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Journal of
FOOD PROCESS ENGINEERING

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MEETINGS

NOVEMBER 1980

November 3—6: UCD-FDA BETTER PROCESS CONTROL SCHOOL. University of California-Davis. Contact R. C. Pearl, Department of Food Science and Technology, University of California, Davis, CA 95616.

November 8—11: 75TH ANNUAL AMERICAN MEAT INSTITUTE CONVENTION. McCormick Place, Chicago, Illinois. Contact Dept. of Convention and Meetings, American Meat Institute, P. O. Box 3556, Washington, D. C. 20007.

November 9—14: WORLD CONFERENCE ON SOYA PROCESSING AND UTILIZATION. Acapulco, Mexico. Contact American Oil Chemists Society, 508 S. 6th St. Champaign, Illinois 61820.

November 10—13: FOOD TECH '80. Auckland, New Zealand. Contact Food Tech '80, Trade Publications, Ltd., P. O. Box 1614, Auckland, New Zealand.

November 16—19: 73RD ANNUAL MEETING OF AMERICAN IN-STITUTE OF CHEMICAL ENGINEERS. Chicago, Illinois. Contact Edward Fochtman, Technical Program Chairman, 73rd AIChE Meeting, IIT Research Institute, 10 W. 35th St., Chicago, Illinois 60616.

November 24—28: COMBINATION PROCESSES IN FOOD IRRADI-ATION. Columbo Sri Lanka. Contact J. H. Kane, International Technical Conference Specialist, Office of Resource Applications, Department of Energy, Washington, D. C. 20545.

DECEMBER 1980

December 2—5: ASAE WINTER MEETING-ENGINEERING A SAFER FOOD MACHINE. Palmer House, Chicago, Illinois. Contact M. S. Purschwitz, ASAE Headquarters, Box 410, St. Joseph, MI 40985.

December 8—10: WORKSHOP ON ENERGY CONSERVATION AND FOOD PROCESSING. O'Hare Ramada Inn, Chicago, Illinois. Contact John J. O'Neil, Food and Nutrition Press, Inc., 265 Post Road West, Westport, CT 06880.

AN APPROACH TO SOLVING THE OPTIMAL TEMPERATURE CONTROL PROBLEM FOR STERILIZATION OF CONDUCTION-HEATING FOODS

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ABSTRACT

In this paper, the optimal sterilization of canned solid foods is discussed. Such foods are influenced by heat conduction in the course of the thermal process. The way of optimizing the temperature control in this paper is to divide the total problem into two separate parts, namely into a process engineering and a control engineering optimization problem. It will be shown that this separation is admissable. With respect to the solution of the total problem, the results will be suboptimal.

INTRODUCTION

Modern control theory enables the engineer to employ new procedures when dealing with very complicated systems and to realize optimal solutions. A relatively new problem is the optimization of so-called distributed parameter systems to which belongs the problem of optimal sterilization of canned solid foods. Similar problems arise, for example, when producing plastics or other synthetic materials, i.e. artificial carbon (Altmann 1974). Two other examples are the optimal tempering of steel and the optimal control of a furnace to heat the ingots (steel billets) so as to manufacture them in a rolling mill (Franke 1974).

The thermal treatment for preserving foods causes two oppositely directed reactions which are both time and temperature dependent (Loncin 1973). The first is characterized by the desired destruction of microorganisms which cause spoilage and the second is characterized by the reduction of vitamins and other valuable substances. Therefore,

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it is necessary to obtain an optimal compromise with regard to quality
and consistency. This forms a very complex optimization problem
which must be solved from the beginning for any new type of canned
food. Furthermore, the problem is nonlinear, since the temperature ap-
pears as the state variable in the argument of exponential functions.
In (Flambert 1973), a survey of important research works is given
which deals with the preservation of packaged foods with the help of
sterilization.

Analytical, numerical and experimental solutions are proposed both
for the solution of the differential equation, which describes the heat
transfer in a container, as well as for the estimation of the sterilization
factor. This sterilization factor normally concerns only a specially
selected form of microorganisms. Saguy and Karel (1979); Teixeira *et
al.* (1975a,b); Thijssen *et al.* (1978) are examples of recent works done
in this area of optimization methods. Some methods use a so-called
critical point for the desired thermal process conditions. Other pro-
cedures are based on mean values for the considered quality factors
averaged over the total volume of the container. Most of the pro-
cedures work with a heating phase followed by a cooling phase
whereby the control temperature on the container wall is kept constant
during each phase. In the newer publications (Saguy and Karel 1979;
Teixeira *et al.* (1975a,b) a variable retort temperature is proposed.

In this work, an alternative way of solving this optimization pro-
blem is presented.

The basic idea consists in the separation of the total problem into
two separate parts, namely a process engineering and a control
engineering problem. The first problem deals with the determination of
a temperature variation for the special contents. The second problem
consists in controlling the temperature on the container wall in such a
manner that the actual temperature of each volume element within the
can tracks the optimal curve. Therefore, this task is called a tracking
problem.

An important issue of this approach is to prove that the separation
into two problems is permissible. It will be shown that this separation
generally leads to modified solutions with regard to the total pro-
blem. These solutions can, however, be regarded as suboptimal if the
tracking problem is based on a quadratic cost functional. Finally, the
results for the quasi-continuous and the optimal two-point control are
given, whereby it is assumed that the desired trajectory is either
parabolic or that it is a step function.

THEORY

A cylindric can with height $2l$ and radius a is considered (see Fig. 1).
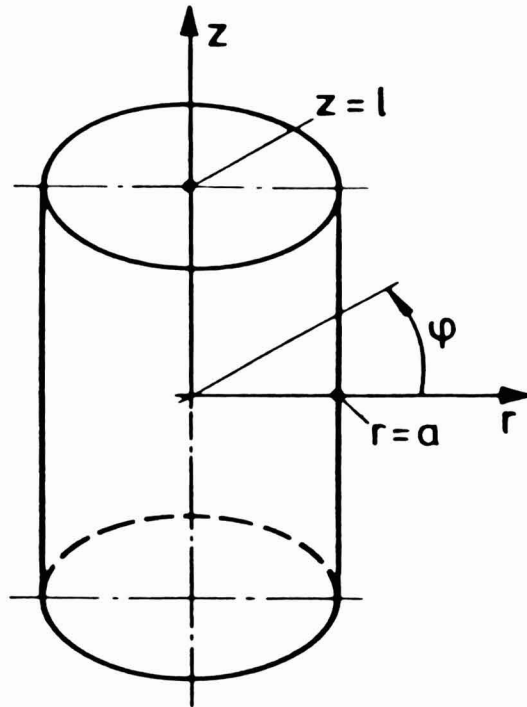
FIG. 1. CYLINDRIC CAN

The following basic assumptions are made:
a) heat conductivity, specific heat and density of the contents are constant,
b) heat is transported only by conduction,
c) the control temperature u_r is the same over the whole surface of the container wall,
d) there are no heat sources inside the container.
In this way one obtains for the temperature $x(t,r,z)$, based on an energy balance and with the aid of Fourier's law, the partial differential equation

$$\frac{\partial x}{\partial t} - \alpha \cdot \left[\frac{\partial^2 x}{\partial r^2} + \frac{1}{r}\frac{\partial x}{\partial r} + \frac{\partial^2 x}{\partial z^2} \right] = 0 . \tag{1}$$

In food technology, Dirichlet's boundary conditions are usually used for such problems, since the heat resistance of the metal wall of the container can be neglected. Practical measurements show good agreement of this assumption with reality (Flambert 1973). The boundary conditions for t > 0 are therefore

$$x(t,r=a,z) = x(t,r,z=\pm \ell) = u_r(t) \tag{2}$$

The initial condition is

$$x(t=0,r,z) = x_o(r,z) \tag{3}$$

In order to gain a state representation in an integral form, the usual formalism is employed in order to calculate the Green's function for distributed parameter systems, (Franke 1968; Gilles 1973). For the linear system in question, the space and time dependent temperature is obtained as

$$x(t,r,z) = \int_{u=o}^{a} \int_{\rho=-\ell}^{+\ell} g_o(t,r,z,u,\rho)\, x_o(u,\rho)\, du\, d\rho$$

$$+ \int_{\tau=0}^{t} g_r(t\text{-}\tau,r,z)\, u_r(\tau)\, d\tau \tag{4}$$

As has already been mentioned, the problem may be solved by separating the general problem. The question for the process engineering problem is: How must the temperature of each volume element inside the can be varied in order to destroy the necessary number of microorganisms and at the same time to save an optimal quantity of quality factors?

The control engineering optimization problem now has the aim of varying the temperature on the container wall so that, at each point in the interior of the can, the actual temperature follows the desired function as closely as possible. The separation will avoid difficulties which would otherwise make the solution very complicated and complex. Furthermore, a better insight into the physical process is possible.

$$J_{ges} = \int_{t=0}^{t_e} \int_{r=0}^{a} \int_{z=-\ell}^{+\ell} \int_{i=1}^{s} f_i(c_{i\,des} - c_i) \; dz \; dr \; dt \qquad (5)$$

$c_{i\,des}$ are desired values for the concentration of quality factors or allowed concentrations of left-over microorganisms,

c_i are the corresponding real values, which are generally dependent on:

$$c_i = c_i(t,r,z,x(t,r,z,u_r),c_i(t=0)),$$

f_i are known functions or constants,

s is the number of the quality components taken into account.

The optimization problem consists of minimizing this cost functional J_{ges} with respect to the control variable u_r. The solution of this problem can be based on the assumption that for a known homogeneous medium in the can there exists a temperature variation $w(t)$ which results in an optimal compromise between quality and durability. A possible time delay between the desired function and the real temperature at any point obviously does not influence the resulting concentration and therefore the value of the cost functional is not affected. For this reason, a time and space dependent desired function can be defined, whereby the spatial dependence on coordinates r and z is restricted to the dead-time $T_t(r,z)$. One obtains

$$w(t,r,z) = w(t-T_t(r,z)) \qquad (6)$$

Equation (6) will be enlarged upon later.

With the aid of the calculus of variations at the generalized point $x(t,r,z) = w(t,r,z)$, the integrand of Eq. (5) is expanded into a Taylor-series with respect to variations of x:

$$J_{ges} = \int_{t=0}^{t_e} \int_{r=0}^{a} \int_{z=-\ell}^{+\ell} \left\{ \left. \sum_{i=1}^{s} f_i(c_{i\,des} - c_j) \right|_{x=w} \right.$$

$$\left. - \sum_{i=1}^{s} \left. \frac{\delta f_i}{\delta x} \right|_{x=w} \cdot (w-x) \right. \qquad (7)$$

$$+ \tfrac{1}{2} \sum_{i=1}^{S} \left. \frac{\delta^2 f_i}{\delta x^2} \right|_{x=w} \cdot (w-x)^2$$

$$+ \text{ higher order terms } \} \, dz \, dr \, dt \qquad .$$

In a short notation one gets

$$J_{ges} = [J_o + J_1 + J_2 + \ldots] \qquad . \qquad (8)$$

The nonlinear cost functional J_{ges} in the first approximation, close to the minimum $x=w$ is described by the term J_2. By substituting the nonlinear cost functional J_{ges} with the second variation J_2 in the minimum, one can expect the solutions to be suboptimal. The resulting error is caused by neglecting the higher order terms; it becomes smaller the nearer one approaches the minimum of the cost functional. In this sense, the existence of a global minimum and a continuous second variation of the cost functional J_{ges} at the generalized point $x=w$ is assumed.

Since no concrete assumptions concerning the functions f_i have been made in this paper, the relation

$$Q_1(t,r,z) = \tfrac{1}{2} \sum_{i=1}^{S} \left. \frac{\delta^2 f_i}{\delta x^2} \right|_{x=w} \qquad (9)$$

is not explicitly given. Therefore, this function is assumed as a constant with a value $Q_1 = 1$. This does not mean any restriction because a time and space dependent function $Q_1 (t,r,z)$ would not cause basic difficulties. In the appendix, a simple example is given which explains the possible nature of functions f_i (Eq. (A6)).

It would seem necessary at this point to give an additional explanation of the desired function $w(t,r,z)$. This function is generally time and space dependent, whereby the space dependency is characterized only by a dead-time $T_t(r,z)$. This desired function is only time-dependent for a very small volume element of a homogeneous medium, which could be temperature controlled at any size desired. This function is step-like, but of course a step function is a rather unrealistic possibility as far as temperature profiles within the interior of the can are concerned. It is therefore necessary, when solving the process engineering prob-

lem, to look for a desired function which is not step-like. This means, that the process engineering problem for a desired function of a special content, must be solved on the condition that this function can be realized approximately at each point in the interior of the can. Only then can the minimum of the functional be approached and, accordingly, with the aid of the second variation, the approximation is mathematically realistic and physically suitable. In the appendix, a supplementary condition in connection with the example is given, which must be adhered to so as to avoid the desired function being step-like (Eq. (A5)).

Theoretically, the best package geometry is in the form of a thin flat plate. However, since this is not practical, it is necessary to find a more suitable function. The results use parabolic and step functions as examples of illustrating how the solution is obtained. The step functions are chosen to show that with the given control temperature u_r it is not possible to realize the desired function approximately at each spatial point. However, the results show that a parabolic temperature curve is attainable at each interior point. The choice of this curve is a way of advising the food process engineer to look for a desired function which has a parabolic form.

The consideration of dead-times leads, with respect to the tracking problem, to two coupled optimization problems which can only be solved numerically. For simplification, the following will be restricted to a time-dependent desired function w(t). In this way, a clearer representation of the results is possible. The application of a purely time-dependent desired function in the time-interval $0 < t \leqslant t_e$ leads further to a type of time-optimal process. The desired function w(t) should be followed within a fixed time interval. According to these considerations, the quadratic term of the cost functional J_{ges} has the form

$$J_2 = \int_{t=0}^{t_e} \int_{r=0}^{a} \int_{z=-\ell}^{+\ell} [w(t) - x(t,r,z)]^2 \cdot Q(t,r,z) \, dz \, dr \, dt \quad (10)$$

With the previous assumption $Q_1 = 1$, an arbitrary weighting function $Q(t,r,z)$ has been retained which allows a proper weighting of the quadratic deviation of the real temperature from the desired function. It is possible to illustrate this procedure in the one-dimensional scalar case. The nonlinear cost functional (8) is substituted by the second variation at the generalized point $x=w$.

These considerations show that the solutions based on this quadratic cost functional are suboptimal for the total problem. The
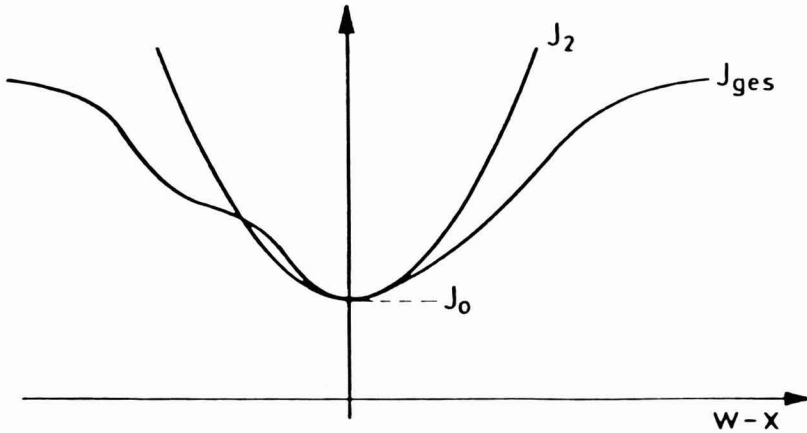
FIG. 2. SUBSTITUTION OF THE NONLINEAR COST
FUNCTIONAL J_{ges} IN THE MINIMUM POINT X = W
BY THE SECOND VARIATION J_2

(example for a one-dimensional scalar case)

advantage of this decoupling is that the two single problems are easier
to solve than the actual total problem.

Summarizing, it can be established that the separation of the total
problem into a process and a control engineering part leads to modified
solutions. These solutions may, however, be regarded as suboptimal.

RESULTS AND DISCUSSION

Equations (4) and (10) are the basic relations for the control
engineering optimization problem which in the classical calculus of
variations is known as the Lagrange problem. For the wall
temperature $u_r(t)$ ($0 < t \leqslant t_e$), a control strategy is sought, which
causes a minimum value for the quadratic cost functional. The underly-
ing assumption is that the control temperature u_r is limited by the
maximal heating temperature M and the minimal cooling temperature
m. In (Hildenbrand 1979) the tracking problem is solved with the aid
of Butkovskiy's version (Butkovskiy 1969) of the maximum principle.
The desired function w(t) is assumed to be known. It is beyond the
scope of this paper to derive the optimum conditions for the tracking
problem and therefore only the solutions and the basic assumptions
are given. The assumption that the wall temperature u_r is the same
over the whole surface of the can means that the calculation of the

temperature can be limited to the region $r,z \geq 0$. The course of the temperature $z < 0$ is symmetric. In order to analyze the system on a digital computer a spatial and a time discretization must be carried out. Therefore, a number of $z_i = 100$ spatial points are fixed at the region $r,z \geq 0$. The choice of $z_i = 100$ spatial points takes its origin from the calculation of a measured temperature curve with a growing number of spatial points. The number of points was raised until the calculated curve and the measured curve became quasi identical and a further raising of the number of spatial points would not have improved the accuracy.
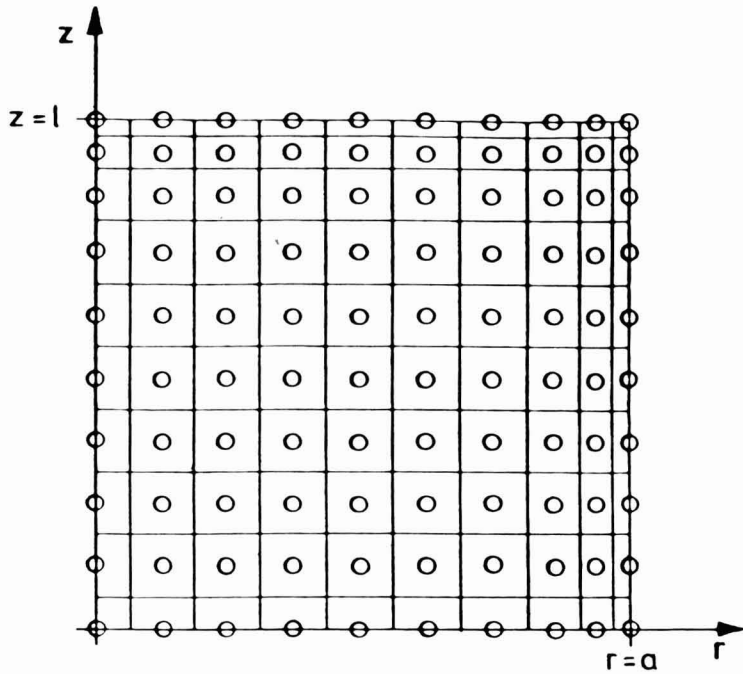
FIG. 3. SPATIAL DISCRETIZATION

O   spatial point for the calculation of the temperature
[O]   related spatial area element

Further, the total time interval $0 \leq t \leq t_e$ is divided into n equidistant intervals of the length $\Delta t = t_e/n$. The time functions are regarded to be constant within each time interval. The real data for the dimension of the cylindrical can, as well as for the coefficient of thermal conductivity, are taken from (Flambert 1973):

height of can: $2l = 10.3$ cm
diameter: $2a = 7.3$ cm
coefficient of thermal conductivity: $a = 0.092$ cm²/min.

The total time for the sterilization process is assumed to be $t_e = 85$ minutes and it is divided into n = 50 time intervals. The large number of time intervals was chosen in order to arrive at a quasi-continuous solution for the control temperature u_r. It should be mentioned that although the accuracy of the calculation of the temperature does not depend on time discretization, the solution for the optimal control curve depends directly on the number of time intervals. The solution for the optimal temperature curves is given in three arbitrarily chosen points P_1 and P_2 and P_3.
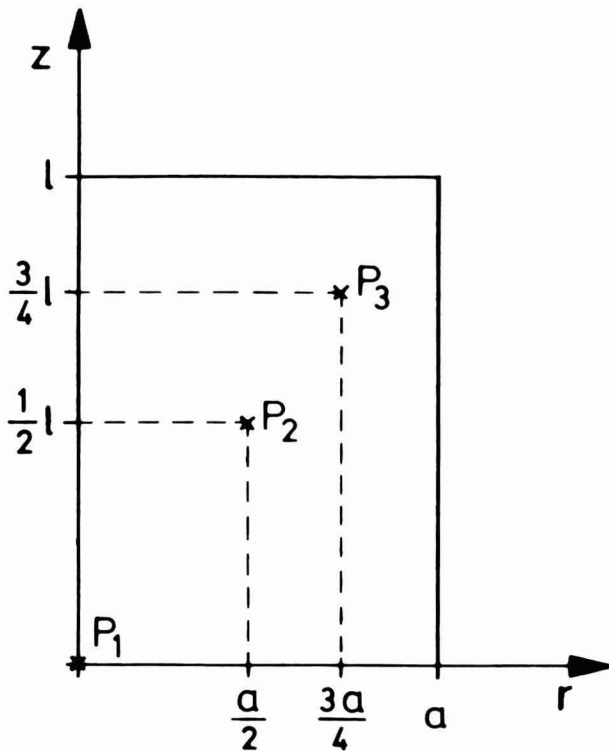
FIG. 4. SELECTED SPATIAL POINTS P_1, P_2 AND P_3
TO ILLUSTRATE THE OPTIMAL TEMPERATURE
COURSE DEPENDING ON THE DESIRED
FUNCTION
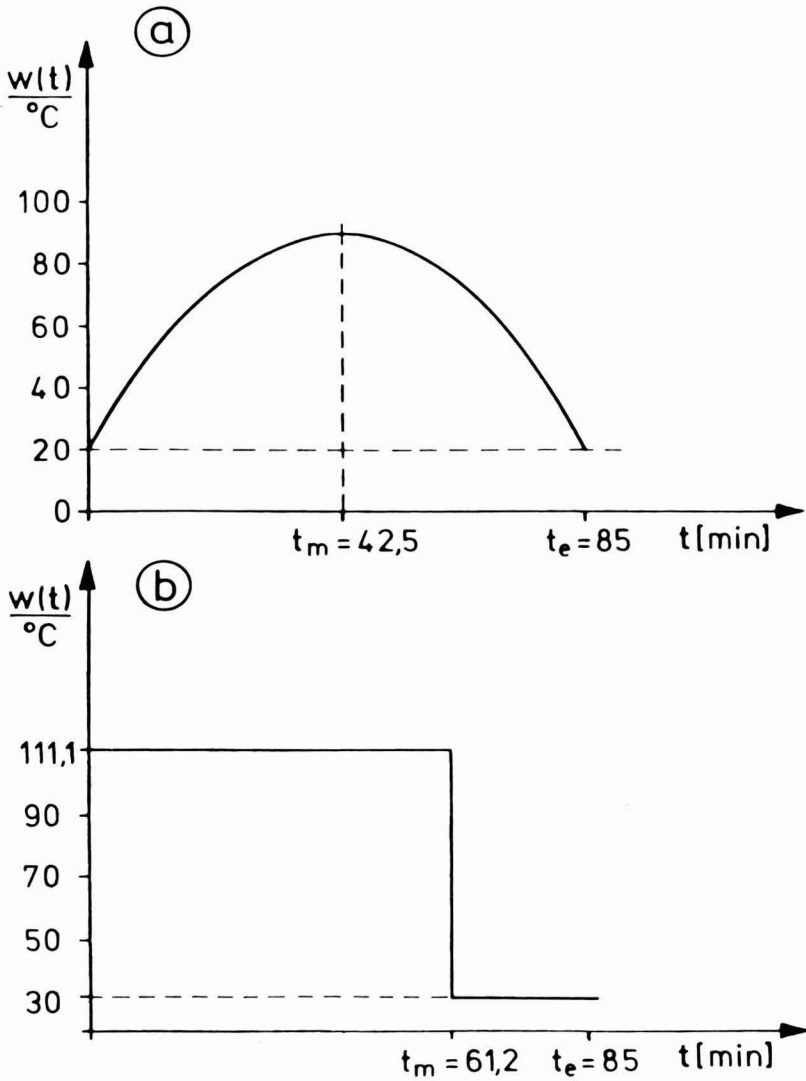
FIG. 5. DESIRED FUNCTIONS:

a) Parabolic function
b) Step function

As has already been discussed, the examples use two types of desired functions. The first is a parabolic function and the second is chosen as a step function. The necessary real data can be taken from Fig. 5. Further, the data for the numerical example, set out in the following table, were chosen arbitrarily:

desired function		parabolic function	step function
homogeneous initial temperature:	x_0	20 °C	30 °C
maximum heating temperature:	M	100 °C	121,1 °C
cooling temperature:	m	15 °C	15 °C

In the following figures, the numerical solutions for a suboptimal two-point control are confronted with the quasi-continuous solution of the discrete maximum principle. This maximum principle is a necessary and a sufficient condition for the minimization of the basic quadratic cost functional (Hildenbrand 1979). By applying the Bang-Bang control, the wall temperature $u_r = M$) or on the lower boundary $(u_r = m)$. Further, by employing the discrete maximum principle, the boundary temperature can have intermediate values. The process of calculating the optimal control is given in detail in (Hildenbrand 1979) and is described briefly in (Hildenbrand 1980). Since a detailed account of the specific procedure used to evaluate the control function u_r would be a lengthy process, it will not be expanded in this paper.

In order to compare the Bang-Bang solution with the quasi-continuous solution of the discrete maximum principle, the "goodness of tracking" is weighted on the basis of the following performance index. The smoother the value J_{min}, the better the temperature tracks the desired function. This value is stated in the Fig. 7,8,9,10.

$$J_{min} = \sum_{j=1}^{n} (\underline{w}_j - \underline{x}_j)\, T_Q(\underline{w}_j - \underline{x}_j) \cdot 10^{-3} \tag{11}$$

The following gives the dimension of the temperature vector $\underline{x}_j$ and its desired function vector $\underline{w}_j$.

$$\dim (\underline{x}_j) = \dim (\underline{w}_j) = 64 \;\; ; \;\; j = 1,2,\ldots,n \qquad .$$

From this it can be seen that not all $z_1 = 100$ spatial points of Fig. 3 are used to calculate the optimal control. Only the points lying in the hatched area of Fig. 6 are taken into account. This is due to the fact that the numerical evaluation was carried out on a PDP 11/45 computer. For reasons of memory capacity, it was not possible to use all the 100 spatial points. The elements of the diagonal matrix Q of the discrete form of Eq. (10) and of Eq. (11) were chosen so that they
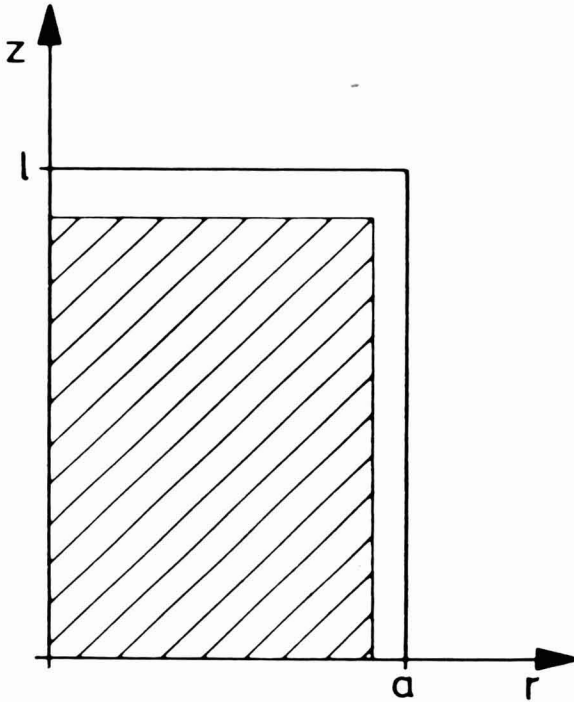
FIG. 6. THE SPATIAL POINTS, TAKEN INTO
ACCOUNT FOR THE OPTIMAL CONTROL PROB-
LEM, CAN BE OBSERVED IN THE HATCHED AREA

characterize the area of the corresponding surface parts. The
characterizing volume weighting factors could, of course, be chosen in-
stead.

Figures 7 and 9 show the results for the parabolic desired function.
In Fig. 8 and 10 the solutions which are based on a step-like desired
function are shown. In each figure, the temperature curves in the
spatial points P_1, P_2 and P_3 as shown in Fig. 4 are given. At the same
time, the allocated desired function w(t) is shown. In the lower part of
the figures, the allocated control function u_r can be observed.

When comparing the values for the performance index of Eq. (11)
for the parabolic as well as for the step-like desired function, it is ap-
parent that the values for the quasi-continuous control functions are
smaller. Consequently, the quasi-continuous solution results in better
tracking.

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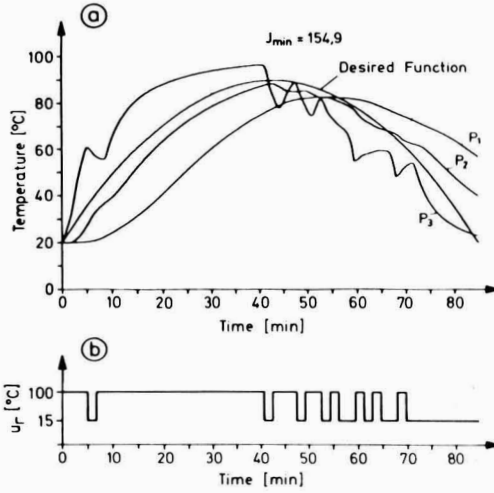
FIG. 7. a) OPTIMAL TEMPERATURE COURSE
(PARABOLIC DESIRED FUNCTION) b) BANG-
BANG CONTROL
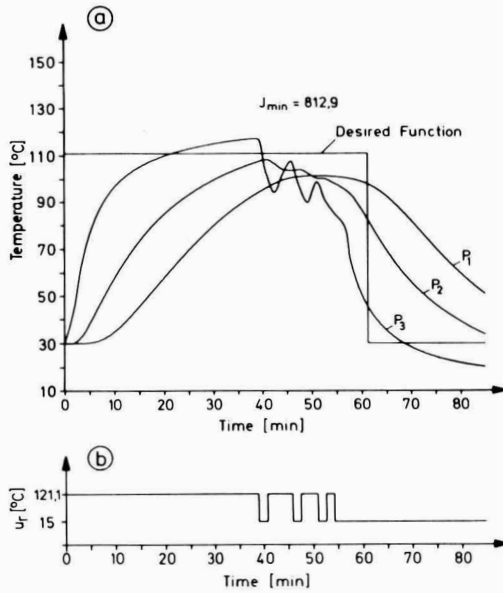
FIG. 8. a) OPTIMAL TEMPERATURE COURSE
(STEP-LIKE DESIRED FUNCTION) b) BANG-
BANG CONTROL
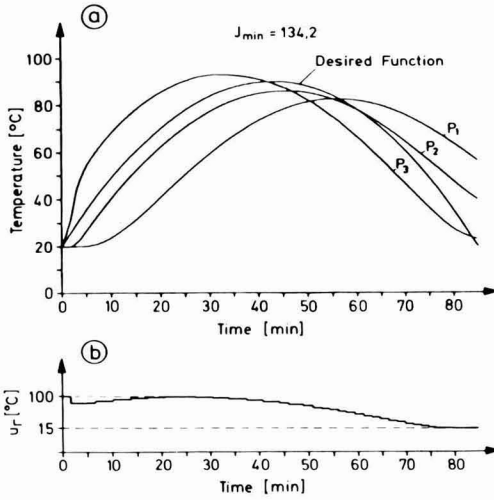
FIG. 9. a) OPTIMAL TEMPERATURE COURSE
(PARABOLIC DESIRED FUNCTION) b) QUASI-
CONTINUOUS CONTROL
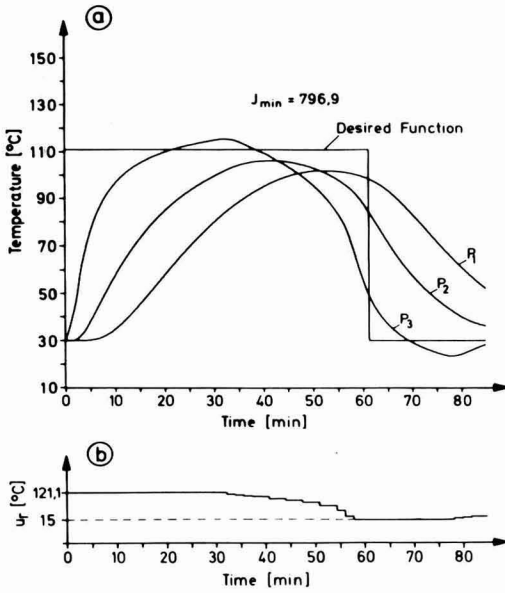
FIG. 10. a) OPTIMAL TEMPERATURE COURSE
(STEP-LIKE DESIRED FUNCTION) b) QUASI-
CONTINUOUS CONTROL

CONCLUSION

The optimal preservation of canned foods by sterilization poses a very complex optimization problem which is made easier by separating it into two single problems. The aim of the process engineering problem is to find the desired function w(t) which consequently results in an optimal compromise with regard to quality and durability. The control engineering problem consists in realizing this temperature variation at any spatial point as well as possible. The separation of this problem has the following advantage. One can imagine that various initial mediums require totally different sterilization processes. For the total problem, the complex optimization should be carried out for every kind of initial medium. By separation, it is sufficient to solve only the process engineering problem for the special desired function. The calculator program, which results from the solution of the control engineering problem, automatically delivers the modified control function for the wall-temperature $u_r(t)$. Here, the Bang-Bang control is suboptimal compared to the quasi-continuous solution but it is easier to realize in practice. The solutions show that a step-like function is only attainable near the container wall. Further, a parabolic-like temperature curve can be obtained for the whole interior of the can. This fact should be taken into consideration when solving the process engineering problem to determine the desired function w(t).

Finally, the results show another property of so-called distributed parameter systems. The optimal design of a control and the results obtainable are strongly influenced by the fact, that control is possible only via the boundaries and that the control variable u_r is limited.

LIST OF SYMBOLS

α coefficient of thermal conductivity

B_o, B_1 Bessel functions

$\varkappa_k$ are values resulting from the calculation of the consecutive zeros $B_o(\varkappa_k \cdot a) = 0$, $k = 1, 2, \ldots$

Eigenvalues

$$\lambda_{ik} = \left[\frac{(2i-1)\pi}{2\ell} \right]^2 + \varkappa_k^2 \qquad i,k = 1,2,\ldots$$

Green's function with respect to the initial condition

$$g_0(t,r,z,u,\rho) = \sum_{i=1}^{\infty} \sum_{k=1}^{\infty} \frac{2}{\ell a^2 B_1^2(\varkappa_k a)} e^{-\alpha \lambda_{ik} t} \; u \; B_0[(\varkappa_k a)\frac{u}{a}]$$

$$\cos [\frac{(2i-1)\pi}{2\ell} \rho] \; B_0[(\varkappa_k a)\frac{r}{a}] \cos [\frac{(2i-1)\pi}{2\ell} z]$$

Green's function with respect to the boundary condition

$$g_r(t-\tau,r,z) = \sum_{i=1}^{\infty} \sum_{k=1}^{\infty} A_{ik} \; e^{-\alpha \lambda_{ik}(t-\tau)} \; \cdot$$

$$\cos [\frac{(2i-1)\pi}{2\ell} z] \; B_0[(\varkappa_k a) \frac{r}{a}]$$

with

$$A_{ik} = \frac{8 \cdot \alpha \cdot \lambda_{ik} \cdot (-1)^{i+1}}{(\varkappa_k \cdot a)(2i-1)\pi \cdot B_1(\varkappa_k \cdot a)} \qquad i,k = 1,2, \dots$$

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APPENDIX

In the light of a strongly simplified example, the basic considera-
tions for the solution of the process engineering optimization problem
are given. In order to find a desired function w(t), $0 \leqslant t \leqslant t_e$, which is
purely time dependent, it is sufficient to regard a small volume ele-
ment of a special content. This volume element is assumed to be con-
trollable by the temperature in an arbitrary manner. For simplification
only one quality component is considered (s = 1).

As has already been shown in Section 2, a time discretization must
be carried out. The total time interval is divided into n = 50 equidis-
tant time intervals with $\Delta t = te/n = 1.7$ min. The temperature in each
time interval is assumed to be constant. In this way, one gets a
temperature vector $\underline{x}$ and a desired function vector $\underline{w}$ with the dimen-
sion

$$\dim(\underline{x}) = \dim(\underline{w}) = 50 \qquad \cdot \qquad \text{(A1)}$$

The performance index for one quality component has the form

$$J_D = f(c_{des} - c(\underline{x}, c_o, t_e)) \tag{A2}$$

Therefore, the function f weighs the difference between the real concentration and the desired concentration to the end time t_e. The necessary condition for an extremum of the functional J_D is that the first variation

$$J_{D1} = \sum_{j=1}^{n} \left. \frac{\partial f}{\partial x_j} \right|_{x_j = w_j} \cdot (w_j - x_j) \overset{!}{=} 0 \tag{A3}$$

equals zero for arbitrary variations $(w_j - x_j)$. The sufficient condition for a minimum of the functional J_D is

$$J_{D2} = \sum_{j=1}^{n} \left. \frac{\partial^2 f}{\partial x_j^2} \right|_{x_j = w_j} \cdot (w_j - x_j)^2 > 0 \tag{A4}$$

In order to avoid the resulting desired vector $\underline{w}$ being step-like, with only one heating and one cooling phase, the condition

$$|\Delta x| \leqslant G \; ; \Delta x = x_{j+1} - x_j \; ; j = 0,1, \ldots ,n\text{-}1 \tag{A5}$$

must be regarded. The value of G depends on the size of the can as well as on the coefficient of thermal conductivity.

The value of G can, in practice, be derived from the maximal value of the first order derivation of the temperature curve with respect to time at the center of the can.

A possible form for the function f of Eq. (A2) is

$$J_D = K_1(c_{des} - c(\underline{x}, c_o, t_e))^2 \tag{A6}$$

whereby K_1 is considered as contant.

Together with the basic assumptions given in (Texeira et al. 1969), the following equations can be stated for each time interval, j, $j = 1, \ldots ,n$

$$c_j = c_{j\text{-}1} e^{-\frac{\Delta t}{D_j}} \tag{A7}$$

$$D_j = D_r e^{-\frac{x_j \cdot T_o}{z}} \tag{A8}$$

c = are concentrations at time t_j and time t_{j-1}

D_j = death rate at temperature x_j, assumed to be constant over the regarded time interval

D_r = death rate at temperature $T_o = 121,1\,°C$

z = temperature difference affecting a tenfold change in D.

For the end time t_e one gets

$$c_n = c(\underline{x},\, c_o,\, t_e) = c_o \cdot e^{\left[-\Delta t \cdot \sum\limits_{h=1}^{n} \dfrac{1}{D_h(x_h)} \right]} \tag{A9}$$

For those intervals where Eq. (A5) is performed, the following equations ((A10) — (A13)) are valid. Should this not be the case, then the condition (A5) limits the possible change of the temperature in the regarded interval. The following condition results from Eq. (A3).

$$\left. \frac{\partial f}{\partial x_j} \right|_{x_j = w_j} \stackrel{!}{=} 0 \tag{A10}$$

which, together with Eq. (A6), leads to

$$\frac{2 \cdot K_1 \cdot \Delta t}{z}\, (c_{des} - c_n) \cdot \frac{c_n}{D_j(x_j)} = 0 \tag{A11}$$

and which in turn must be solved under condition (A5). The sufficient condition for a minimum is

$$\frac{1}{2}\, \frac{\partial^2 f}{\partial x_j^2} = \frac{K_1 \cdot \Delta t}{z^2} \cdot \frac{1}{D_j(x_j)^2} \left[c_n^2 \cdot (2\Delta t - D_j(x_j)) - \right.$$
$$\left. - c_{des} \cdot c_n \cdot (\Delta t - D_j(x_j)) \right] > 0 \tag{A12}$$

It can easily be seen that for $c_n = c_{des}$ the term

$$\frac{K_1 \cdot \Delta t}{z^2} \cdot \frac{1}{D_j(x_j)^2} \left[c_{des}^2 \cdot \Delta t \right] > 0 \tag{A13}$$

and consequently the functional, has a minimum for $x = w$.

RHEOLOGICAL BEHAVIOR OF ULTRA-HIGH TEMPERATURE STEAM INJECTED DAIRY PRODUCTS ON AGING[1]

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ABSTRACT

Milk (0.5, 3.25, and 10.5% fat) was processed in an ultra-high-temperature steam injection system. Process time-temperature relationships included 138, 143, and 149°C for 20.3s; 143 and 149°C for 6.9 s; and 149°C for 3.4 s. Product from each run was stored at 4, 24, and 40°C. Using four commercial viscometer assemblies the flow behavior constants of the power-law equation were determined for each treatment through 60 weeks of storage. Regression analysis of the calculated power law constants resulted in a graphical summary of the history of flow properties for each treatment.

Age-thickening (gelation) was found to be associated with process residence time, storage temperature, storage time, and fat level. Process temperature was not a significant factor. The shorter the residence time the faster gelation occurred. The earlier a sample gelled the faster its rate of gelation. For a given residence time, gelation developed first in samples of low percent fat and progressed through higher fat contents.

A gelation prediction nomograph based on fat level, residence time, and storage time is presented for a 24°C storage temperature.

INTRODUCTION

With the advent of commercially available aseptic packaging units, interest in a safe, consumer acdeptable sterile milk has increased.

To produce a safe product it is generally accepted that the product be free of pathogens and proliferating spoilage bacteria. Speck and Busta (1967) reported a 12 D time-temperature relationship for PA3679 spores of 3.42 s at 149 °C. They noted this treatment is well above requirement for destruction of spores of *C. botulinum*. Generally research on ultra-high-temperature (UHT) aseptic dairy products has been done at heat treatments close to the above. Hansen *et al.* (1980) reported the onset of gelation occurring which limited the storage life even when acceptable flavor remained. To produce a consumer acceptable product, gelation must either be prevented or delayed beyond the point of consumption.

Many investigators have formulated gelation theories. Hunziker (1949) attributed age-thickening to the colloidal swelling and hydration of proteins. Hostettler *et al.* (1957) suggested the cause was a reactivated protease. Murthy *et al.* (1958) proposed that a protease specific for β-casein either may have survived the heat treatment or may have been reactivated during storage. They noted that standard UHT time-temperature relationships reversibly or incompletely denatured the protease enzyme enabling it to hydrolyze the β-casein. They also found that higher heat treatments either completely inactivated or irreversibly denatured the enzyme.

Circle *et al.* (1964) reported the effectiveness of low concentrations of disulfide-reducing reagents (e.g. sodium sulfide, cysteine) in depressing gelation of protein globulins. This finding was interpreted as being indicative of participation of disulfide bonds in the gelation process.

Ashton and Jackson (1966) established a seasonal trend justifying the reactivation theory based on levels of native enzymes for seasonal variation.

Samuelsson and Holm (1966) reported an inverse relationship between the degree of protein decomposition and the time of onset of gelation. This was attributed to reactivation of proteolytic enzymes.

Corradini *et al.* (1967) found gelation with directly heated UHT milk but did not find it with indirectly heated milk. They concluded that the action of steam injection on β-lactoglobulin was in some way responsible for gelation.

Anon (1967) associated gelation with the ion balance of the milk which varies according to breed, cow health, and general hygiene. They also postulated that reactivation of enzymes originally present in the raw milk played an important part in gelation. By increasing the process heat treatment they reported no increase in viscosity.

Samel *et al.* (1971) postulated that the browning reaction producing lysine and arginine residues of K-casein molecules blocked the coagula-

tion mechanism. Andrew and Cheeseman (1971) proposed that a Maillard type reaction may be partly the cause of streak elec- trophoretic gel patterns indicating instability of cross linking between protein chains.

Bengtsson et al. (1973) suggested that psychrotrophic organisms in raw milk produce extracellular proteases which can survive UHT pro- cessing.

Zadow and Chituta (1975) also observed a seasonal trend similar to Ashton and Jackson (1966). This observation supported enzymatic theory but because the discrepancies in enzyme activity loss found at $40\,°C$ did not seem reasonable, they chose not to support any theory on gelation mechanism.

Möller et al. (1977 a,b, and c) did extensive work investigating the Maillard reaction occurring in stored UHT milk. They suggested that carbohydrate in the caseins resulting from the reactions imparts resistance towards enzymatic hydrolysis of the adjacent peptide bonds.

Some of the above theories have been questioned. Samel et al. (1962) reported that hydration and swelling of protein particles as suggested by Hunziker, could not account for the magnitude of the increase in viscosity upon aging or for the decrease of viscosity on increasing shear rates.

Catsimpoolas and Meyer (1970) demonstrated that 0.1% N-ethylmaleimide (a reagent capable of blocking sulfhydryl groups and, therefore inhibiting sulfhydryl-disulfide interchange) has no effect on gelation. This finding indicates that participation of disulfide bonds in the gelation process does not exist.

Andrews and Cheeseman (1971) found that calcium sensitivity giv- ing rise to product instability precedes gelation. Definite evidence was not found relating this sensitivity to the mechanism causing gelation. It was suggested that the sensitivity may be a valid measure for predicting the stability of the system.

Additional information about rheological behavior changes during aging may be useful in support of some of the above theories. The pur- pose of this study was to monitor the flow characteristics of UHT aseptic milk during aging and relate those changes to the process and handling conditions associated with them. It was the further intention of this study to identify optimum process and handling conditions which would hinder or even prohibit gelation. Additional evidence for one or more of the above theories was sought.

MATERIALS AND METHODS

Summer milks at three standardized fat levels (0.5, 3.25 and 10.5%) were processed in an UHT steam injection system as described by Hansen *et al.* (1980). Aseptic packaging was carried out in 250 ml 5 ply laminated cartons by a Tetra Brik filler Model AB-250[2] as described by Hsu (1970). Process time-temperature relations included 138, 143, and 149 °C for 20.3 s; 143 and 149 °C for 6.9 s; and 149 °C for 3.4 s. All six process conditions were used with each of the three fat levels. Process runs were duplicated. Samples from each of the 36 process runs were stored at 4, 24, and 40 °C. At four week intervals from zero through 60 weeks, samples from each storage temperature were randomly selected and examined. Visual observations were made through 92 weeks.

At the onset of this study it was assumed that all milk used would be of high quality. Therefore no attempt was made to randomly select which runs would be made on the six different milk supplies. Because of physical limitations the 36 runs were distributed between 6 days of system operation — different milk supply for each day. Separation of conditions per milk supply resulted from which residence time was to be used. In no case were duplicate runs made on the same milk supply.

Raw milk from each process run was pasteurized in a high-temperature-short-time (HTST) unit[3] at 71.7 °C for 15 s. Fresh HTST samples were tested and results used as the base for comparison (control). Just prior to processing the following plate counts were performed on all runs using standard procedures: standard, psychotrophic, lipolytic, and proteolytic psychrotrophic.

Measurements

Four viscometer assemblies were utilized. Over eighty percent of all measurements were made with the Brookfield Synchro-lectric viscometer Model LVT[4] with ultra-low adapter and bob (.367 $\leqslant \dot{\gamma} \leqslant$ 73.4). As the apparent viscosity (η)[5] increased for some samples, a small sample adapter with bobs number 18 (.396 $\leqslant \dot{\gamma} \leqslant$ 79.5) and 31 (.101 $\leqslant \dot{\gamma} \leqslant$ 20.3) was used. At later stages when milk

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[2]Tetra Brik (AB), Lund, Sweden.
[3]Chester-Jensen Model HTC-55, Chester, PA
[4]Brookfield Engineering Laboratories, Stoughton, Mass.
 Reproducibility of 2×10^{-5} Pas at minimum range of 0-.01 Pas.
[5]Symbols used throughout text are described in the nomenclature.

gelled, the Haake Model RV-1[6] with NV cup and bob was used ($19 \leqslant \dot{\gamma} \leqslant 3140$). The NV geometry consists of a double gap for the fluid produced by an open end hollow cylinder rotating within a cup of annular cross-section.

All viscometers were equipped with temperature control baths adjusted to 4 °C. To insure uniform sample temperatures all samples were refrigerated at 4 °C the night prior to measurement. For each process run at each sampling and measurement time two samples were measured. In all but two of the process runs only one sample was taken from each carton. For two cases (149 °C for 3.4 s; 0.5 and 3.25% fat) extracting two samples from one carton became necessary after 40 weeks because of a dwindling number of cartons. Since each treatment had a duplicate run, each treatment was examined four times at each sampling and measurement time. In all cases scale readings were taken twice at each viscometer speed — once while increasing speed sequentially and once while decreasing speed sequentially. Thus, for each process treatment at each sampling and measurement time at each viscometer speed, eight scale readings were recorded. The number of viscometers and the number of speeds used for each sample at any storage time depended on the sample's viscous nature. As a sample became more viscous (onset of gelation) the number of observations increased.

Sample extraction from the carton was done with a calibrated theft tube. Units were shaken slightly when taken from storage prior to refrigeration to reduce the effect of fat separation. The slight shaking was not used when samples were noted to be increasing in viscosity — gelation. In these cases the top layer of fat was removed before the sample was taken.

Calculations

The power law model[7] commonly used for non-Newtonian foods is expressed as:

$$\tau = K \, \dot{\gamma}^N \qquad . \tag{1}$$

The value of N usually decreases for foods as the material becomes more solid in nature. K increases with increasing solid character.

In Eq. 1, γ is also equivalent to the velocity gradient. For rotational viscometers $\dot{\gamma} = - r \, (dw/dr)$ where w is the angular velocity and r is the

[6]Haake, Inc., Saddlebrook, N.J.
[7]Also known as the Ostwald-de Waele-Nutting Model

radial distance to the element of fluid being considered. Eq. 1 then becomes:

$$\tau = k \ (\text{-r} \ \frac{dw}{dr})^N \tag{2}$$

The torque at the fluid element is represented by

$$M = 2 \ \pi \ r^2 h \ \tau \qquad . \tag{3}$$

By substituting Eq. 2 into Eq. 3, integrating from the bob to the cup wall and taking logs of each side, the relationship given by Van Wazer *et al.* (1963) results:

$$\log \Omega = \log \left[\frac{N \left(\dfrac{1}{(R_b)^{\frac{2}{N}}} - \dfrac{1}{(R_c)^{\frac{2}{N}}} \right)}{2 \ (2 \ \pi \ hK)^{\frac{1}{N}}} \right] + \frac{1}{N} \ (\log M) \qquad . \tag{4}$$

This is the slope-intercept form of the equation of a straight line with log M versus log Ω. These relations hold for any single gap rotary viscometer and were used with the measurements obtained from the Brookfield LVT utilizing the UL and small sample adapters.

Similar relations were derived for the Haake using the NV double gap cylinder adapter. Utilizing superposition and recognizing that M is the total of the torques in both gaps results in:

$$\log \Omega = \log \frac{N}{2} \left[\frac{\left(\dfrac{1}{(R_b)^{\frac{2}{N}}} - \dfrac{1}{(R_c)^{\frac{2}{N}}} \right) + \left(\dfrac{1}{(R'_b)^{\frac{2}{N}}} - \dfrac{1}{(R'_c)^{\frac{2}{N}}} \right)}{(2 \ \pi \ hK)^{\frac{1}{N}}} \right]$$

$$+ \frac{1}{N} \log M \qquad . \tag{5}$$

Note R'_b and R'_c correspond to the dimensions of the inner gap. The torque represented by each scale reading was determined by a percent of full scale torque of the particular instrument. When the Brookfield instruments were used, τ was calculated using conversion units

supplied by the manufacturer for the particular bob. This was possible because the fluids were close to Newtonian, when these instruments were used. In the case of the Haake instrument, end effects were considered negligible.

Once K and N were determined from Eq. 4 and 5 by the slope intercept method the flow behavior for each condition was known. At specified values of $\tau, \dot{\gamma}$ was calculated by Eq. 1 and η determined by

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{6}$$

As an additional tool for sample comparison the product of N and K was utilized. This results from Eq. 1 by taking the 1st derivative of τ with respect to $\dot{\gamma}$ and specifying $\dot{\gamma} = 1$ s^{-1}. On the τ versus $\dot{\gamma}$ flow curve this is the slope at the point where $\dot{\gamma} = 1$ s^{-1}.

RESULTS AND DISCUSSION

A high quality milk supply was furnished in most cases. There were selected standardized milks which were less than high quality.

The standard plate count on the raw milk ranged from 5.9×10^2 to $\geqslant 3 \times 10^6$; psychotrophic plate count, 2.1×10^2 to $\geqslant 3 \times 10^6$; lipolytic plate count, 1.2×10^1 to 3×10^6. No statistically reliable relationship could be established between time of onset of gelation, plate counts, and process heat treatment. Samples with high counts gelled no faster than their duplicates with low counts.

Sampling for viscometry measurement became a particular problem throughout the study. Initial planning called for the use of a theft tube for sample extraction from an undisturbed carton. At approximately storage time (t_s) of 12 weeks a cream layer developed for those samples stored at 24 and 40 °C. The separated fat layer, upon refrigerating, solidified and became particularly unmanageable. Since gentle shaking prior to refrigeration eliminated the problem, this then became the procedure for all samples except those that gelled.

Gelled samples were refrigerated without agitation and the top thick fat layer was removed prior to sampling.

The effect of process temperature at a given residence time (r_t) was not a significant factor (.05 level) contributing to any increase in η. This was probably because of the narrow process temperature range and the large difference in heat treatments resulting from variation of

r_t. Storage-temperature (Ts) proved to be the most important factor affecting the increase in η. As an example, Fig. 1 represents the typical trend for all treatments with $r_t = 3.4$ s. The change in η at $\dot{\gamma}$ of 20 s^{-1} is most dramatic at storage temperature of 24 °C where there was a rapid rise after 20 weeks storage. Samples stores at 4 °C increased only slightly while those at 40 °C actually decreased slightly with storage time.
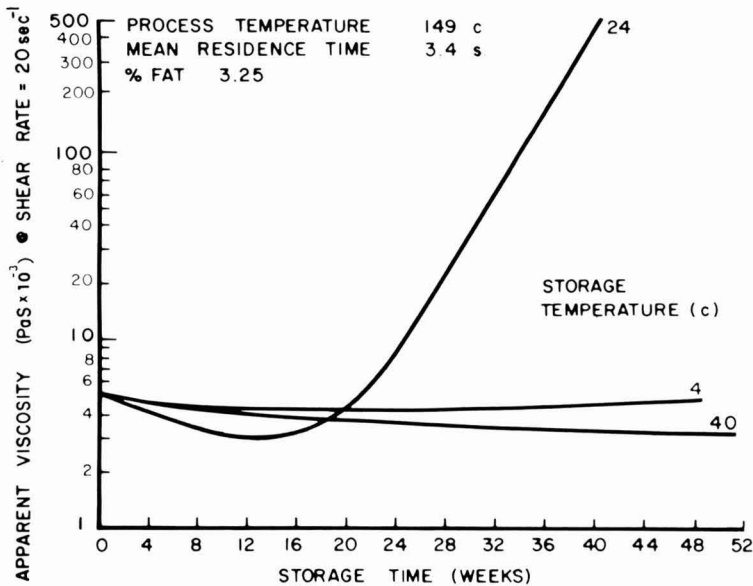
FIG. 1. THE EFFECT OF STORAGE TEMPERATURE AND
STORAGE TIME ON THE APPARENT VISCOSITY OF UHT
PROCESSED WHOLE MILK AT RESIDENCE TIME 3.4 s AND
PROCESS TEMPERATURE OF 149 °C

Figure 2 demonstrates the effect of r_t on η ($\dot{\gamma} = 20$ s^{-1}) and a T_s of 24 °C. Samples processed at 3.4 s demonstrated age-thickening by 24 weeks. Samples processed at 6.9 s were gelling between 40 and 48 weeks while those processed at 20.3 s showed early signs of gelation at 90 weeks. Figure 2 also shows η of the pasteurized product (control) always less than the UHT treated product. The gelation visible line indicates the viscosity when laboratory personnel could visibly detect a difference in the consistency of the product.

To develop as complete a flow model as possible prediction
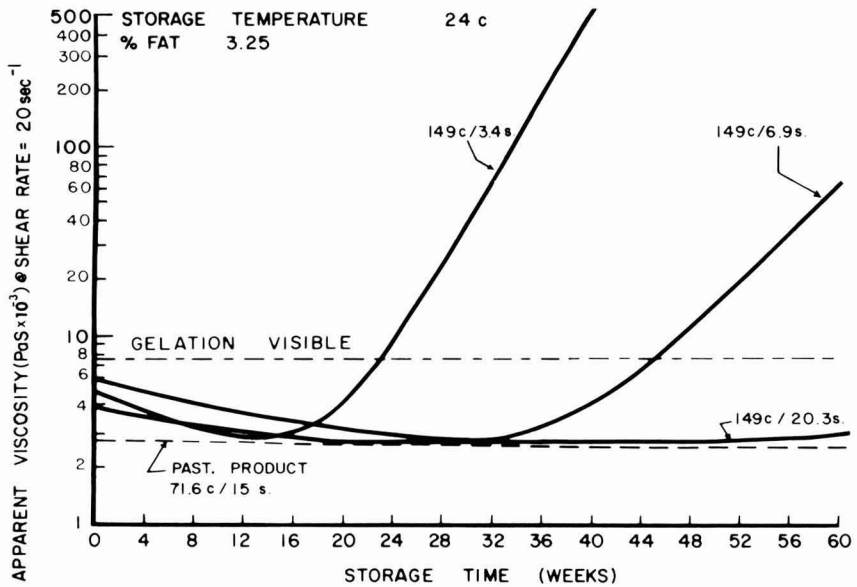
FIG. 2. THE EFFECT OF PROCESS RESIDENCE TIME AND
STORAGE TIME ON THE APPARENT VISCOSITY OF WHOLE
MILK STORED AT 24 °C

equations developed by least squares regression were used to show the
relation of t_s versus N and t_s versus log K. At the onset of gelation a
dramatic change occurred in the flow properties. A modified quadratic
model (phase before gelation, phase after) demonstrated the change in
properties most significantly. The model used for predicting N was:

$$N = a + b\,t_s + c\,t'_s + d\,t''_s \tag{7}$$

where

t'_s = 0 if T_s = 4 or 40 °C
     or if r_t = 20.3 s
     or if T_s = 24 and t_s < 20 for r_t = 3.4 s
     or if T_s = 24 and t_s < 40 for r_t = 6.9 s

t'_s = $(t_s\text{-}20)^2$ if T_s = 24, t_s > 20 for 3.4 s
t'_s = $(t_s\text{-}40)^2$ if T_s = 24, t_s > 40 for 6.9 s.

t''_s = 0   if T_s = 24
        and t_s > 20 for r_t = 3.4 s
        or t_s > 40 for r_t = 6.9 s.

otherwise
$$t_s'' = (t_s)^2.$$
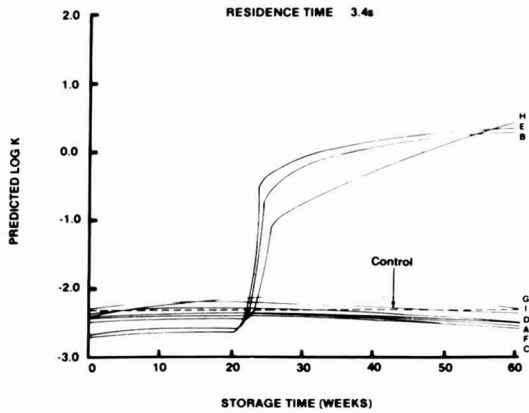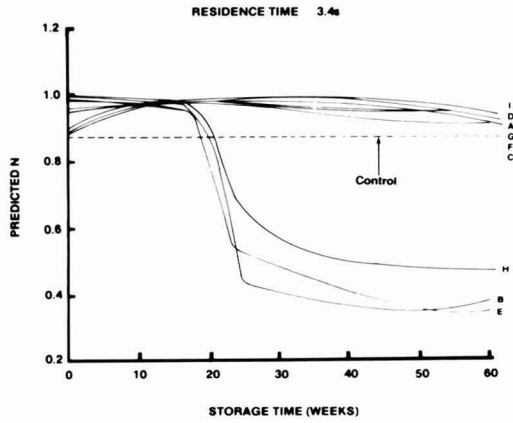
FIG. 3. THE EFFECT OF STORAGE TEMPERATURE AND
% FAT ON POWER LAW MODEL CONSTANTS OF UHT
PROCESSED DAIRY PRODUCTS WITH RESIDENCE
TIME OF 3.4 s

(A = 0.5% fat, 4 °C storage temperature: B = 0.5%, 24 °C; C =
0.5%, 40 °C; D = 3.25%, 4 °C; E = 3.25%, 24 °C; F = 3.25%,
40 °C; G = 10.5%, 4 °C; H = 10.5%, 24 °C, I = 10.5%, 40 °C).
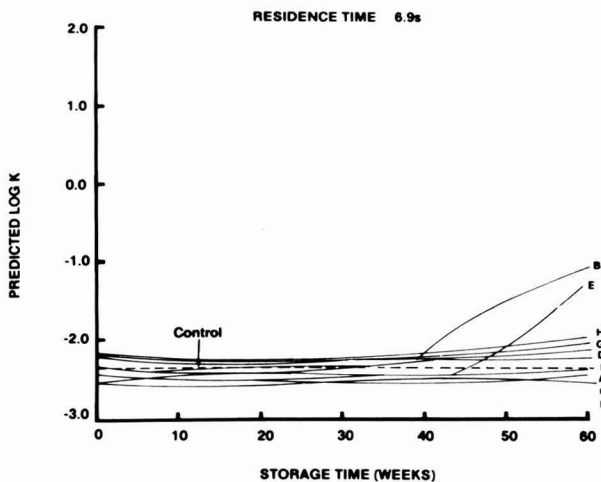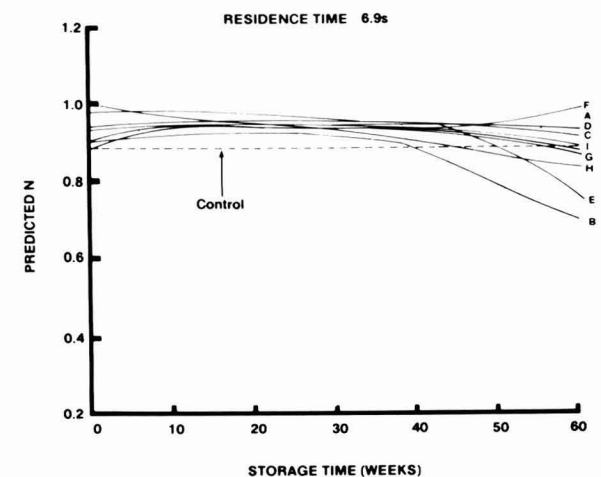
FIG. 4. THE EFFECT OF STORAGE TEMPERATURE AND
% FAT ON POWER LAW MODEL CONSTANTS OF UHT PRO-
CESSED DAIRY PRODUCTS WITH RESIDENCE TIME OF 6.9 s

(A = 0.5% fat, 4 °C storage temperature; B = 0.5%, 24 °C; C = 0.5%,
40 °C; D = 3.25%, 4 °C; E = 3.25%, 24 °C; F =3.25%, 40 °C; G = 10.5%,
4 °C; H = 10.5%, 24 °C; I =10.5%, 40 °C).
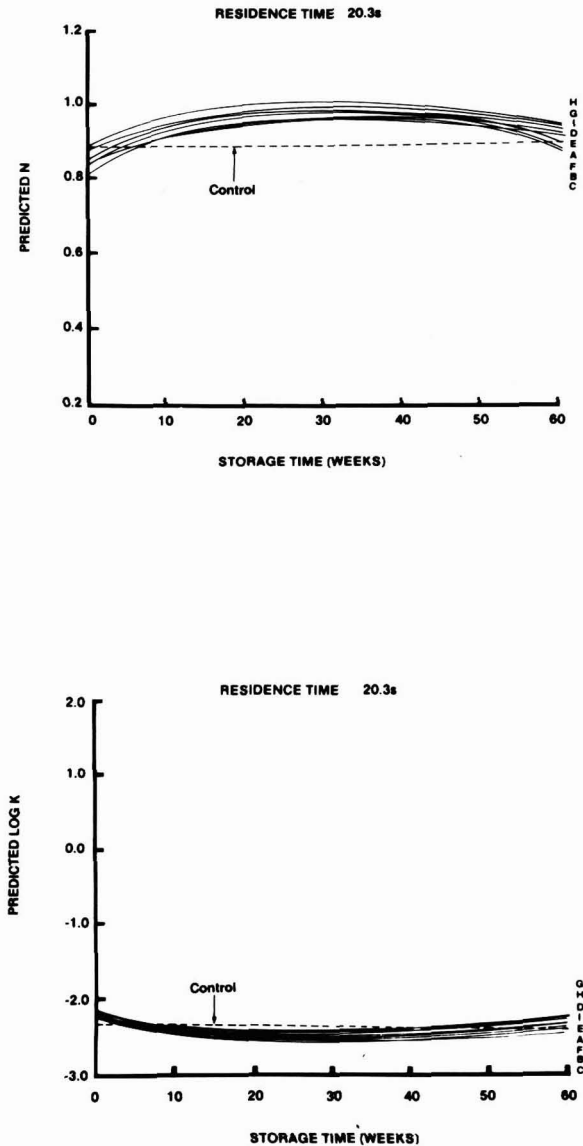
FIG. 5. THE EFFECT OF STORAGE TEMPERATURE AND %
FAT ON POWER LAW MODEL CONSTANTS OF UHT PRO-
CESSED DAIRY PRODUCTS WITH RESIDENCE TIME OF
20.3 s

(A = 0.5% fat, 4 °C storage temperature; B = 0.5%, 24 °C; C = 0.5%,
40 °C; D = 3.25%, 4 °C; E = 3.25%, 24 °C; F = 3.25%, 40 °C; G = 10.5%,
4 °C; H = 10.5%, 24 °C; I = 10.5%, 40 °C.
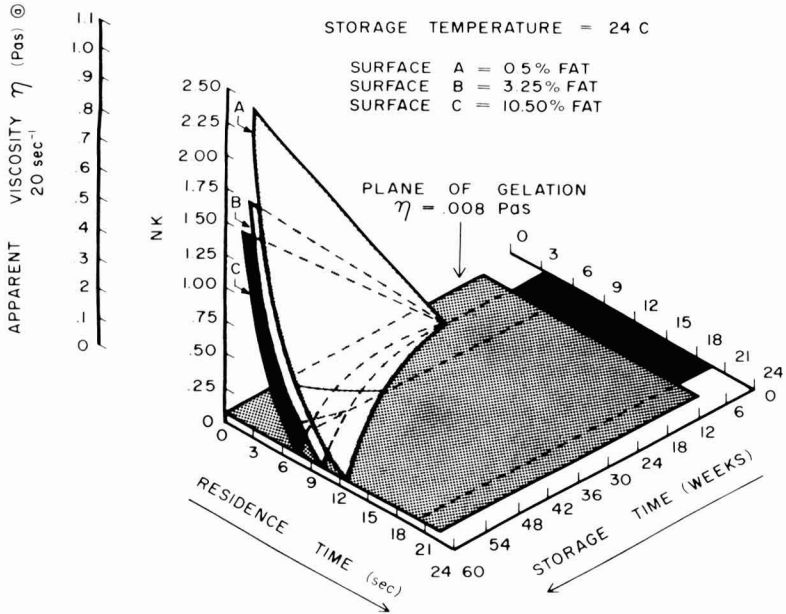
FIG. 6. THE EFFECT OF RESIDENCE TIME, STORAGE TIME,
AND % FAT ON APPARENT VISCOSITY AND NK FOR UHT
PROCESSED MILK STORED AT 24 °C

The same model was used to predict log K. The study consisted of
8496 individual observations. R-square values for η, N and log k for
samples that gelled ranged between .87-.92. Estimates of a, b, c, and d
and corresponding standard errors are tabulated (Swartzel 1979).

Figures 3, 4, and 5 show the relations graphically. Again samples
stored at 24 °C were the only ones in which flow properties changed
dramatically. As the sample gelled it became more solid, the N
decreased and the K increased. As can be noted in Fig. 2, the change
occurred sooner with the smallest r_t. It is also noted (comparing Fig. 3,
4, and 5) that the smaller the r_t the faster gelling progressed after the
onset.

As a reference, mean values of N and log K for the pasteurized pro-
ducts (control) are shown in Fig. 3, 4, and 5. They are N = .893 with σ
= ±.0098; log K = -2.357 with σ = ± .029.

Least squares multiple regression was again utilized to demonstrate
the relation of r_t, t_s, and % fat on the flow properties of samples stored
at 24 °C. The model

$$NK = a + bt_s + ct_s^2 \tag{8}$$

was utilized for all treatments held at 24 °C except after the beginning of gelation. With the onset of gelation ($r_t = 3.4$ s at $t_s > 20$ weeks and $r_t = 6.9$ s at $t_s > 40$ weeks) the term $t_s^2 >$ was dropped from Eq. 8. This led to a better statistical model than using the basic quadratic for all treatments. Estimates of a, b, and c and corresponding standard errors have been tabulated (Swartzel 1979).

Utilizing the prediction equations originating from Eq. 8, Fig. 6 was generated. Apparent viscosity ($\dot{\gamma} = 20$ s^{-1}) is also shown with the NK axis. Until gelation begins the relative viscosities of the three fat contents are of similar magnitude as the pasteurized products. That is, η of the 10.5% fat product is greater than the 3.25% fat which is greater than the 0.5% fat. Upon gelation the pattern reverses. Again as demonstrated with the predicted N's and predicted log K's, the smaller the r_t, the sooner gelation begins, and the faster it progresses. The line of visible gelation shown in Fig. 2 is now depicted as the plane of gelation.

With associated equations, Fig. 6 presents estimated flow characteristics of the samples stored at 24 °C and aids in the prediction of flow properties for other treatments at various storage times.

Continued visual inspection of those samples stored at 4 °C have shown that for a r_t of 3.4 s and a fat content of 0.5% gelation begins at approximately 92 weeks. This suggests that other nomographs (as Fig. 6) could be produced for other storage temperatures. This also indicates that an empirical relation could be established including storage temperature as an independent variable. Additional studies would be required to establish an upper limit since through 92 weeks no gelation occurred at 40 °C.

Since there appeared to be no relationship between plate counts and the onset of gelation, this study is inconsistent with that Bengtsson *et al.* (1973). It can only be concluded that milk of the quality described herein does not gel at a rate based on initial counts. This would suggest that the major mechanism of gelation is not based on enzyme survival. However, the data presented does align well with the reactivated enzyme theory. Since no gelation occurred at 40 °C, the Maillard reaction blockage (at higher storage temperatures as reported by Samel *et al.* 1971; and Möller *et al.* 1977 a, b, and c) is supported by this study. A more detailed investigation involving rheological behavior of the gel formation is reported in a companion paper (Swartzel *et al.* 1980). A discussion of the effect of fat on gelation rate and additional gelation theory comments are reserved for that paper.

ACKNOWLEDGEMENT

NOMENCLATURE

a,b,c,d	predicted regression model constants	variable
h	height of liquid on inner cylinder	m
M	torque at the fluid element	N·m
N	flow behavior index, a power-law parameter	dimension-less
NK	slope at the point $\dot\gamma = \mathrm{s}^{-1}$ on τ versus $\dot\gamma$ plot	Pa·s
K	Consistency index, a power-law parameter	Pa·s
R_b	radius of the inner cylinder or bob (associated with outer gap for double gap cylinder)	m
R'_b	radius of the innermost cylinder of a double gap rotary viscometer	m
R_c	radius of the outer cylinder or cup (outermost cup for double gap rotary viscometer)	m
R'_c	radius of the outer cup for the inner gap of a double gap rotary viscometer	m
r	radius varying over gap of the fluid	m
r_t	process residence time	s
T_s	storage temperature	°C
t_s	storage time	weeks
t'_s, t''_s	modified storage time for quadratic regression models	weeks
$\dot\gamma$	shear rate	s^{-1}
η	apparent viscosity defined as the shear stress divided by the shear rate	Pa·s
π	3.1415926 . . .	dimension-less
σ	*standard deviation*	
τ	shear stress	N/m²
Ω	angular velocity of the inner cylinder	rad/s
-r(dw/dr)	velocity at the rotating spindle wall	s^{-1}

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RHEOLOGICAL MODELING OF UHT MILK GELS USING A CONE AND PLATE CREEP-RELAXATION TEST[1]

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ABSTRACT

The gelation of UHT steam injected dairy products was investigated by determining rheological model constants. Fat percentages of the products were 0.5, 3.25 and 10.5. Each product was processed at 143 °C for 6.9 s and stored at 24 °C. At four week intervals, from 55 to 85 weeks storage, cone and plate creep-relaxation data was collected. Three element rheological models were developed for each test date. Empirical relationships were derived relating the elastic and viscous constants to storage time for each fat content. The strongest parameter for all samples was the series elastic constant. The viscous constant was next with the Kelvin elastic constant being much weaker. All constants for all samples increased over storage time as gelling progressed (apparent viscosity increased). The lower the percent fat, the sooner gelling started and the faster the rate. The gelation of UHT milk was examined and found to depend heavily on the percent fat.

INTRODUCTION

In an earlier paper (Swartzel *et al.* 1980), the gelling phenomenona of UHT milk was studied and flow behaviors were established. This paper dealt with process and storage conditions which most affected the initiation and rate of gelling. The data tended to support the reactivated enzyme theory with Maillard reaction blockage at high storage

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temperatures (Samel *et al.* 1971). In an effort to add support to this theory, rheological characteristics of the gel were investigated. Since chemical and enzymatic theories (Swartzel *et al.* 1979) involve various elastic and viscous bonding mechanisms, tagging the gelling changes by mathematical modeling should give insight to the theory. Similar work has been carried out relating the action of rennet on the physical changes in milk (Scott Blair and Burnett 1958, 1959a and 1959b).

Special problems existed for the rheological parameter determinations. Naturally occurring UHT milk gels have extremely weak model constants and gelling occurs very slowly when compared to rennet gels. Sample volume of the actual gel was also limited. With these facts in mind the development of an appropriate test was imperative.

The purpose of this study was to develop apparatus for determining rheological model constants of UHT milk gels, and to relate the model constants to existing gelling mechanism theory.

MATERIALS AND METHODS

Summer fluid milk at three standardized fat levels (0.5, 3.25, and 10.5%) was processed in an UHT steam injection system as described by Hansen *et al.* (1980). Process time-temperature was 143 °C for 6.9 s. Aseptic packaging was carried out in 250 ml 5 ply laminated cartons by a Tetra Brik filler Model AB-250[2] as described by Hsu (1970). Process runs were duplicated. Samples from each of the three duplicate runs were stored at 24 °C. Measurements were made beginning at 55 weeks of storage through 85 weeks.

Measurements

Three viscometer assemblies were utilized throughout the course of this study. All measurements were made with the Wells-Brookfield Synchrolectric Micro viscometer Model 2.5 × LVT[3] open face with cone/plate (C/P) adapter. (Note Fig. 1 for description). Sixty percent of the measurements were made with cone spindle and plate SCP-0.8 ° with $\alpha = 10.8°$, and r = 3.3 cm. Other C/P assemblies used were unit CP-7A ($\alpha = 0.8°$, r = 2.4 cm), and unit CP-7 ($\alpha = 1.565°$, r = 2.4 cm).

During all measurements temperature was maintained at 4 °C. Calibration was performed prior to measurements at each storage
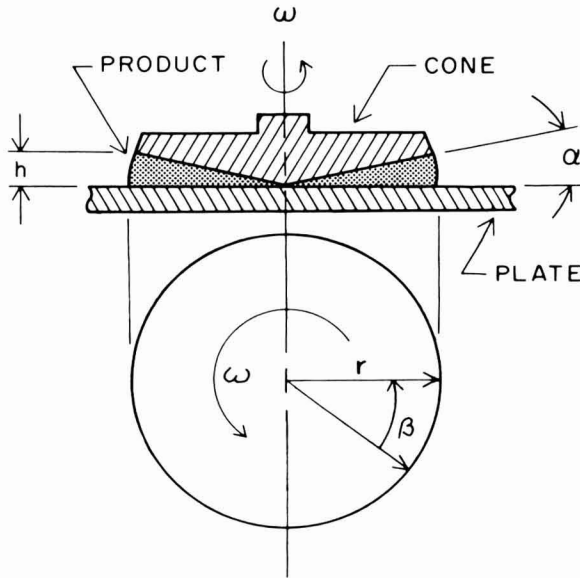
FIG. 1. CONE AND PLATE ASSEMBLY

time (Anon 1973). One sample was taken from each carton. Duplicate cartons were examined for each fat content at each sampling and measurement time. To ensure uniform sample temperatures, all samples were refrigerated at 4 °C the night prior to measurement.

Sample extraction from the carton was done with a calibrated theft tube. Any fat which had separated and solidified was removed prior to measurement.

Creep-relaxation measurements were made as described by Patton (1966). Volumetric samples of 1 ml for the CP-7 and CP-7A and 1.5 ml for the SCP −0.8° were utilized throughout the study. Prior to the introduction of the milk sample, the helical spring of the viscometer was rotated manually to a prescribed position (percent of full scale torque). After introduction of the milk into the sample cup, the spring was released. The rate of scale reading decay was noted by the use of a stop watch. The general creep-relaxation curve is shown in Fig. 2.

Calculations

One of the most common models which has been used to predict
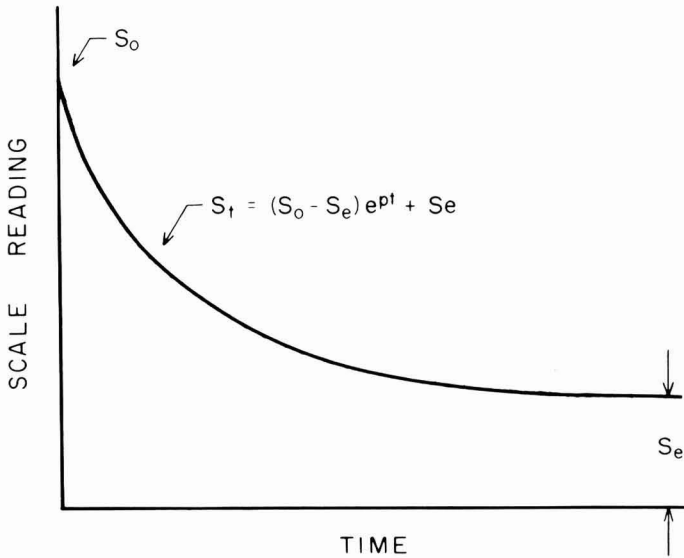
FIG. 2. CONE AND PLATE CREEP-RELAXATION CURVE

rheological behavior in many materials is the 4-element Burgers model (Mohsenin 1970). This model is composed of a spring and dashpot in series (Maxwell model) connected to a spring and dashpot in parallel (Kelvin model). By addition of strains through the models, noting stress is the same in every segment connected in series, and combining the model segments into one equation the result is

$$\ddot{\varepsilon} + \frac{E_r}{\eta_1}\, \dot{\varepsilon} = \frac{1}{E_o}\left[\ddot{\sigma} + \left(\frac{E_o}{\eta_2} + \frac{E_o}{\eta_1} + \frac{E_r}{\eta_1}\right)\dot{\sigma} + \left(\frac{E_o}{\eta_1}\, \frac{E_r}{\eta_2}\right)\sigma\right]. \tag{1}$$

This equation is given by Morrow (1965) and discussed by Mohsenin (1970) for total axial stress (σ) and total axial strain (ε). Since the torque as shown in Fig. 2 never decreased to zero, the terms with η_2 drop out. Equation 1 then reduces to

$$\ddot{\varepsilon} + \frac{E_r}{\eta_1} \dot{\varepsilon} = \frac{1}{E_o} \left[\ddot{\sigma} + \left(\frac{E_o}{\eta_1} + \frac{E_r}{\eta_1} \right) \dot{\sigma} \right] \tag{2}$$

which is for the 3 element model shown in Fig. 3.
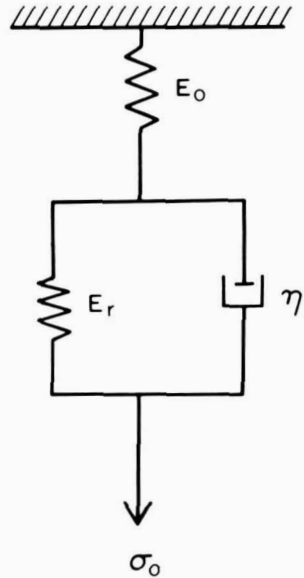
FIG. 3. 3-ELEMENT
RHEOLOGICAL MODEL

The shear stress exerted on the test fluid when the scale reading is S_t, as shown by Patton, (1966) is

$$\sigma_t = \frac{3 \, M_{100} \, S_t}{200 \, \pi \, r^3} . \tag{3}$$

The curve shown in Fig. 2 was represented by the equatiion

$$S_t = (So\text{-}Se) \, e^{pt} + Se \tag{4}$$

where "p" was determined by least squares regression analysis. Substituting Eq. 4 into Eq. 3, taking the first and second derivatives, and substituting these into Eq. 2 yields

$$\ddot{\varepsilon} + \frac{E_r}{\eta_1} \dot{\varepsilon} = .00477 \frac{p\, M_{100}\, (So\text{-}Se)}{r^3} \left[\frac{p}{E_o} + \frac{1}{\eta_1} + \frac{E_r}{E_o \eta_1} \right] e^{pt} \qquad (5)$$

By determining the particular and homogeneous solutions to Eq. 5 the strain (ε_t) was found to be

$$\varepsilon_t = \frac{\sigma_e\, (E_o + E_r)}{E_o\, E_r} \left(\frac{\sigma_e\, (E_o + E_r)}{E_o\, E_r} + \frac{C}{p^2 + \dfrac{E_r}{\eta_1}\, p} \right) e^{\left(\dfrac{-E_r}{\eta_1} \right)}$$

$$+ \frac{C e^{pt}}{p^2 + \dfrac{E_r\, p}{\eta_1}} \qquad (6)$$

where

$$C = .00477 \frac{p\, M_{100}\, (So\text{-}Se)}{r^3} \left(\frac{p}{E_o} + \frac{1}{\eta_1} + \frac{E_r}{E_o \eta_1} \right)$$

and

$$\sigma_e = .00477 \frac{M_{100}\, Se}{r^3} \quad .$$

Equation 6 is a representation of the rheological parameters in terms of stress (σ) and strain (ε). Spring relaxation violates the small strain definition ($\varepsilon = \tan \varepsilon$). Therefore, Eq. 6 cannot be used in the sense of calculating strains in this case. It is, however, valid for force-deformation, torque-angular rotation, or angular ratio relationships. In this study, ε_t of Eq. 6 was replaced by the angular ratio-relationship, β/α (Fig. 1). The rheological parameters (E_o, E_r, and η_1) were then estimated.

Normally, the initial elastic parameter E_o is equal to the initial stress divided by the initial strain. In this case

$$E_o = \frac{\sigma_o}{\beta_\circ}\, \alpha \quad . \qquad (7)$$

Since at relaxation time zero ($\beta = 0$) no determination could be made, a reference of 2 s was chosen so that $\beta_o = \beta_{2s}$. By recognizing that $360° = 115.5$ scale readings then

$$\beta_t = 3.116 \, (S_o\text{-}S_t) \tag{8}$$

With σ referenced to the scale readings at 2 s and substituting Equations 3, 4 and 8 into Eq. 7,

$$E_o = .00153 \frac{S_o \, M_{100}}{r^3 \, (S_o\text{-}S_{2s})} \quad \alpha \qquad . \tag{9}$$

At the end of the relaxatiion decay the scale reading becomes constant (equilibrium). Since angular displacement associated with the two elastic parameters are additive,

$$\frac{\sigma_e \, \alpha}{\beta_e} = \frac{E_o \, E_r}{E_o + E_r} . \tag{10}$$

Substituting Eq. 3, 4, and 8 into Equation 10 results in

$$\frac{E_o E_r}{E_o + E_r} = .00153 \, \frac{M_{100} Se \, \alpha}{(So\text{-}Se) \, r^3} \qquad . \tag{11}$$

Inserting values for E_o from Eq. 9, E_r from Eq. 11, and β/α (ε_t) depicted in Fig. 1 into Eq. 6 and using an iterative approach (Swartzel 1979) η_1 was found.

Shear rate ($\dot{\gamma}$), shear stress (τ), and apparent viscosity (η) were determined by a procedure similar to that described by Patton (1966). Noting Fig. 1,

$$\dot{\gamma} = \frac{\omega}{\alpha} \tag{12}$$

where

$$\omega = \frac{d\beta}{dt} = \frac{-dS_t}{dt} \, \frac{2\pi}{115.5} \qquad .$$

By substituting the first derivative of Eq. 4 and noting a decreasing angular rational rate during relaxation (minus sign) Eq. 12 becomes

$$\dot{\gamma} = -.054 p (S_o\text{-}S_e) e^{pt} \; \alpha^{-1}. \tag{13}$$

Shear stress is determined by Eq. 3 and 4 at the same relaxation time as $\dot{\gamma}$. The apparent viscosity (η), determined at the same $\dot{\gamma}$ as in Eq. 13, then becomes

$$\eta = \frac{\tau}{\dot{\gamma}} \; . \tag{14}$$

RESULTS

Estimates of the rheological model (E_r, E_o, η_1) and η (@ $\dot{\gamma} = 1 \; s^{-1}$) were obtained on each of the three fat levels and their duplicates. To establish visual trends the statistical model $X = a + bt + ct^2$ was used; where, X = interested parameter, t = storage time, and a, b and c are model constants to be fitted. Estimates of a, b and c and corresponding standard errors are tabulated (Swartzel 1979). Figures 4, 5, 6, and 7 are visual representation of these statistical models.
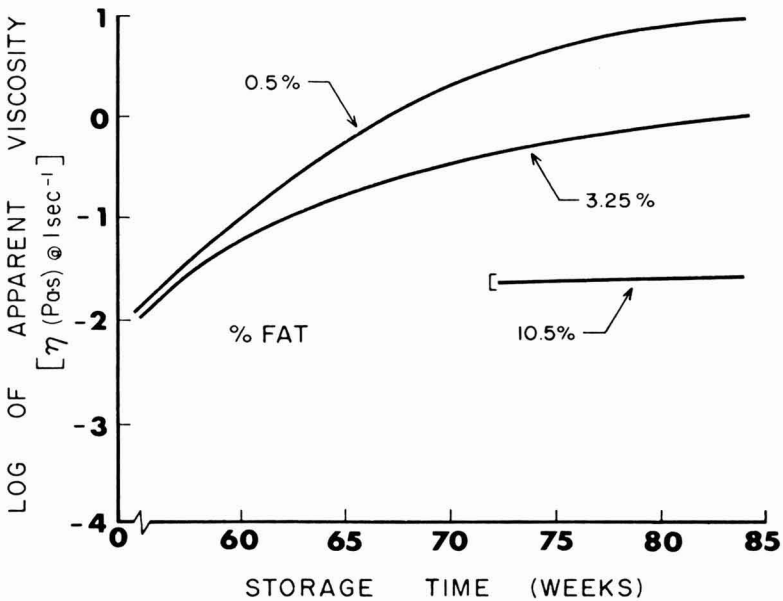
FIG. 4. APPARENT VISCOSITY @ 1 s⁻¹ DURING STORAGE TIME
OF THREE FAT LEVELS OF UHT STEAM INJECTED MILK
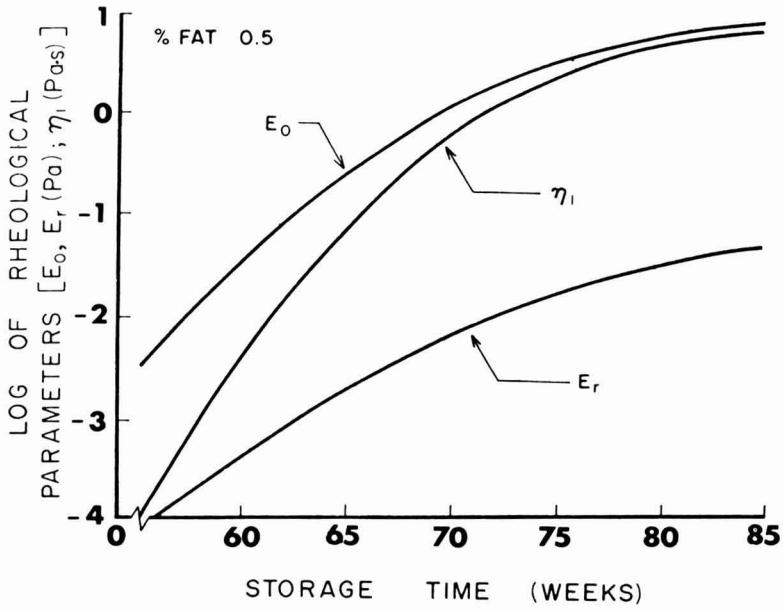
FIG. 5. CHANGE OF RHEOLOGICAL PARAMETERS DURING
STORAGE TIME OF 0.5% FAT UHT STEAM INJECTED MILK
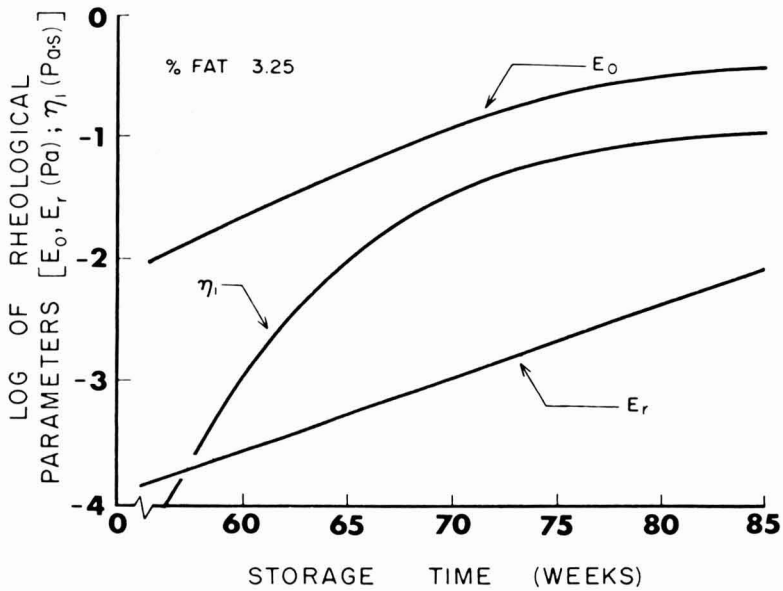
FIG. 6. CHANGE OF RHEOLOGICAL PARAMETERS DURING
STORAGE TIME OF 3.25% FAT UHT STEAM INJECTED MILK
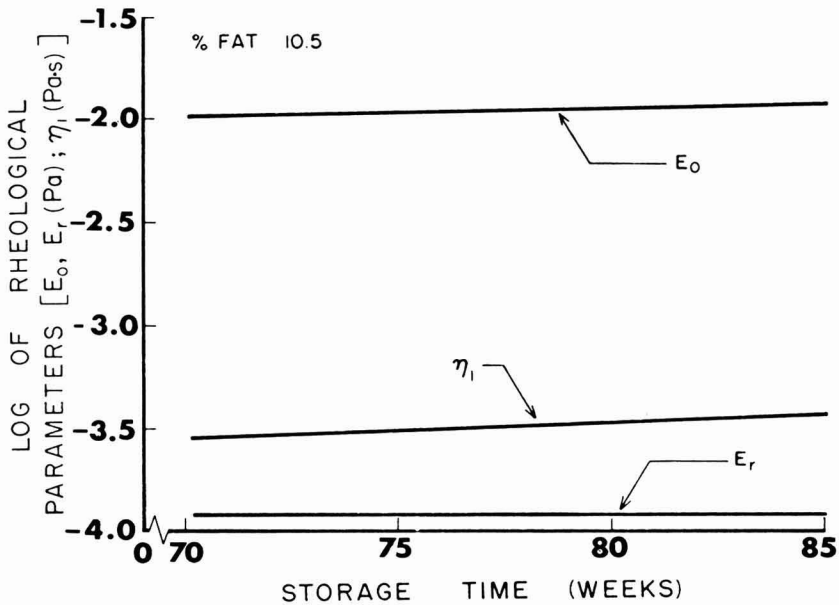
FIG. 7. CHANGE OF RHEOLOGICAL PARAMETERS DURING
STORAGE TIME OF 10.5% FAT UHT STEAM INJECTED MILK

Figure 4 depicts η at $\dot{\gamma}$ of 1 s^{-1} for each of the three fat levels. No
data was collected with the C/P on 10.5% product until it was 72
weeks into storage. Before this time the product was not viscous
enough to obtain accurate readings. Through 84 weeks the 10.5% pro-
duct only increased slightly in apparent viscosity, indicating only mild
gelation. With decrease in percent fat the apparent viscosity increased.
Also the rate of increase of η of any sample increased with a decrease
in fat level. This information confirms and adds to the findings
reported earlier (Swartzel et al. 1980).

Functions fitted to estimates of the rheological parameters E_o, E_r,
and η_1 for the three fat levels are shown in Fig. 5, 6, and 7. These
curves increased as the fat level decreased similar to the trend of ap-
parent viscosity. For each fat level the series elastic constant (E_o) was
the strongest with the viscous constant (η_1) next, and the Kelvin
elastic constant (E_r) the weakest. As gelation progressed E_o and η_1
became similar in magnitude, increasing in a decreasing rate.

The analysis indicated that as the gelation structure progressed the
E_r parameter became stronger. This can be associated with the increas-
ed solid nature of the samples at the very low shear rates present at

the end of the creep-relaxation test. In the 10.5% product E_r was extremely weak. Stress decayed to near zero. When fat percent was lower and gelling progressed, E_r became stronger.

DISCUSSION

During aging the percent fat played an important role in the rate of gelation. Throughout the storage, coalescing and clustering of fat and eventually separation occurred. Microscopic examination at 20 °C demonstrated that coalescing was predominant with clustering present but limited. The rate of fat separation was temperature dependent with little or no visual separation at refrigeration temperatures (Swartzel 1979).

It may be postulated that as the gel structure builds, it is hampered by constant interference of fat globules. This creates a disorder of linkage throughout the gel pattern and increases as the percent fat increases. Concentration of enzymes is known to occur on the fat globule membrane (Jenness and Patton 1959; Webb and Johnson 1965). As the fat coalesces and clusters during the early stages of aging the effective surface area of the globules is reduced. The enzyme exposed to protein with gelling potential is reduced because of the decreased surface area of the fat globules. Although with increased percent fat it might be thought that a high concentration of effective reactivated enzymes would be present, the increased coalescing, clustering, and bulk interference effects of higher percent fat may nullify this influence.

It may also be postulated that low fat milk, in later aging stages, possess higher effective globule surface areas within the bulk media than milk with somewhat higher percent fat. This is based on the assumption that during aging the globules in the low percent fat milk would have to travel much further to coalesce or cluster with other globules; thereby retaining their homogeneous diameter longer than those in the higher percent fat milk.

The combined effect of fat interference and reduction of effective reactivated enzyme may explain why higher percent fat milk gels slower than lower percent fat milk.

The C/P creep-relaxation test for model constants may prove useful for other products in research and quality control. The commercial availability of the equipment and the ease and quickness of the test are definite advantages. The main disadvantage is the iterative procedure required for determination of η_1. It may be possible to eliminate this problem for a given product by establishing an empirical relation-

ship for η_1 based on the other model constants and/or process and storage conditions. Also, this apparatus may be used with products which require more complicated modeling than the 3-element model used in this study.

ACKNOWLEDGEMENT

NOMENCLATURE

a,b,c	predicted regression model constants	variable
Eo	initial elastic parameter	Pa
Er	retarded elastic parameter	Pa
p	exponential power constant for equation of relaxation curve	dimensionless
M_{100}	Maximum torque (torque @ 100% of scale reading)	N/M
r	cone radius	m
S_{2s}	scale reading after 2 s relaxation	scale units
So	scale reading @ time zero	scale units
Se	scale reading @ equilibrium	scale units
S_t	scale reading @ time t	scale units
t	time (subscript) regression parameter	s
X	(Eo, Er, η_1, or η)	as noted for each parameter
α	angle between cone and plate	rad.
$\dot{\gamma}$	shear rate or velocity gradient	sec^{-1}
β	rotation angle of cone	rad.
ε	strain (normal or shear)	m/m
ε_t	strain @ time t	m/m
$\dot{\varepsilon}$	strain rate (dε/dt)	m/ms
$\ddot{\varepsilon}$	time-rate of change of the strain rate	m/ms^2
η	Newtonian viscosity coefficient	Pas
η_1	Viscosity coefficient of dashpot in the Kelvin portion of the rheology model	Pas
η_2	Viscosity coefficient of the dashpot in the Maxwell portion of the rheology model	Pas
π	3.1415926 . . .	dimensionless
σ	normal or shear stress	N/m^2

$\dot{\sigma}$	rate of stress $(d\sigma/dt)$	
$\ddot{\sigma}$	time-rate of change of rate of stress $(d^2\sigma/dt^2)$	N/m^2s^2
σ_e	total stress @ equilibrium	N/m^2
τ	shear stress	N/m^2
ω	angular velocity of cone	rad/s

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LIERATURE ABSTRACTS

ABSTRACTS FROM THE JOURNAL OF FOOD SCIENCE

COMPUTER CALCULATION OF THERMAL PROCESSES. M.A. Tung and T.D. Garland. J. Food Sci. *43*, 365-369.

A series of computer oriented procedures was developed to facilitate the rapid handling of heat penetration data while providing estimates of the process time required to achieve a specified lethality at a single point within the product for a particular product, container size, fill and processing conditions. Accepted thermobacteriological equations were employed with variation in the heating rate used to establish confidence limited on processes. This statistical approach was considered more pragmatic than the traditional use of the slowest or mean single value of the heating rate in sterilization calculations while the amount of heat penetration data necessary for process determination was minimized. Varying computer program versions permit the application of these process determination procedures to high or low acid products that are convection or conduction heating providing they exhibit single or broken straight-line heating curves.

MEASUREMENT OF THE THERMO-PHYSICAL PROPERTIES OF COMMON COOKIE DOUGH. F.A. Kulacki and S.C. Kennedy. J. Food Sci. *43*, 380-384.

Despite the volume of research done on food properties, the literature reports little related to the baking industry, particularly confectionary dough. This paper reports results of an experimental study of thermal conductivity, specific heat, density and thermal diffusivity of two different types of cookie dough. Thermal conductivity of both doughs were moderately dependent on temperature. Thermal diffusivities of each type dough were evaluated using experimental values of thermal conductivity, specific heat and density. Total experimental uncertainty in the thermal diffusivity was estimated as 0.134 for the AACC formula and 0.15 for the hard-sweet formula dough.

ENZYMATIC SOLUBILIZATION OF LEAF PROTEIN CONCENTRATE IN MEMBRANE REACTORS, R.E. Payne, C.G. Hill Jr. and C.H. Amundson. J. Food Sci. *43*, 385-389.

The enzymatic solubilization of an insoluble alfalfa protein fraction was studied in a batch reactor and in a continuous flow semi-batch membrane reactor. The "white" cytoplastic protein fraction obtained by heat precipitation was solubilized using the proteolytic enzyme trypsin. Batch studies were carried out at pH 7.7 with a trypsin to substrate ratio of 2×10^{-3} (by weight). At 27, 37 and 47°C the hydrolysis reaction was found to be first-order in substrate concentration. The activation energy was 23.25 kcal. At 47°C the conversion level was 97% after 4 hr. For the membrane reactor system, the effects of the variables pH, temperature, substrate concentration, and trypsin to substrate ratio were evaluated with the aid of factorial design methods. The optimum operating conditions for the membrane reactor studies were 47°C at pH 7.7 with a trypsin to substrate ratio of 4×10^{-3} at a 0.5% solids concentration level. The hydrolysis process was enhanced if the reactor was operated in a washout mode prior to the addition of enzyme suggesting that soluble low molecular weight inhibitors were washed out

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of the reactor with the permeate. 55% of the total amount of insoluble starting material
appeared as products in the permeate after 4 hr of operation under the conditions
stated above.

APPARATUS FOR STUDYING FOULING OF HEATED SURFACES BY
BIOLOGICAL FLUIDS, A.C. Ling and D.B. Lund. J. Food Sci. *43*, 390-393.

A laboratory-scale double tube heat exchanger was developed to study fouling
phenomena of biological fluids. The test fluid is continuously circulated through the
system exposing the solution to an electrically-heated stainless steel section. The
stainless steel section is 0.635 cm (0.25 in.) diameter and is located on the center-axis of
2.54 cm (1 in.) diameter glass-pipe. The heat transfer characteristics of the system were
determined and egg albumin solution was used to test the applicability of the system
for studying fouling. The system was found to be stable and reproducible. The system
can be used to study the effects of operational variables (such as surface temperature,
velocity and fluid temperature) and fluid characteristics on fouling rates. The test sur-
face can be easily interchanged to study surface characteristics also.

KERNEL HARDNESS OF WILD RICE AS AFFECTED BY DRYING AIR
TEMPERATURE AND MOISTURE GRADIENT. M.A. Wirakartakusumah and D.B.
Lund. J. Food Sci. *43*, 394-396.

Samples of wild rice (38—40% moisture, wet basis) were dried to 7-8% moisture using
three different drying air temperatures and relative humidities, namely, 110 °C—63%
relative humidity, 110 °C—6% relative humidity, and 60 °C—20% relative humidity.
Samples were subsequently hulled immediately (hulled hot) or hulled after storing 24 hr
at room temperature (hulled cold). Evaluations included determination of hulling effi-
ciency, kernel hardness and degree of gelatinization. Hulling was most efficient when a
high drying air temperature was used and where the kernels were hulled immediately
after drying. Total yield was not affected by temperature gradient or moisture gradient
which the wild rice kernels experienced during drying. Head yield was maximum when
wild rice was dried under conditions of a high drying air temperature and low moisture
gradient, and when it was hulled hot, immediately after drying. The kernel hardness
test confirmed that kernels were harder when dried under high temperature, low
moisture gradient conditions. Degree of gelatinzation studies confirmed that about
90—95% of the starch in kernels dried under these conditions was gelatinized. The
gelatinization of starch was influenced by both temperature and rate of drying. Suscep-
tibility of kernels to cracking increased with increasing magnitude of moisture gradient
within kernels. The best processing conditions for wild rice using criteria previously
described consist of drying under high air temperature and a low moisture gradient,
and hulling the kernels hot, immediately after drying.

EVALUATION OF BIRD CHILLER WATER FOR RECYCLING IN GILBERT
FLUMES. H.S. Lillard, USDA. J. Food Sci. *43*, 401-403.

For tests of the feasibility of recycling water from the bird chiller, water samples from
eight points in a commercial processing plant were evaluated by the following criteria:
fecal coliform levels, total aerobic counts, salmonellae incidence and levels. BOD and
COD. Probably due to a great dilution factor, water from the gizzard splitter was of
better microbiological quality for recycling than water from the bird chiller but was
higher in level of organic contaminants. If bactericidal treatment is omitted, water from
the gizzard splitter would be preferable for recycling the giblet flumes. If bacterial
treatment is used, water from the bird chiller would be preferable because of its lower
level of organic contaminants.

REDUCTION IN CHEMICAL OXYGEN DEMAND OF OZONATED SUGAR SOLU-
TIONS BY CHARCOAL. RL.H. Walter and R.M. Sherman. J. Food Sci. *43*, 407-410.

Sugar solutions at a concentration of 0.10% were subjected to low-level ozonation, in
order to oxidize the solute directly or indirectly to the corresponding aldonic acid. Char-
coal adsorption of the nonsaccharides was more efficient after ozonation. The chemical
oxygen demand of a standard gluconic acid solution was reduced to 10% of its original
concentration by charcoal alone.

OPTIMAL MODES OF OPERATION FOR MICROWAVE FREEZE DRYING OF
FOOD. T.K. and J.D. Ford and D.C.T. Pei. J. Food Sci. *43*, 648-649.

In freeze-drying, the quality of the resulting product often determines the attrac-
tiveness of the drying process. Therefore, the temperature profiles within the material
are as important as the total drying time required. For microwave assisted freeze dry-
ing, it has been indicated both experimentally and in theoretical analysis that 130 v/cm.
Beyond this point melting to the ice core and/or overheating in the dried layer will oc-
cur. However, the drying time can be further reduced by 20% if a simple preprogramm-
ed stepwise adjustment of the applied field is introduced.

HYDROLYSIS OF LACTOSE IN ACID WHEY USING β-GALACTOSIDASE AD-
SORBED TO A PHENOL FORMALDEHYDE RESIN. M.R. Okos, E.A. Grulke and
A. Syverson. J. Food Sci. *43*, 566-571.

The feasibility of using immobilized enzymes for the commercial hydrolysis of lactose in
acid whey was investigated. The areas of enzyme kinetics, enzyme adsorption, mass
transfer, reactor design and economics were considered. All reaction studies were car-
ried out at 40°C, pH 4. Adsorption of β-galactosidase into a porous phenol for-
maldehyde resin (Duolite ES-762) followed an S shaped isotherm at low concentrations
and adsorption increased with temperature at 0, 24 and 26°C. Up to 0.6g of Wallerstein
enzyme preparation (4043 activity units) were adsorbed to 1 g of dry resin. Adsorption
had little effect on enzyme activity. An integral reactor of immobilized β-galactosidase
retained nearly full activity for over 120 days of continual hydrolysis of lactose in acid
whey and pure lactose solutions. Throughout the long term study, mold was effectively
inhibited by the use of 0.1% w/v of potassium sorbate and CO_2. Based on 120-day
catalyst life and the integral reactor data for Duolite ES-762 loaded with 0.23g en-
zyme/g dry resin, the catalyst cost for the hydrolysis of 1 lb of lactose is 1.65 cents at
40% conversion using 50 mesh resin.

EFFECT OF CHEMICAL COMPOSITION ON COMPRESSIVE MECHANICAL
PROPERTIES OF LOW ESTER PECTIN GELS. W.J. Kim, V.N.M. Rao and C.J.B.
Smit. J. Food Sci. *43*, 572-575.

Low-ester pectin gels were prepared by acid and a combination of acid and ammonium
hydroxide demethylation. The chemical compositional data such as percent esterifica-
tion, acid amide, free carboxyl, galacturonic acid and apparent molecular weight of the
gels were determined. The maximum stress, modulus of elasticity and relaxation time
prior to fracture of the low ester pectin gels were evaluated using a universal testing
machine. Significant correlations were obtained between (a) relaxation time during frac-
ture propagation and percent esterification; (b) maximum stress and molecular weight;
and (c) maximum stress and percent acid amide groups. It was also found that percent
acid amide groups in the pectin contributed to the strength of the gels over and above
the effect of decreasing percent free carboxyl groups.

FREE AMINO ACID AND RHEOLOGICAL MEASUREMENTS ON HYDROLIZED
LACTOSE AND CHEDDAR CHEESE DURING RIPENING. J.C. Weaver, M. Kroger
and M.P. Thompson. J. Food Sci. 43, 579-583.

Three lots each of conventional Cheddar and hydrolyzed lactose Cheddar cheese (HLCC)
were analyzed for free amino acid content and examined for texture properties at
28-day intervals during an eight-month ripening period. The average increases in total
free amino acids in the control and the HLCC were from 500 μg/g and 14.564 μg/g,
respectively. Aspartic acid, proline and histidine were the only amino acids not
significantly different between the two cheeses. Arginine, glycerine, alanine, methionine
and isoleucine increased slowly during ripening and accounted for 12% of the total free
amino acids. Valine tyrosine, phenylalanine, glutamic acid, leucine and lysine showed
greater increases and accounted for about 80% of the total amount of free amino acids
at all stages of ripening. Leucine increased the most, from 23.2 μg/g to 1959.5 μg/g in
the control cheese and from 68.2 μg/g to 2875.9 μg/g in the HLCC. Arginine and
methionine were the only two amino acids whose mole percentages did not significantly
differ between the control and the HLCC. The mole percentages of aspartic acid,
tyrosine, histidine, alanine, proline, lysine and glutamic acid decreased during the
study in both the control and the HLCC. The mole percentage of leucine increased the
most, from 5 to 15% in the control cheese and from 12% to 25% in the HLCC. The tex-
ture measurements for toughness, mechanical hysteresis, and modulus of elasticity
showed significant (α0.05) treatment effects, but the changes during ripening were not
significant. The toughness of the HLCC after eight months was 69% higher than in the
control. Stiffness (modulus of elasticity) was 54% less and the damping capacity
(mechanical hysteresis) was 93% more in the HLCC cheese than in the control. The dif-
ferences in the texture measurements (toughness, mechanical hysteria) was 93% more in
the HLCC cheese than in the control. The differences in the texture measurements
(toughness, mechanical hysteresis, and modulus of elasticity) on the controls and HLCC
were greater after 3 or 4 months and remained relatively constant thereafter. Therefore,
proteolysis is related to texture and quality of cheese and accelerated ripening does
seem to enhance desirable body and texture characteristics.

SLOPES OF MOISTURE SORPTION ISOTHERMS OF FOODS AS A FUNCTION
OF MOISTURE CONTENT. P. Viollaz, J. Chirife and H.A. Iglesias. J. Food Sci. 43,
606-608.

Values of the isotherm factor,$(5/a_w/\partial X)_T$, for various dried foods (apple, beef, cod, car-
rots, fennel, peas, peanut, potato and trout) were calculated and reported as a func-
tion of moisture content. The effect of sorption hysteresis on the isotherm factor was
also calculated for some foods. It is hoped that these data may be useful for adequately
interpreting experimental results on rates of moisture sorption and desorption in dried
foods.

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SINGLE CELL PRO-
TEIN CURD. T.D. Tsintsadze, C.H. Lee and C.K. Rha. J. Food Sci. 43, 625-630.

The microstructure and the physical and mechanical properties of yeast protein curds
were investigated by using the scanning electron microscope and the Instron Universal
Testing Machine. Model 1122. Heat treatment of the protein dispersion prior to
precipitation did not remarkably influence the structure and waterholding capacity of
the curds. Yeast protein curd precipitated with calcium had a micro-network surface
which was not apparent in the isoelectric point precipitated curd. The calcium
precipitated curd had a low value of hardness, high water-holding capacity, low value of
adhesiveness and high cohesiveness compared to the isoelectric precipitated curd. The

precipitated curd also showed a strong structural potential which was revealed upon freezing. The freeze-thawing of the curd in general increased hardness and springiness but decreased the adhesiveness and cohesiveness. The physical, structural and mechanical properties of yeast protein curds were compared with those of soybean protein curds studied previously.

KINETIC PARAMETERS FOR THERMAL INACTIVATION OF PANTHOTHENIC ACID. D.J. Jamm and D.B. Lund. J. Food Sci. *43*, 631-633.

Decimal reduction values (D values) and activation energies for destruction of pantothenic acid (PA) in KH phthalate buffer, pH 4.5 and 6, in meat puree and in pea puree at temperatures between 118—143 °C were determined using the thermal death time tube method and the Arrhenius equation. Samples were assayed according to AOAC (1975) microbiological method for pantothenic acid. Activation energies ranged from 20,000—38,000 cal/mole. As pH increased from 4 to 7, activation energies also increased. The D_{121} values ranged from 4—32 hr for buffered systems and from 38—40 hr for food systems. These large D values for foods would indicate that PA is not very heat labile and is more stable in these food systems compared to buffered systems.

ALFALFA PROTEIN FRACTIONATION BY ULTRAFILTRATION. D.E. Eakin, R.P. Singh, G.O. Kohler and B.E. Knuckles. J. Food Sci. *43*, 544-547.

Ultrafiltration was used to fractionate the chloroplastic and the soluble protein components of alfalfa juice. In agreement with trends predicted by theoretical considerations, permeate volume flux increased with increased feed velocity, pressure and temperature, and decreased with increased feed concentration. The recovery of the protein in the permeate increased with increased feed velocity and decreased temperature and was essentially insensitive to feed concentration. Under conditions of low turbulence, the protein recovery decreased with increased pressure.

OPTIMIZATION OF A LACTOSE HYDROLYSIS PROCESS. F.C. Knopf, M.R. Okos, D.A. Fouts and A. Syverson. J. Food Sci. *44*, 896-900.

A design equation was developed which represents the enzymatic hydrolysis of lactose in whey. Various processing schemes and conditions were studied to determine an optimal cost for the hydrolysis process. Consideration was given to catalyst costs, product value and reactant concentration.

DIFFERENTIAL SCANNING CALORIMETRY (DSC) STUDIES ON THE FREEZING PROCESSES OF WATER-ETHANOL MIXTURES AND DISTILLED SPIRITS. K. Koga and H. Yoshizumi. J. Food Sci. *44*, 1386-1389.

DSC measurements of the freezing of water-ethanol mixtures in various ethanol concentrations were made in a temperature range from +20 °C to −160 °C. One exotherm was observed in the freezing process of each mixture. The freezing point, heat associated with the freezing process and kinetic constant of freezing (k_f) were determined over the entire range of the ethanol concentration from the DSC thermogram. Log k_f was plotted as a function of the reciprocal of the absolute temperature (T^1) of the mixtures. The freezing process of the mixtures in an ethanol concentration of 0~75 (V/V)% obeyed first order kinetics but at a concentrationm above 75%, the freezing did not do so. The slope of log k_f versus 1/T plots (d log hg/dT¹) of the mixture at the concentration below 75% was determined. The concentration below 75% was divided into four

ranges (0~18.5%, 18.5~40%, 40~60%, 60~75%), from a variation in the slope of the linear plots. The results of the DSC measurements of freshly distilled spirits (raw whiskey from barley malt and grain spirits) coincided with those of simple water-ethanol mixtures. It is considered that the freezing reaction of distilled spirits proceeded by the same mechanism as that of the simple mixture of the corresponding ethanol concentration. Structures of distilled spirits as a function of ethanol concentration were discussed in terms of the interaction of water and ethanol molecules.

REFRIGERATED SEAWATER SPRAY SYSTEM MODEL FOR SHRIMP
VESSELS. E. Kolbe. J. Food Sci. 44, 1420-1424.

A mathematical simulation of refrigerated seawater spray cooling of shrimp onboard a vessel is described. The simulation describes compressor and chiller performance and heat transfer between shrimp and recirculating spray water. It also accounts for conduction through the hull and for pump energy. A series of temperature measurements onboard vessels verified the model which can be used to evaluate the sensitivity of cooling rates to changes in equipment of operating procedures.

NATURE OF THE DEPOSIT ON REVERSE OSMOSIS MEMBRANES DURING
CONCENTRATION OF PECTIN/CELLULOSE SOLUTIONS. A Watanabe, S.
Kimura, Y. Ohta, J.M. Randall and S. Kimura. J. Food Sci. 44, 1505-1509.

Model solutions consisting of varying concentration of the major fouling components of mandarin orange juice, pectin and cellulose, were studied. The deposits were scraped from the membranes and analyzed for weights and concentrations of pectin and cellulose. Suspensions of cellulose with no pectin caused no decrease in permeation flux, but large declines in permeation flux occurred during concentration of solutions of pectin and cellulose. Weights and concentrations of both components in the deposits changed with altering operating conditions and decreased with increasing mean flow velocity. Resistance of the deposit to water permeability (R_g) was related to the concentration of solute at the membrane surface (C_g) by the empirical equation, $R_g = \alpha\, Cg^{1.7}$ (α = constant). Accumulation of deposit on the membrane was in two forms: viscous layer and film layer. The former was a common form of deposit accumulated during the concentration of model solution under all conditions, but the latter was formed only under conditions at higher permeation flux with moderately high mean flow velocity. The film layer was insoluble in water but soluble in 0.1N HCl solution.

A NEW PACKAGING METHOD FOR PEANUTS AND PECANS, C.E. Holaday, J.L.
Pearson and W.O. Slay, USDA. J. Food Sci. 44, 1530—1533.

An economical method for packaging low-moisture peanuts and pecans for long-term storage is presented. The method utilizes the CO_2 adsorption properties of these commodities and involves placing them in plastic pouches impervious to air and CO_2, flushing them with CO_2 and then heat-sealing the pouches. CO_2 is adsorbed into the pores of the commodities forming vacuums inside the pouches. Both shelled, raw and shelled, roasted and blanched peanuts were quality factors for 12-months: pecans were protected for 27 wk and roasted salted-in-the-shell peanuts were protected for 27 wk and roasted salted-in-the-shell peanuts were protected for 4 months. This method of packaging process perishable commodities for extended periods of time, is inexpensive and requires little energy.

ANALYSIS AND RENOVATION OF RETORT COOLING WATER. R.H. Walter, R.L. Mitchell, D.L. Downing.

Retort effluent was found by gravimetry to contain generally 20 40 ppm lubricant in the form of a stable micro-emulsion. Adsorption-filtration columns of aluminum filings, aluminum powder or charcoal removed lubricant from this effluent at a rate of 18-20 lb per 10^2 lb of adsorbent, with possibly greater efficiency being realized by proper attention to a small number of performance characteristics. Aluminum had an advantage over charcoal, because of its ease of regeneration in situ by elutriation with an organic solvent. Absorbance data, obtained from a series of standard emulsions, showed that accurate measurements of lubricant concentrations could be made photometrically, in the absence of interfering matter, at the levels found in retort effluent. Retort effluent was renovated experimentally by a two-stage adsorption filtration process, involving aluminum (1st stage) and charcoal (2nd stage). The combined eluate had an absorbance (A) of zero until the break-through volume when turbidity increased rapidly at A = 0.005-0.010.

A THEORETICAL COMPARISON OF LOSSES IN FOODS UNDER FLUCTUATING TEMPERATURE SEQUENCES. T.P. Labuza, J. Food Sci. 44, 1162-1169.

The prediction of shelf life of foods is dependent on certain physical factors which change as a function of time in distribution. Temperature has been the most studied factor. With respect to fluctuating temperature studies in all cases in the literature, a zero order reaction rate has been assumed. In actuality, many foods degrade by first order rates thus the equations for prediction of losses are incorrect if applied. New equations for first order degradation have been developed. Comparisons for losses in a theoretical temperature distribution show that for less than 50% degradation the losses are about the same for zero and first order at anytime, thus determination of the order is not critical. This study shows that the temperature sensitivity (Q_{10}) of the reaction is very important in making predictions.

MATHEMATICAL MODELING OF THE VISCO-ELASTIC CHARACTERISTICS OF EGG YOLK. S.C. Sharma. J. Food Sci. 44, 1123-1128.

Dynamic shear moduli and particle size distribution of egg yolk were studied, as function of pH, NaCl concentration and holding time. Levels of the treatment variables were selected according to a three-factor central composite design. Mathematical formulations were developed to predict the selected visco-elastic characteristics. Using these equations optimum levels of the treatment variables were predicted. A pH range of 6.15 ± 0.45 and NaCl concentration of 5.00 ± 0.75% were found to be optimum to maintain preprocessing elastic modulus to a minimal level. The equations developed herein can be used for optimization of shelf life and functional characteristics of egg yolk.

BOOK REVIEW

Coffee Technology. Michael Sivetz and Norman W. Desrosier, AVI Publishing Co., Westport, Connecticut. 1979. 716 pp. $49.00.

This book is an updating of the two-volume set, "Coffee Processing Technology," written by Sivetz and Foote and published in 1963. It also effects a condensation of the earlier material to a single, but large, volume.

The coverage is encyclopedic, dealing with all aspects of the coffee industry. This includes historical aspects, horticulture, roasting, packaging, production of instant coffees by spray drying or freeze drying, coffee chemistry, and physiological effects. The approach is almost entirely descriptive, serving as an effective introduction to the industry and a useful reference on industrial practices for persons not already highly familiar with growing, handling and processing of coffee. This reviewer picked up a large number of interesting incidental facts relating to coffee, of which he had not previously been aware. To my knowledge, there is no other existing book which plunges as comprehensively into all aspects of coffee processing.

For purposes of this review, I shall concentrate upon the chapters dealing with instant-coffee manufacture, drying and decaffeination, since these relate most closely to the subject matter of this journal. Here we have the problem that breadth discourages depth, and the descriptive approach does not effectively convey a sound understanding of the mechanistic factors at play. One prime example occurs in the area of retention and manipulation of coffee volatiles during instant-coffee processing. Volatiles loss during spray drying is interpreted in terms of the Rayleigh equation for batch vaporization, along with a tacit assumption of ideal solutions. Such an approach is utterly unrealistic, since it ignores the facts that activity coefficients of coffee volatiles in solution were quite large (up to 1000 and greater) and that volatiles retention is very much a rate-governed process. The substantial recent literature stemming from the selective-diffusion concept of Thijssen receives no mention. Another example occurs for percolation (extraction), where we are not told the engineering reasons for using the rotating-bed-position, pseudo-countercurrent system, and where process analysis is carried out by an equilibrium triangular diagram without consideration of rate effects. The discussion of freeze drying does not go into the factors governing drying rates and does not delve into the important question of the effects of drying temperature on color and collapse tendencies.

Another unfortunate aspect is that the book is rather isolated from pertinent literature that does not deal directly with coffee. The spray drying references are all quite old and do not include any of the work of Thijssen and associates. For drying in general, there is no tie-in with the conventional literature, such as standard material presented in the books by Keey and Treybal, or in the section by McCormick in Perry's *Chemical Engineers' Handbook*. Despite the facts that about 30% of instant coffee is freeze dried and that considerable recent engineering development went into freeze-dried coffee, there is no tie-in with the abundant freeze-drying literature.

Several recent important processing developments are treated only briefly or not at all. The section on freeze concentration deals with classical scraped-surface chillers, without any indication of the recent process based upon crystal ripeners and pulsed wash columns. The frontispiece shows schematics of alternate freeze-drying processes, including ones that are continuous. However, the reader is given no textual information which might be useful in understanding how these processes work or in deciding which might be more useful in different situations. The section on decaffeination is much shorter than is warranted by the very considerable amount of current attention to that area. It covers the classical methods of solvent and water extraction of caffeine from green coffee briefly and lists a number of patents from the period 1972-75, but does not delve at all into the currently active question of decaffeination by extraction with liquid of supercritical carbon dioxide.

The book is generally attractive in appearance, and is, with a few exceptions, well organized and indexed. The principal difficulty in production seems to have been rather poor reproduction quality of the many photographs which are included.

Despite the misgivings expressed in the previous paragraphs, this is still the reference to which I would first turn in seeking general information about any aspect of coffee processing.

C. JUDSON KING

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JOURNALS AND BOOKS
IN
FOOD SCIENCE AND NUTRITION

JOURNAL OF FOOD SERVICE SYSTEMS — G. E. Livingston and
   C. M. Chang

JOURNAL OF FOOD BIOCHEMISTRY — Herbert O. Hultin
   Norman F. Haard and John R. Whitaker

JOURNAL OF FOOD PROCESS ENGINEERING —
   Dennis R. Heldman

JOURNAL OF FOOD PROCESSING AND PRESERVATION —
   Theodore P. Labuza

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   Alina S. Szczesniak

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   E. Donald Murray and David H. Lees

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   N. W. Lord and P. E. Cheremisinoff

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   Herbert O. Hultin and Max Milner

PRINCIPLES OF FOOD SCIENCE — Georg Borgstrom
   VOLUME 1 — FOOD TECHNOLOGY
   VOLUME 2 — FOOD MICROBIOLOGY AND BIOCHEMISTRY

THE SCIENCE OF MEAT AND MEAT PRODUCTS,
SECOND EDITION — James F. Price and B. S. Schweigert

GUIDE FOR AUTHORS

Typewritten manuscripts in triplicate should be submitted to the editorial office. The typing should be double-spaced throughout with one-inch margins on all sides.

Page one should contain: the title, which should be concise and informative; the complete name(s) of the author(s); affiliation of the author(s); a running title of 40 characters or less; and the name and mail address to whom correspondence should be sent.

Page two should contain an abstract of not more than 150 words. This abstract should be intelligible by itself.

The main text should begin on page three and will ordinarily have the following arrangement:

Introduction: This should be brief and state the reason for the work in relation to the field. It should indicate what new contribution is made by the work described.

Materials and Methods: Enough information should be provided to allow other investigators to repeat the work. Avoid repeating the details of procedures which have already been published elsewhere.

Results: The results should be presented as concisely as possible. Do not use tables *and* figures for presentation of the same data.

Discussion: The discussion section should be used for the interpretation of results. The results should not be repeated.

In some cases it might be desirable to combine results and discussion sections.

References: References should be given in the text by the surname of the authors and the year. *Et al.* should be used in the text when there are more than two authors. All authors should be given in the References section. In the Reference section the references should be listed alphabetically. See below for style to be used.

DEWALD, B., DULANEY, J. T. and TOUSTER, O. 1974. Solubilization and poly-acrylamide gel electrophoresis of membrane enzymes with detergents. In *Methods in Enzymology*, Vol. xxxii, (S. Fleischer and L. Packer, eds.) pp. 82–91, Academic Press, New York.

HASSON, E. P. and LATIES, G. G. 1976. Separation and characterization of potato lipid acylhydrolases. Plant Physiol. *57*, 142–147.

ZABORSKY, O. 1973. *Immobilized Enzymes*, pp. 28–46, CRC Press, Cleveland, Ohio.

Journal abbreviations should follow those used in *Chemical Abstracts*. Responsibility for the accuracy of citations rests entirely with the author(s). References to papers in press should indicate the name of the journal and should only be used for papers that have been accepted for publication. Submitted papers should be referred to by such terms as "unpublished observations" or "private communication." However, these last should be used only when absolutely necessary.

Tables should be numbered consecutively with Arabic numerals. The title of the table should appear as below:

Table 1. Activity of potato acyl-hydrolases on neutral lipids, galactolipids, and phospholipids

Description of experimental work or explanation of symbols should go below the table proper.

Figures should be listed in order in the text using Arabic numbers. Figure legends should be typed on a separate page. Figures and tables should be intelligible without reference to the text. Authors should indicate where the tables and figures should be placed in the text. Photographs must be supplied as glossy black and white prints. Line diagrams should be drawn with black waterproof ink on white paper or board. The lettering should be of such a size that it is easily legible after reduction. Each diagram and photograph should be clearly labeled on the reverse side with the name(s) of author(s), and title of paper. When not obvious, each photograph and diagram should be labeled on the back to show the top of the photograph or diagram.

Acknowledgments: Acknowledgments should be listed on a separate page.

Short notes will be published where the information is deemed sufficiently important to warrant rapid publication. The format for short papers may be similar to that for regular papers but more concisely written. Short notes may be of a less general nature and written principally for specialists in the particular area with which the manuscript is dealing. Manuscripts which do not meet the requirement of importance and necessity for rapid publication will, after notification of the author(s), be treated as regular papers. Regular papers may be very short.

Standard nomenclature as used in the engineering literature should be followed. Avoid laboratory jargon. If abbreviations or trade names are used, define the material or compound the first time that it is mentioned.

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