0	9	49	50	17	20.8	87	3.7
	10	8	17	47	18	15.2	63	7.3
	Fluctuating, 0 to 20	8	12	70	23	15.3	64	3.6

[a] Values are averages of 3 replications.
[b] Calculated on moist basis using weights of raw and cooked beans and of cooking water.
[c] Total possible score was 24.

For snap beans, a total of 24 points possible: odor 2, flavor 3, texture 3, surface appearance 5, color 7, and general acceptability 4.

For peas, a total of 27 points possible: odor 2, flavor 3, skin texture 3, cotyledon texture 3, surface appearance 5, color 7, and general acceptability 4.

For strawberries, a total of 18 points possible: odor 2, flavor 2, texture 3, surface appearance 3, color 4, and general acceptability 4.

The average deviation from the mean for the total palatability score on the various frozen foods was generally 10 to 15%. To aid in comparison of scores of the different foods, palatability has been recalculated as per cent of the maximum possible score.

SAMPLING PERIODS. Tests were made on the foods entering experimental storage, and at 4-, 8-, and 12-month intervals thereafter.

RESULTS

For all products, the moisture loss from the package was greatest in the 10° F. freezer, whereas the products undergoing wide temperature fluctuation in general lost no more moisture than did the foods at a uniform 0° F. It is possible that a lower relative humidity characterized the 10° F. freezer; this would account for a more rapid water-vapor transmission. Owing to the excellent protection afforded by the packaging materials, the expected desiccating effect of extreme temperature fluctuation was not evident when weight loss from the package was the criterion of desiccation. However, the foods exposed to temperatures above 0° F., and particularly to fluctuating temperatures, were subjected to a drying out within the package as evidenced by a marked accumulation of frost (cavity ice) in the air spaces in these packages. Further evidence was the dry appearance of the vegetables themselves, and of the ends of some of the pork roasts. The strawberries, which were a solid pack, did not appear dry.

Experimental storage of the frozen pork confirmed the importance of temperatures as low as 0° F. in maintaining quality over many months (Table I). After 4 months, the fat of all samples not stored at 0° F. was in incipient rancidity. At the 8- and 12-month samplings, these roasts had noticeably rancid odors and similar high peroxide values, whereas the controls at 0° F. were still essentially unchanged. The palatability scores dropped during storage of the meat, but after 1 year the roasts held at 0° F. still rated significantly higher than those at 10° F. or fluctuating between 0° and 20° F. The superiority of zero storage was particularly evident in the scores for aroma and for flavor of fat. The thiamine content was not altered significantly during storage of the frozen pork, regardless of the experimental condition.

For frozen strawberries (Table II), the total palatability scores were only slightly affected by the temperature condition during storage. The color darkened, however, according to the Maerz and Paul color chart—that is, the berries stored at the higher temperatures contained more gray and the slices of berry seemed more ragged and mushy. Ascorbic acid was lost during the first 4 months, but remained constant thereafter. The loss at 10° and at 0° to 20° F. amounted to two thirds of the total vitamin originally present; at 0° F. approximately one third was lost from the frozen strawberries. The ascorbic acid was approximately equally divided between berries and sirup.

Snap beans deteriorated rapidly and strikingly when exposed to storage temperatures above 0° F. (Table III). The palatability score changed but slightly during 1 year at 0° F. but dropped 30% within 4 months at higher temperatures. An increase in gray color was noted. This darkening of the color accompanied the changes in palatability. Even zero storage was not sufficient to prevent a significant loss of ascorbic acid. After 1 year, 30% of this vitamin was lost at 0° F., but at 10° or at 0° to 20° F. the loss amounted to approximately 80%. No consistent trend associated with storage time or temperature was noted in the retention of ascorbic acid during cooking.

Because the 1944-pack frozen peas initially used were 9 months old at the time they were first put in storage under the experimental conditions set up for this study, it seemed desirable to check on the possible deleterious effect of any lowered palatability and vitamin content in the initial sample (Table IV). Accordingly, a second lot of peas from the new, 1945 frozen pack was later included in the study (Table V). The new lot, while having a somewhat higher initial ascorbic acid content, failed to score as high in the taste tests owing to the inclusion of peas of various sizes and degrees of maturity (garden-run pack).

No appreciable decline in palatability was observed in the 1944-pack peas. The score for the 1945 pack held up at 0° F. but declined at the other experimental temperatures owing to a development of haylike flavors and odors and loss of sweetness usually associated with insufficient blanching. Semiquantitative tests for peroxidase (*6*) on the 1944-pack peas were entirely negative, but presence of the enzyme in the 1945 pack was indicated

TABLE IV. EFFECT OF STORAGE UNDER VARIOUS TEMPERATURE CONDITIONS ON QUALITY OF COMMERCIALLY FROZEN PEAS (1944 PACK)[a]

Time of Storage, Months	Condition of Storage, ° F.	Total Solids, %	Ascorbic Acid in Raw Peas, Mg./100 G. Dry Wt.	Ascorbic Acid Retention, %[b]		Palatability Score[c]		Moisture Loss, G./Pkg.
				Cooked peas	Liquid	Total	% of maximum rating	
0	...	21	78	102	10	24.1	89	...
4	0	19	89	88	19	22.7	84	2.2
	10	19	69	89	17	19.3	71	3.6
	Fluctuating, 0 to 20	20	49	76	16	19.4	72	3.5
8	0	21	81	72	22	22.3	83	3.6
	10	21	56	83	19	22.1	82	3.7
	Fluctuating, 0 to 20	20	57	73	18	23.1	86	2.1
12	0	22	75	77	18	21.8	81	4.8
	10	21	44	82	18	19.3	71	7.5
	Fluctuating, 0 to 20	20	53	82	19	22.1	82	2.4

[a] Values are averages of 3 replications.
[b] Calculated on moist basis using weights of raw and cooked peas and of cooking water.
[c] Total possible score was 27.

TABLE V. EFFECT OF STORAGE UNDER VARIOUS TEMPERATURE CONDITIONS ON QUALITY OF COMMERCIALLY FROZEN PEAS (1945 PACK)[a]

Time of Storage, Months	Condition of Storage, ° F.	Total Solids, %	Ascorbic Acid in Raw Peas, Mg./100 G. Dry Wt.	Ascorbic Acid Retention, %[b]		Palatability Score[c]		Moisture Loss, G./Pkg.
				Cooked peas	Liquid	Total	% of maximum rating	
0	...	18	107	76	20	21.9	81	...
4	0	19	99	78	24	20.2	75	0.8
	10	18	96	67	21	19.7	73	0.9
	Fluctuating, 0 to 20	18	73	72	20	19.9	74	0.5
8	0	20	108	82	16	22.2	82	1.5
	10	19	64	71	27	17.2	64	1.4
	Fluctuating, 0 to 20	19	90	72	20	18.6	69	0.4
12	0	19	107	73	21	21.4	79	2.3
	10	20	45	81	20	17.5	65	3.4
	Fluctuating, 0 to 20	19	54	77	18	15.7	58	1.3

[a] Values are averages of 4 replications.
[b] Calculated on moist basis using weights of raw and cooked peas and of cooking water.
[c] Total possible score was 27.

in the guaiacol test by strong color development after 3 to 5 minutes.

In contrast to snap beans, the ascorbic acid of peas was completely retained in both packs during 1 year at 0° F. At 10° or at 0° to 20° F. the peas lost approximately half of the vitamin in 12 months. Although no carotene determinations were made on the samples entering experimental storage, duplicate samples of the 1945-pack peas after storage for 12 months showed average carotene contents of 20.5, 20.2, and 19.8 micrograms per gram at 0°, 10°, and 0° to 20° F., respectively. Hence the temperature coefficient for destruction of carotene in frozen peas must be very low.

DISCUSSION

The many striking changes in quality of the four frozen foods during storage appear to be attributable to exposure to temperatures above 0° F. The rancidification of pork, the loss of ascorbic acid from strawberries, beans, and peas, and the drop in palatability of all these items (except the peas of the 1944 pack) when subjected to frequent cycling of the temperature between 0° and 20° F. were qualitatively and quantitatively similar to the changes observed when the storage temperature was held at 10° F.

After completion of this study, a paper by Woodroof and Shelor (*11*) indicated that strawberries deteriorated more rapidly at a temperature fluctuating from 0° to 10° F. than when held constant at 10° F. The nature of the cycle of temperature fluctuation was not further defined. A possible explanation of their contradictory findings on strawberries is that no sirup or sugar was added as it interfered with tenderometer and leakage experiments (used as criteria of quality). Presence of the usual sugar sirups should afford a stabilizing influence.

These data suggest that with the possible exception of a desiccating effect, temperature fluctuation, as such, does not seriously impair the quality of frozen foods. These findings confirm and extend the observations of Hustrulid and Winter (*4*) in this respect. Before broad generalizations can be drawn, however, further studies on additional products should be made. These results again emphasize the importance of maintaining storage temperatures at 0° F. or lower if the eating qualities and nutritional values of frozen foods are to be maintained from season to season.

ACKNOWLEDGMENT

The authors wish to thank the Sears, Roebuck Company, Chicago, Ill., and the Jewett Refrigerator Company, Buffalo, N. Y., for their cooperation in providing freezer cabinets and Birds Eye-Snider, Inc., Albion, N. Y., for the frozen strawberries, beans, and peas used in this study. Acknowledgment is made to Marjorie Swisher, formerly of the staff of the Bureau of Human Nutrition and Home Economics, for some of the analyses reported here.

LITERATURE CITED

(1) Christensen, P. B., *Food Freezing*, **1**, 25 (1945).
(2) DuBois, C. W., and Colvin, D. L., *Fruit Prod. J.*, **25**, 101 (1945).
(3) Guest, W. E., *Ice and Refrig.*, **96**, 339 (1939).
(4) Hustrulid, A., and Winter, J. D., *Agr. Eng.*, **24**, 416 (1943).
(5) Kokatnur, V. R., and Jelling, M., *J. Am. Chem. Soc.*, **63**, 1432 (1941).
(6) Masure, M. P., and Campbell, H., *Fruit Prod. J.*, **23**, 369 (1944).
(7) Nelson, W. L., and Somers, G. F., Ind. Eng. Chem., Anal. Ed., **17**, 754 (1945).
(8) Research Corp. Committee, *Cereal Chemists Bull.*, 2 (May 1942).
(9) Shrewsbury, C. L., Horne, L. W., Braun, W. Q., Jordan, R., Milligan, O., Vestal, C. M., and Weitkamp, N. E., *Purdue Agr. Expt. Sta. Bull.* **472** (1942).
(10) Woodroof, J. G., *Ga. Agr. Expt. Sta. Bull.* **201** (1938).
(11) Woodroof, J. G., and Shelor, E., *Food Freezing*, **2**, 206 (1947).

Received March 7, 1947. Work was performed in cooperation with the Bureau of Human Nutrition and Home Economics, United States Department of Agriculture.

COPPER 8-QUINOLINOLATE INDUSTRIAL PRESERVATIVE

P. G. BENIGNUS

Monsanto Chemical Company, St. Louis, Mo.

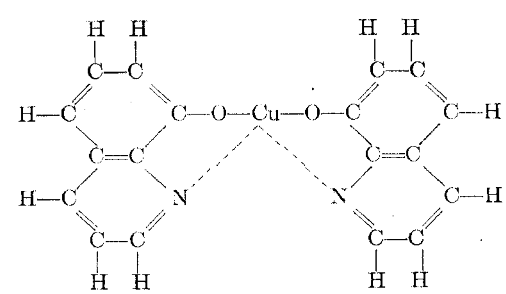
Copper 8-quinolinolate is the copper derivative of 8-quinolinol which was prepared in 1880 by Bedall and Fischer (*2*) from quinoline sulfonic acid by fusion with sodium acetate and subsequent hydrolysis of the fusion mass. During this same year, Skraup (*6*) described a preferred method for preparing 8-quinolinol, using *o*-nitrophenol, *o*-aminophenol, glycerol, and sulfuric acid; this reaction became well known as the Skraup synthesis.

Although 8-quinolinol and numerous derivatives have been used for many years as pharmaceutical agents, the commercial manufacture and use of the copper derivative are recent developments as a result of the urgent need for a superior preservative for military fabrics during the recent war.

Copper naphthenate, copper hydroxynaphthenate, copper oleate, copper stearate, copper ammonium fluoride, pentachlorophenol, 2-chloro-*o*-phenylphenol, 2,2′-dihydroxy-5, 5′-dichlorodiphenylmethane, tetrabromo-*o*-cresol, 2,3-dichloro-1, 4-naphthoquinone, organo-mercuric compounds, salicylanilide, zinc dimethyldithiocarbamate, and various quaternary ammonium compounds were studied and used in connection with this program but none completely met the over-all requirements.

PHYSICAL AND CHEMICAL PROPERTIES OF COPPER 8-QUINOLINOLATE

Structural Formula:



Molecular Weight = 351.83

Copper 8-quinolinolate, a light yellowish-green, odorless powder, is a practically nonionizable, chelated compound. Its copper is bonded firmly by both primary and secondary valence linkages, making the chemical very stable. It is quantitatively insoluble in acid or alkali within the pH range of 2.7 to above 12