Retention of nutrients in canned buffered aqueous solutions as influenced by rotational sterilization in a steam/air retort

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Abbatemarco, C. and Ramaswamy, H.S. 1995. **Retention of nutrients in canned buffered aqueous solutions as influenced by rotational sterilization in a steam/air retort**. Can. Agric. Eng. 37:345-350. Aqueous mixtures of ascorbic acid and thiamin hydrochloride dissolved in 0.1 M phosphate buffer (pH 5.5) were sealed in small glass vials (1.8 mL capacity) and placed in 307 X 409 (87 mm diameter, 115 mm height) cans filled with vegetables. In addition, aqueous buffered solutions of vitamins were also directly filled into separate 307 X 409 cans and vacuum sealed. All cans were subjected to approximately equivalent accumulated process lethalities ($F_o \sim 10$ min) in a rotary steam/air retort. Process variables were retort temperature (110-130°C) and rotation speed (0-20 rpm). Percentage retention of the vitamins in each sample was determined experimentally using a HPLC technique. The study indicated that processing at 110°C yielded significantly lower retention ($p<0.05$) of both vitamins, relative to 120 and 130°C. However, the rotation speed did not have a significant effect ($p > 0.05$) on the retention of vitamin in either cans or in-can vials. Estimated ascorbic acid and thiamin retention using experimental time-temperature data and published kinetic parameters showed fairly good agreement with experimental data.

Un mélange aqueux d'acide ascorbique et de thiamine ajouté à une solution tampon (pH 5.5) est placé dans des ampoules de verre puis scellé. Les ampoules sont ensuite placées dans des boîtes de conserve (307 X 409: 87 mm X 115 mm) remplies de légumes. Parallèlement, le même mélange est directement introduit dans des boîtes de conserve de même gabarit puis scellé sous-vide. Les conserves sont alors soumises à un traitement thermique d'une durée approximative de $F_o \sim 10$ min dans un autoclave rotatif à vapeur et air forcé. Les facteurs étudiés sont la température (110°C, 120°C, et 130°C) et la vitesse de rotation (0 rpm, 10 rpm, et 20 rpm). Le taux de rétention de vitamines dans chaque échantillon est mesuré à l'aide d'un HPLC. Les résultats obtenus démontrent que le traitement thermique à 110°C procure un taux de rétention d'acide ascorbique et de thiamine significativement inférieur ($p<0.05$) à celui obtenu à partir des deux autres températures. Par contre, pour les milieux d'immersion et les températures étudiées, la vitesse de rotation n'a pas d'effet ($p > 0.05$) sur le taux de rétention de vitamines. Les taux de rétention de vitamines ont été prédits à partir de paramètres de cinétique de destruction thermique trouvés dans la littérature combinés aux données de temps-température. Les valeurs expérimentales trouvées concordent bien avec les données prédites.

INTRODUCTION

The major task of the food industry is to provide a wide variety of food products which are considered safe and of consistently high quality. This represents quite a challenge to processors since nutrients and other desirable attributes are also subject to thermal degradation as are microbial populations which cause spoilage and public health concern. There has been considerable effort in the past to estimate and/or quantify thermal degradation of quality factors in foods for optimization of thermal processing conditions (Teixeira et al. 1969; Lenz and Lund 1977; Laing et al. 1978; Leonard et al. 1986; Fox et al. 1982; Mohr and Kirschstein 1988).

It is generally recognized that ascorbic acid and thiamin are severely lost when subjected to processing operations such as blanching and boiling, more so because of leaching due to their water solubility than heat destruction. When subjected to canning processes, thermal destruction of nutrients tend to be more significant. Commercial developments such as rotational sterilization have been used as effective alternatives to conventional still retorting since they can provide increased heat transfer rates and, hence, high quality food products (Eisner 1988). Sorman and Zajac (1974) demonstrated that rotational sterilization may be used to advantage for the increased retention of B vitamins (thiamine, riboflavin, and pyridoxine) in meats subjected to rotational sterilization as compared to static or still sterilization. Most investigators studying kinetics of the thermal degradation of vitamins in canned products have used still retort operations (Ramaswamy et al. 1990).

The main objective of this study was to evaluate the thermal degradation of ascorbic acid and thiamin in canned vegetable products as influenced by rotary sterilization to determine if the induced agitation due to container rotation would improve their retention. A second objective was to verify if the experimental data on vitamin destruction in cans as a result of rotary sterilization can be reasonably predicted using product time-temperature data during processing and kinetic parameters for the thermal destruction of the vitamins.

MATERIALS AND METHODS

A 0.1 M phosphate buffer solution was prepared by mixing 7.098 g Na_2HPO_4 and 6.805 g KH_2PO_4 with 300 mL HPLC grade water (Millipore Ltd., Mississauga, ON). The pH of the solution was adjusted to 5.5 (Chemcadet pH meter, Cole Parmer, Chicago, IL) with 3% metaphosphoric-acetic acid solution prepared as per AOAC (1990) method. L-ascorbic acid (Analar, BDH Inc., Montreal, QC) and thiamin hydrochloride (BDH Inc., Montreal, QC) were added to the buffer solution (0.05 g/100 mL buffer). The solution was then sub-

jected to a vacuum at room temperature for 1 h to eliminate oxygen. Glass vials (1.8 mL capacity) with TFE-lined cap (Fisher Scientific, Montreal, QC) were filled with the vitamin solutions using a syringe and stored overnight in a refrigerator before processing. Duplicate vials containing the vitamin-buffer mixture were placed in each of 6 cans (307 X 409: 87 mm diameter, 115 mm height) containing a vegetable mixture as described in Abbatemarco and Ramaswamy (1994). Furthermore, buffered solutions containing vitamins were also filled into separate cans of the same size containing no vegetables. All cans were vacuum sealed to 20 mm Hg using a seaming machine (Continental Can Company, New York, NY) prior to retorting.

Heat penetration tests were conducted in a Lagarde Rotary Simulator (Autoclaves Lagarde, Montelimar, France) retort with a horizontal shell (diameter: 521 mm) and a rotary cage (275 mm height x 295 mm width x 570 mm length). The space along the central axis was used for positioning open cans with installed CNS (copper-constantan) needle thermocouples (Ecklund-Harrison Technologies, Cape Coral, FL) to measure the retort temperature. The heating medium used was a mixture of steam and air consisting of 75% steam and 25% air regulated by maintaining the appropriate retort temperature (steam pressure) and total pressure (steam + air). A turbo fan mounted on the retort door kept the medium well mixed for achieving uniform temperature distribution. CNS needle-type thermocouples were also inserted into the geometric centre of all test cans. In several cans containing vials and vegetables, the rigid CNS thermocouple was inserted into the vial through the rubber cap. Its tip was positioned in the vitamin solution inside so that the temperature of the vitamin solution could be measured during the process.

A full factorial design was employed with three processing temperatures (110, 120, and 130°C) and three rotation speeds (0, 10, and 20 rpm). Temperature readings were recorded via a datalogger with 16 channels (Dash-8, Metra-Byte Corp., Tauton, MA) attached to a desk top computer at 15 second intervals as cans containing vitamin solutions alone and those containing vials and a vegetable mixture were subjected to end-over-end rotation. Each experimental run consisted of a 10 min come-up time and a 3 min pressure cooling followed by a 20 min regular cooling time (water temperature = 5°C) after the established process time to achieve the desired lethality. Control vials and control cans, not processed, were kept in the refrigerator (4°C) until analysis the following day.

To determine the required process times which would give an equivalent accumulated lethality (F_0) for the vegetable pieces, four test cans with a thermocouple tip inserted into the largest vegetable piece (usually potato) were used. Additional details on the heat penetration data gathering are given in Abbatemarco and Ramaswamy (1994). Time-temperature data from these test cans were simultaneously gathered and displayed in terms of accumulated F_0 values using an Ellab A/S data acquisition system (Ellab A/S, DK, Rodovre, Denmark). The cans were processed until the accumulated lethality reached a value of approximately 10 min at which time the heating was stopped and cooling initiated. Data gathering was continued until the cooling was completed to determine the total lethality achieved in each can equipped with a thermocouple.

Data processing

The accumulated process lethality (F_o during the entire process) was calculated for each processing condition by numerical integration of time-temperature data (z = 10°C) (T denotes temperature in °C):

$$F_o = \int 10^{(T-121.1)/10}\, dt \qquad (1)$$

Process time employed for representing the process was calculated based on the traditional 42% effectiveness (Stumbo 1973) as operator's process time (time after come-up) plus 42% of the come-up time (which was a 10 min fixed time in these studies).

The cook value was also computed by numerical integration using a z value of 33°C which represents an average z value for typical food quality factors (Eisner 1988):

$$C_o = \int 10^{(T-100)/33}\, dt \qquad (2)$$

Since process lethalities between test runs for experimental data (uncorrected for initial and set retort temperatures) were not identical, the ratio C_o/F_o was used as an indicator of the relative degree of cooking at various retort temperatures and rotation speeds (Mohr and Kirschstein 1988).

Quantitative estimation of vitamin retention

The percentage retention of ascorbic acid and thiamin hydrochloride following processing was determined using a HPLC technique similar to that described by Ramaswamy et al. (1990) with a few modifications. A Waters HPLC system (Waters Chromatography Division, Milford, MA) which consisted of a Waters Model 501 HPLC Pump and 486 Tunable UV Absorbance Detector was used for analysis. The column was a m-Bondapak C18 3.9 mm x 300 mm stainless steel column (125 A and 10 µm diameter). The mobile phase consisted of methanol, HPLC-grade water, and low UV PIC B6 (Waters) (hexane sulfonic acid) in the following amounts, respectively: 197.50 g, 751.50 g, and 4.00 g. The mobile phase was mixed using a magnetic stirrer for 10 min and filtered through a Millipore GV type 0.22 µm filter (Millipore, Bedford, MA) while simultaneously degassed. The flow rate was set at 1 mL/min and the mobile phase was administered isocratically. The UV detector was set to 254 nm with 0.1 AUFS (Absorbance Unit Full Scale).

After processing, the vials and cans were stored again at refrigerated conditions overnight before analysis. Quantitative analysis of the results was done using Baseline 810 Chromatography Software (Millipore Corp., Milford, MA). Separate calibration curves for each vitamin (based on the area of the peak) were generated from mixtures prepared in buffered solutions as described previously. The average of triplicate measurements was used for each point on the curve. The following calibration equations were obtained (X = vitamin peak response, Y = concentration x injection volume):

$$\text{Ascorbic acid: } Y = (4.82 \times 10^{-7})\, X - 0.1168 \qquad R^2=1.00 \qquad (3)$$

$$\text{Thiamin: } Y = (7.21 \times 10^{-7})\, X - 0.0397 \qquad R^2=1.00 \qquad (4)$$

An 8 μL sample of the vitamin mixture from each glass vial and can was removed using a Microliter Syringe (#802, Hamilton Co., Reno, NV) and injected directly into the chromatograph injection port. Retention times for ascorbic acid and thiamin were 2.93 and 8.33 min, respectively. Peak responses of the vitamins were determined by the chromatographic software based on calibration curves. For each experimental condition, percentage retention was determined by dividing the concentration of the processed sample by the concentration of the unprocessed control sample.

Table I: Experimental percentage retention for ascorbic acid and thiamin in cans and in-can vials.

Temp. (°C)	Rotation speed (rpm)	PT# (min)	F_o (min)	C_o (min)	C_o/F_o	Thiamin (%)	AA* (%)
Can							
110	0	142.2	15.5	326.2	21.0	60.6	87.8
110	10	139.1	15.7	325.4	20.7	54.1	92.6
110	20	138.3	15.3	318.5	20.8	59.3	91.4
120	0	28.4	22.5	112.3	5.0	78.5	99.3
120	10	27.3	21.6	105.9	4.9	81.9	99.5
120	20	25.6	16.9	74.9	4.4	86.6	96.4
130	0	14.3	49.6	78.4	1.6	82.3	99.4
130	10	12.8	52.3	78.1	1.5	76.9	96.9
130	20	10.6	52.0	73.6	1.4	78.7	89.5
Vials							
110	0	142.2	15.0	325.0	21.7	57.5	90.6
110	10	139.1	13.3	307.5	23.1	56.8	92.4
110	20	138.3	15.1	316.4	21.0	58.1	91.5
120	0	28.4	17.3	102.8	5.9	78.7	97.5
120	10	27.3	22.4	108.6	4.8	80.0	93.7
120	20	25.6	14.9	72.1	4.8	86.0	95.5
130	0	14.3	43.5	77.0	1.8	82.2	95.5
130	10	12.8	38.2	69.8	1.8	79.6	100.0
130	20	10.6	39.0	66.2	1.7	79.1	96.6

* AA = Ascorbic acid
\# PT = Process time

RESULTS AND DISCUSSION

Table I shows data on percentage retention of thiamin and ascorbic acid both in cans and in-can vials as influenced by rotational processing. Notably, differences between the percentage retention of vitamins in cans and in-can vials were small and so were the differences with respect to rotation speed. However, temperature showed a dominant influence with the lower temperature (110°C) resulting in a much lower residual vitamin content due to the greater destruction rate for these vitamins as compared to that of microorganisms. These observations were confirmed by an analysis of variance test. Statistical data on the differences between the mean values of ascorbic acid and thiamin as influenced by temperature and rotation speed are presented in **Table II.**

Figure 1 shows the temperature profiles of the vitamin solution in the vial (Fig. 1a) at 120°C subjected to both 0 rpm and 20 rpm. As expected, the natural convection for vials processed in the static mode was as efficient as the rotation induced agitation caused by processing at 20 rpm. Figure 1b shows the temperature profile at 120°C and 0 rpm of an in-can vial placed at the thermocouple tip and that of the can filled only with the vitamin solution. It was expected that the temperature of the liquid in the in-can vial would show a considerable lag with respect to the temperature of the liquid bulk in the can, at least with respect to the can which was filled with only the liquid. Furthermore, the differences between the two were expected to be more noticeable in the still mode (Fig. 1b) of retort operation (zero rpm). However, the associated temperature lags were small and accounted for only very small differences in the residual vitamin content as indicated in Table I. Similarly, differences in temperature profiles as a result of rotation speed were small and these differences were not significant ($p > 0.05$) with respect to vitamin retention possibly due to the existence of dominant free convection in cans. The small temperature lags that were apparent during the heating were probably compensated by similar lags during cooling, thus providing similar overall heat treatments.

Retort temperature in the range 110°C to 130°C, however, contributed to significant differences ($p < 0.05$) in the percentage retention of vitamins; higher temperature resulting in better retention. An equation to predict thiamin retention (in-can vials) based on rotation speed and temperature was developed using multiple regression: ($R^2 = 0.94$; SEE = 2.89; P-value = 0.0109; DF (error)= 3) (S = rotation speed, rpm; T_r = retort temperature, °C):

$$\% \text{ Thiamin Retention} = -1893 + 31.67\,T_r + 0.8967\,S - 0.1269\,T_r2 + 0.01467\,S^2 - 0.009250\,T_r\,S \quad (5)$$

The results of the above regression model are shown in Fig. 2 along with experimental values of thiamin retention. Experimental data are represented by points; the regression values (Eq. 5) are indicated as lines through the experimental data. The convex nature of the curves with a peak between

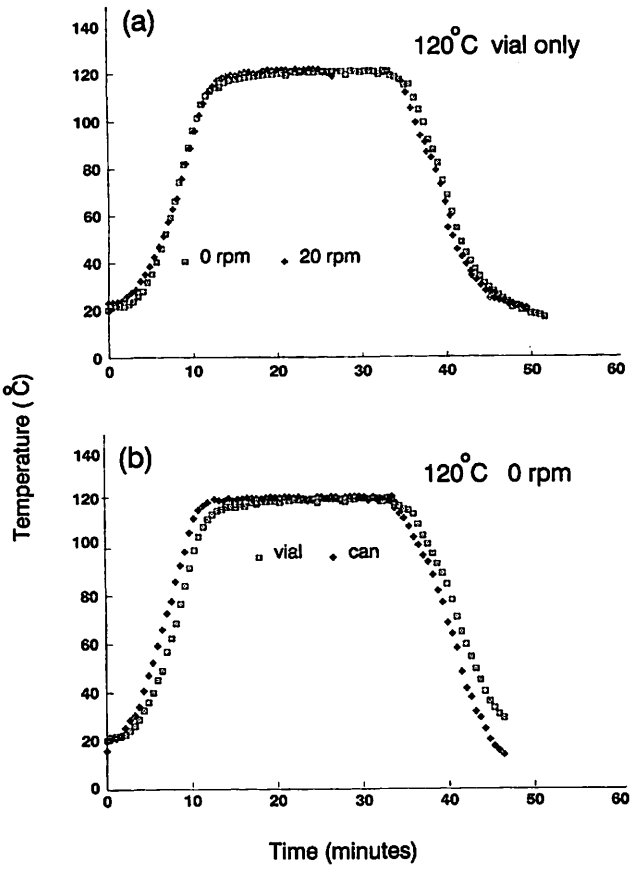

Fig. 1. Typical time-temperature profiles of cans and in-can vials during processing at 120°C in a rotary retort a) for vial only at 0 and 20 rpm; b) for vial and can at 20 rpm.

Table II: Mean values for ascorbic acid and thiamin percentage retention in cans (C) and in-can vials (V)

	Ascorbic acid		Thiamin	
	V	C	V	C
Temperature (°C)				
110	91.5[a]	90.5[a]	57.5[a]	57.8[a]
120	95.6[b]	98.4[b]	81.5[b]	82.3[b]
130	97.6[b]	95.7[b]	80.3[b]	79.3[b]
Rotation speed (rpm)				
0	94.6[a]	94.9[a]	72.8[a]	73.8[a]
10	95.6[a]	95.9[a]	72.2[a]	71.0[a]
20	94.5[a]	92.1[a]	74.4[a]	77.2[a]

For both temperature and rotation speed, means followed by a different superscript letter within the same column are significantly different ($p < 0.05$).

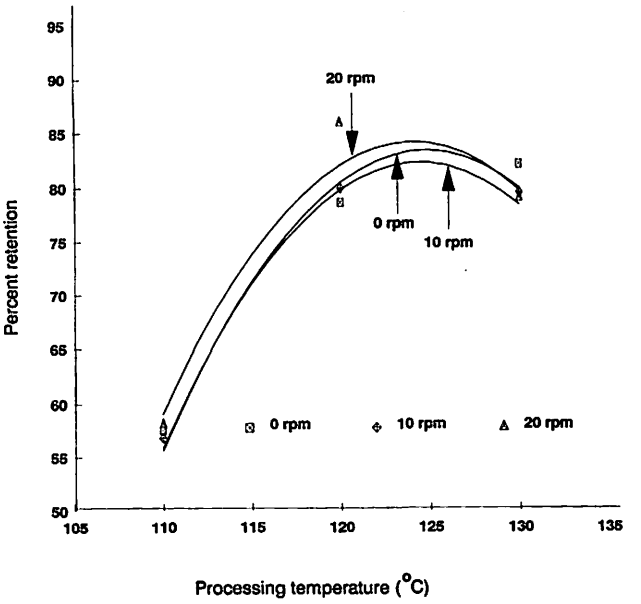

Fig. 2. Percentage thiamin retention as a function of processing temperature at various rotation speeds.

120 and 130°C was only based on the regression model as a result of the steep increase in values between 110 and 120°C, and only marginally increase or slightly decrease thereafter at 130°C. Although statistically not significant, the model shows a slightly better retention of vitamin while being processed at 20 rpm as compared with processing at 10 rpm. The natural convection dominated still processing was closer to the 10 rpm process at 110°C crossing over toward the 20 rpm process at 130°C (indicating improved natural convection at the higher temperature). However, it is clear that with respect to vitamin retention, processing temperature had the major effect.

Cook value is generally taken to represent quality changes associated with thermal processes (Eisner 1988). The process times for various conditions were based on giving an F_0 value of approximately 10 min for the vegetables. While this process lethality was approximately maintained for vegetables, the liquid portion in the cans invariably received a higher process lethality, especially for 130°C. As a result, the cook values also varied somewhat (Table I). The effectiveness of such processes may be evaluated using C_o/F_o values. Lower C_o/F_o ratios yield better processes with respect to quality factor retention. C_o/F_o values at 110°C were approximately ten times those at 130°C, supporting the significantly higher retention of vitamins at the higher temperature. Simple linear regression of actual cook values versus vitamin retention yielded very good correlations; R^2 results for ascorbic acid and thiamin were 0.964 and 0.969, respectively.

The percentage retentions of thiamin at different temperatures are in close agreement with those reported by Feliciotti and Esselen (1957) who determined thiamin destruction rates in phosphate buffer at pH 5.5. They found that a solution heated for 151 min at 109°C had a retention of 59%; heating for 31 min at 119°C yielded a retention of 79% and heating

for 11 min at 129°C yielded a retention of 81%. Temperatures and heating times in the present study varied slightly (Table I); however, the percentage retentions are still within a similar range of values.

The relatively high percentage retention of ascorbic acid in vials and cans at both varying temperatures and rotation speeds indicated that it is more stable to thermal destruction than thiamin. This finding was also noted by Ghazala (1989) who observed that ascorbic acid alone in aqueous solutions in cans (pH = 4.05) had an experimental D_0 value of 2320.8 min in the temperature range of 110-127°C (z value = 30°C). The high percentage retention of ascorbic acid in this study also points in this direction; on an average, 97% retention was found at 120°C following a 27 min process time. This would yield a decimal reduction time (D_{120}) of 2040 min. Similarly, based on 91% retention after a 140 min process, D_{110} was calculated as 3420 min. From a 12.6 min process at 130°C with 96.3% retention, D_{130} was calculated as 770 min. Regression of this data in the form of log D vs temperature gave a D_0 (D at 121.1°C) of 1610 min and a z value of 31°C. These kinetic parameters for ascorbic acid retention compare well with earlier findings (Ghazala 1989). A similar analysis of data yields a z value of 30.3°C with a D_o of 139 min for thiamin. This z value for thiamin also compares fairly well with the z value of 26.4-30.6°C reported by Ramaswamy et al. (1990).

Ghazala (1989) also pointed out that ascorbic acid degraded much slower in cans than in ampoules (4 mL) due to the possibility of low concentrations of the can wall material leaching into the solution and acting as an electron donor, reducing the overall rate of ascorbic acid destruction. Despite reports which seem to attribute this higher retention of ascorbic acid to the reducing activity of the tinplate of the can (Nagy 1980), the results provided in this study tend to disagree with this hypothesis as percentage retentions in both vials and cans were not found to differ significantly as previously mentioned. For ascorbic acid in foods, the rate of destruction under anaerobic conditions is known to be one-tenth that under aerobic conditions (Liao and Seib 1988; Priestley 1979; Kefford et al. 1959). Although this information cannot be extrapolated directly to model aqueous systems, it may explain the relatively high resistance to thermal destruction in this study in which available oxygen in the vials and cans was present at very low levels or was lacking completely. The variable thermal destruction behavior of ascorbic acid, therefore, still remains to be explored.

Predictions for percentage retention were done employing time-temperature data from the current set of experiments and kinetic parameters for the destruction of ascorbic acid and thiamin from Ghazala (1989). Associated errors with predicted values as compared with experimental values are shown in Table III. The greatest errors were generally associated with 130°C, although all percent errors had a tendency to overestimate retention within a relatively small margin of error.

Table III: Estimated errors in the percentage retention of ascorbic acid and thiamin using time-temperature data and documented kinetic parameters

Temp. (°C)	rpm	Vial	Can
Ascorbic acid			
110	0	+3.0	+6.3
110	10	+1.1	+0.8
110	20	+2.2	+2.3
120	0	+0.2	-1.7
120	10	+4.2	-2.0
120	20	+3.0	+2.2
130	0	+2.8	-1.2
130	10	-1.8	+1.3
130	20	+2.2	+10.4
Thiamin			
110	0	-0.5	-4.8
110	10	+1.8	+6.5
110	20	+0.9	-1.5
120	0	+4.4	+2.3
120	10	+1.1	-4.0
120	20	+1.9	+0.6
130	0	+2.8	+3.2
130	10	+5.7	+9.8
130	20	-11.6	+10.0

CONCLUSIONS

The results of this study indicate that the percentage retention of both ascorbic acid and thiamin in buffered aqueous solutions processed in the rotational mode (10 and 20 rpm) was not significantly different from that of similar solutions processed in the static mode. Percentage retention for solutions processed at 110°C was significantly lower ($p<0.05$) than those processed at 120°C and 130°C. These results, however, seem to suggest that although rotational sterilization may achieve increased retention of nutrients in viscous, conduction-heating foods, the process does not appear to offer significant advantages in retention for experimental conditions outlined in our study.

The thermal destruction behavior of ascorbic acid in this study was found to be somewhat unpredictable. This finding may be related to the effects of oxygen.

Good predictions were obtained for estimations of experimental thiamin and ascorbic acid retention in a model system using experimental time-temperature data and literature values of kinetic parameters.

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