Quality in Frozen Food

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Shelf Life Testing: Procedures and Prediction Methods for Frozen Foods

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19.1 Introduction

The shelf life of a food can be defined as the time period within which the food is safe to consume and/or has an acceptable quality to consumers. Just like any other food, frozen foods deteriorate during storage by different modes or mechanisms, as summarized in Table 1. Microbes usually are not a problem since they cannot grow at freezing temperatures unless subjected to extensive temperature abuse above the freezing point. Enzymes are a big concern for frozen foods, which can cause flavor change (lipoxygenase) in non-blanched fruits and vegetables and accelerated deterioration reactions in meat and poultry (enzymes released from disrupted membranes during precooking). Cell damage or protein and starch interactions during freezing cause drip and mushiness upon thawing. Discoloration could occur by non-enzymatic browning, bleaching, and freezer burn. Vitamin C loss is often a major concern for frozen vegetables. Physical changes, such as package ice formation, moisture loss, emulsion destabilization, recrystallization of sugars and ice of frozen desserts are often accelerated by fluctuating temperatures.

For any specific frozen product, which mode determines its shelf life, depends on the product characteristics (raw materials, ingredients, formulation), pre-freezing treatment, freezing process, packaging film and processes, and of course storage conditions. All of the quality deterioration and potential hazards are usually exaggerated or complicated by a fluctuating time-temperature environment (e.g. freeze/thaw cycle) during storage. On the other hand, the shelf life of a frozen food can be extended through ingredient selection, process modification and change of package or storage conditions, as discussed in Section 3 of this book.

This chapter will focus on shelf life testing of frozen foods for product development and market practices. Shelf life testing consists basically of selecting the quality characteristics which deteriorate most rapidly in time and the mathematical modeling of the change. Table 19.1 can be used as a reference for the selection of quality characteristics, which depends on the specific product and usually requires professional judgment. Mathematical modeling of quality deterioration will be discussed next.

Table 19.1 Deterioration modes of frozen foods

Frozen Foods	Deterioration Modes
Frozen meats, poultry and seafood	Rancidity
	Toughening (protein denaturation)
	Discoloration
	Desiccation (freezer burn)
Frozen fruits and vegetables	Loss of nutrients (vitamins)
	Loss of texture (temperature abuse)
	Loss of flavor (lipoxygenase, peroxidase)
	Loss of tissue moisture (forming package ice)
	Discoloration
Frozen concentrated juices	Loss of nutrients (vitamins)
	Loss of flavor
	Loss of cloudiness
	Discoloration
	Yeast growth (upon temperature abuse)
Frozen dairy products	Iciness (recrystallization of ice crystals)
(ice cream, yogurt, etc.)	Sandiness (lactose crystallization)
	Loss of flavor
	Disruption of emulsion system
Frozen convenience foods	Rancidity in meat portions
	Weeping and curdling of sauces
	Loss of flavor
	Discoloration
	Package ice
Frozen bakery products (raw dough,	Burst can (upon temperature abuse) (dough)
bread, croissants)	Loss of fermentation capability (dough)
	Staling (becoming leathery)
	Loss of fresh aroma

19.2 Modeling of quality deterioration

19.2.1 Basic equation

A frozen food starts to degrade once it is produced (Figure 19.1). The rate and the degree of degradation depends on both the composition and the environmental conditions during storage and distribution. In general, the loss of food quality or shelf life is evaluated by measuring a characteristic quality index, "A". The change of quality index A with time (dA/dt) can usually be represented by the following kinetic equation:

$$- dA/dt = k A^n \qquad (19.1)$$

where k is called a rate constant depending on temperature, product and packaging characteristics; n is a power factor called reaction order which defines whether the rate

of change is dependent on the amount of A present. If environmental factors are held constant, n also determines the shape of deterioration curve.

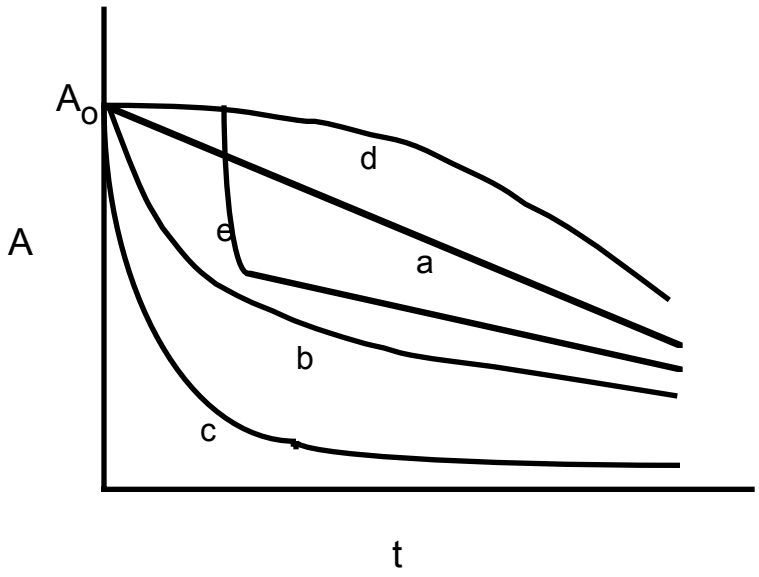

Figure 19.1 Quality deterioration curves: a) linear; b) exponential; c) hyperbolic; d) quadratic; e) complex.

19.2.2 Zero and first order kinetics

Equation 19.1 can also be written as:

$$f(A) = k\,t \qquad (19.2)$$

where f(A) is the quality function, k and t are the same as above. The form of f(A) depends on the value of n. When n is equal to zero it is called zero order reaction kinetics, which implies that the rate of loss of quality is constant under constant environmental conditions (curve (a) in Fig. 19.1). If n is equal to one it is called first order reaction kinetics, which results in an exponential decrease in rate of loss as quality decreases (curve (b) in Fig. 19.1, which becomes a straight line if plotted on a semi-log plot). These quality functions can be expressed as follows:

$$f(A) = A_o - A = k_z t \qquad \text{zero order} \qquad (19.3a)$$

$$f(A) = \ln A_o - \ln A = k_f t \qquad \text{first order} \qquad (19.3b)$$

where A_o is the initial quality value. If A_e corresponds to the quality value at the end of shelf life, the shelf life (θ) of the food is inversely proportional to the rate constant:

$$\theta = (A_o - A_e) / k_z \qquad \text{zero order} \qquad (19.4a)$$

$$\theta = \ln (A_o/A_e) / k_f \qquad \text{first order} \qquad (19.4b)$$

It should be noted that most chemical reactions leading to quality loss in frozen food systems are much more complex. However, the reaction kinetics can be simplified into either pseudo-zero order or pseudo-first order kinetics. In the case of complex reaction kinetics with respect to reactants, an intermediate or a final product (e.g. peroxides or hexanal in lipid oxidation) could be used as a quality index. There are few cases where neither zero nor first order kinetics apply. Curve (c) in Fig. 19.1 shows the degradation curve for a 2nd order reaction (with single reactant), which also shows a straight on a semi-log paper. A fractional order should be used to describe the curve (d) in Fig. 19.1.

Sometimes, there is an induction period or lag time before the quality deterioration begins (e.g. browning pigment formation in the Maillard reaction or a microbial growth lag phase, as shown in curve (e) in Fig. 19.1. The length of the lag depends on many factors, but temperature is a predominant factor. Given this, modeling of both the induction or lag period and deterioration phase are necessary for accurate prediction of quality loss or shelf life remaining. An example of such work has been demonstrated by Fu et al. (1991) for the growth of bacteria in milk.

In certain circumstances (e.g. A represents a sensory hedonic score), a non-kinetic approach, e.g. a statistical data fitting technique can also be used to describe the deterioration curves. Varsanyi and Somogyi (1983) found that the change in quality characteristics as a function of time could be approximately described with linear, quadratic and hyperbolic functions and that storage temperature and packing conditions affected the shape of the deterioration curves. However, the parameters determined by data fitting are difficult to use for prediction under variable storage conditions except for the linear curve.

19.2.3 Temperature dependence of deterioration rate

19.2.3.1 Arrhenius kinetics

Once a frozen product is made and packaged and starts its journey from the manufacturer's plant to warehouse, distribution center, retail store and finally

consumer's freezer, the rate of quality loss is primarily temperature dependent (Zaritzky, 1982). The Arrhenius relationship is often used to describe the temperature dependence of deterioration rate where for either zero or first order:

$$k = k_o \exp(-E_a/RT) \quad (19.5a)$$

or $$\ln k = \ln k_o - E_a/(RT) \quad (19.5b)$$

where k_o is a pre-exponential factor; E_a is an activation energy in cal/mol; R is the gas constant in cal/mol K and equal to 1.986; T is an absolute temperature in K (273 + °C). Thus, a plot of the rate constant on semi-log paper as a function of reciprocal absolute temperature (1/T) gives a straight line as shown as Fig. 19.2. The activation energy is determined from the slope of the line (divided by the gas constant R). A steeper slope means the reaction is more temperature sensitive, i.e., a small change in T produces are large change in rate.

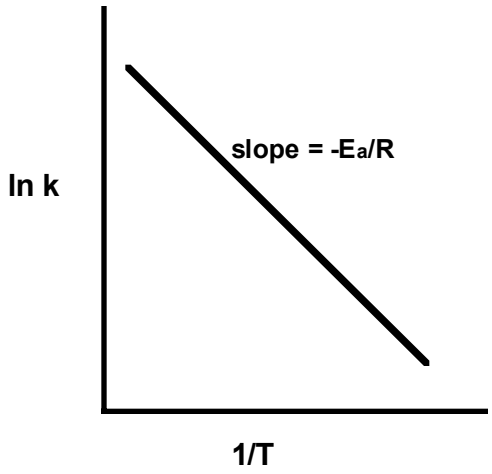

Figure 19.2 Arrhenius plot

Thus, by studying a deterioration process and measuring the rate of loss at two or three temperatures (higher than storage temperature), one could then extrapolate on an Arrhenius plot with a straight line to predict the deterioration rate at the desired storage temperature. This is the basis for accelerated shelf life testing (ASLT), which will be discussed later. One should note however that in some cases a straight line will not ensue for a variety of reasons, especially if a phase change occurs (Labuza

and Riboh, 1982). Thus for frozen foods, extrapolation from temperatures above 0∞C are meaningless for shelf life prediction.

19.2.3.2 WLF kinetics

Besides the Arrhenius equation, another popular equation at least in the more recent food literature, is the Williams Landau Ferry (WLF) model (Williams et al., 1955). Its original form was based on the variation of the viscosity in the temperature range above T_g as addressed in Chapter 3. When the rate constant at T_g' is substituted for T_g (T_g' is the T_g of a maximally freeze-concentrated system), the WLF model can be written as follows:

$$\log (k_T/k_g) = C_1(T-T_g')/[(C_2+(T-T_g')] \quad (19.6a)$$

$$\text{or} \quad [\log (k_T/k_g)]^{-1} = (C_2/C_1)/(T-T_g') + 1/C_1 \quad (19.6b)$$

where C_1 and C_2 are constants. Thus a plot of $[\log (k_T/k_g)]^{-1}$ vs. $(T-T_g)^{-1}$ will be a straight line with the slope equal to C_2/C_1 and the intercept equal to $1/C_1$. As can be seen this is a two parameter temperature dependent model as is the Arrhenius equation.

Frozen foods stored below T_g' are stable to ice recrystallization and other physical changes. Levine and Slade (1988) postulated that stability is related to the temperature difference between storage temperature and T_g'. This cryostabilization of foods assumes stability below T_g' and rapid decrease of stability above T_g' according to the WLF relationship, exhibiting an increase in reaction rate, much higher than expected from the Arrhenius kinetics. However, this may not be true since the rate of chemical reactions can be expected to be influenced by temperature increase in a complex way: (i) an increase of the rate constant, resulting from both the viscosity decrease and the increased molecular mobility (Fennema 1996); (ii) a decrease of the reaction rate as a consequence of the increasing dilution of the reactants Roos et al. (1996). For these reasons, it seems that the WLF model over predicts the temperature effect of rate constant (Simatos et al., 1989). As noted by Nelson and Labuza (1994), because of the small temperature range over which foods are stored, e.g., about Δ30°C for dry foods and Δ20°C for frozen foods, both the Arrhenius and the WLF model give good correlations as long as one does not use the universal coefficients suggested by Slade and Levine (1991). In fact as shown by Nelson and Labuza (1994), their use of the Lim and Reid (1991) data for enzymatic activity in the frozen state as shown in 19.3 is not proof that the Arrhenius relationship does not apply, WLF was assumed because the rate was negligible below -10°C which was the measured Tg. But as seen in

Figure 19.3b if the data is plotted as Arrhenius plot an r^2 of 0.999 ensues. The challenge in applying the WLF model for stability or shelf life prediction is that (1) T_g is not known; (2) T_g is difficult to determine; and (3) the universal coefficients of Levine and Slade (1986) are not applicable.

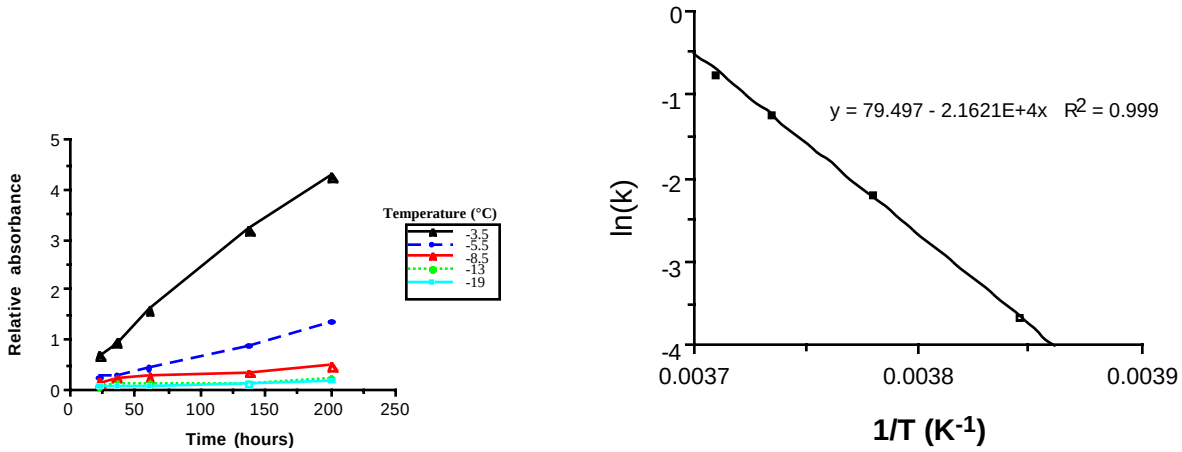

Figure 19.3 Hydrolysis of maltodextrin in the frozen state (Lim and Reid; 1991)

a. Rate as a function of temperature (Note T_g is -10 °C)

b. Arrhenius plot

19.2.3.4 Shelf life model

Most published data related to quality deterioration do not give rates or rate constants but rather are in the form of an overall shelf life (end-point analysis) as a function of storage temperature. Since the temperature range used is usually quite narrow, the following exponential relationship exists between shelf life and storage temperature:

$$\theta = \exp(-bT+c) \qquad (19.7a)$$

or $$\ln \theta = -bT+c \qquad (19.7b)$$

where θ is shelf life at temperature T in °C, b is the slope of the semilog plot of θ vs T and c is the intercept or reference temperature as shown as Fig. 19.4. Practically, this is used frequently for shelf life determination and prediction due to its simplicity and straightforwardness.

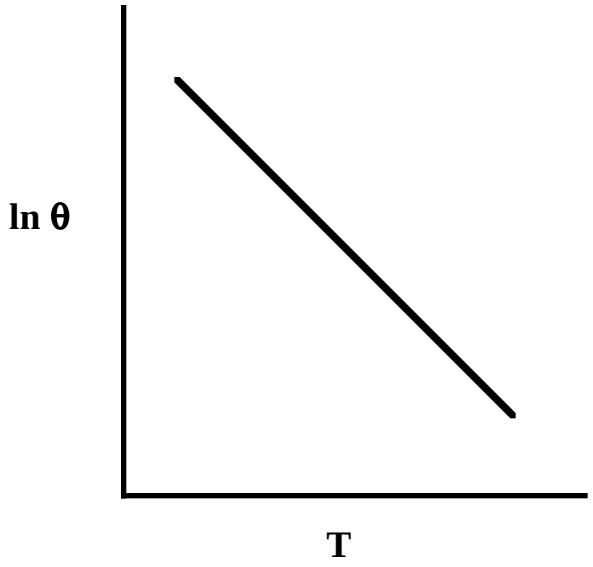

Figure 19.4 Shelf life plot

19.2.3.4 Q_{10} or q_{10}

The Q_{10} approach is also often used for estimation of the temperature acceleration of shelf life, which is defined as :

$$Q_{10} = \text{rate @ } T_1+10\ °C \text{ / rate @ } T_1 \quad (19.8a)$$

$$Q_{10} = \text{shelf life @}T_1 \text{ / shelf life @}T_1+10\ °C \quad (19.8b)$$

$$Q_{10} = (q_{10})^{1.8} \quad (19.8c)$$

where T_1 is temperature in °C. If the temperature unit is in °F, then the term q_{10} is used, which in fact is more often used than Q_{10} in the frozen food literature.

The magnitude of Q_{10} depends on the food system, the temperature and the absolute range. Q_{10} values from 2 up to 20 have been found for frozen foods (Labuza, 1982) Labuza and Schmidl, 1985. Q_{10} can be shown to be related to the Arrhenius equation and the shelf life model through the following expression:

$$Q_{10} = \exp [10\ E_a/(R\ T\ (T+10)] \quad (19.9a)$$

$$Q_{10} = \exp (10\ b) \quad (19.9b)$$

Thus Q_{10} is not constant but depends on E_a and the absolute temperature T.
Some data gleaned from July (1989) and Labuza (1982) is shown in Table 19.2.

Table 19.2
Estimate of the Q_{10} for shelf life of selected frozen foods

Item	Days of HQL -10°C	-20°C	Q_{10}
pork sausage	20	120	4
pork	50	400	8
beef	60	200	3.3
ground hamburger	250	800	3.2
fried hamburger	35	250	7
raw poultry	200	700	3.5
fried poultry	25	700	3.2
fatty fish	7	60	9

19.2.3.5 Other models

The following models have also been proposed to describe the temperature dependence of the rate constant (Kwolek and Bookwalter, 1971) for frozen systems:

$$k_T = a + b\,T \qquad (19.10a)$$

$$k_T = a\,T^b \qquad (19.10b)$$

$$k_T = a / (b - T) \qquad (19.10c)$$

where a, and b are constants. In most cases, Equation 19.10c fits data better. However, all these have very limited practical application.

19.2.4 Time-temperature tolerance

Frozen foods are often exposed to a variable temperature environment, e.g. during distribution or due to freezing/defrosting cycle in retail or home freezers. In general, the value of the quality function, f(A), at time t under changing environmental conditions can be estimated from:

$$f(A) = \int k_{[T(t)]}\,dt \qquad (19.11)$$

where T(t) is the temperature as a function of time. The form of f(A) depends on the reaction order as discussed previously. If an effective temperature, T_{eff}, is defined as

that constant temperature exposure which causes the same quality change as the variable temperature condition, as proposed by Schwimmer et al. (1955), then

$$f(A) = k_{eff}\, t \qquad (19.12)$$

The rate constant at that defined temperature is termed the effective rate constant, i.e. k_{eff}. To estimate the quality change under variable temperature conditions, one needs to either solve for f(A) numerically or know the value of T_{eff} or k_{eff} that corresponds to the variable conditions.

The numerical approach for a randomly variable temperature history is essentially the same as the Time/Temperature/Tolerance (TTT) approach initiated by Van Arsdel et al. (1969) and derived empirically in the 1960's for the prediction of shelf life of frozen foods (July, 1984). It is assumed that the temperature history of the product is known. Thus the fraction of shelf life consumed, f_{con}, was calculated as the sum of the times at each temperature interval, t_i, divided by the shelf life at that temperature, θ_i:

$$f_{con} = \Sigma\, (t_i / \theta_i) \qquad (19.13)$$

Thus the remaining shelf life at a reference temperature is equivalent to $(1-f_{con})*\theta$.

Equation 19.13 assumes that the rule of additivity is valid for frozen foods (July, 1984), which means that the loss of remaining storage life or quality can be calculated from knowledge of the prior time-temperature episodes the product has been exposed to. This also implies that the prior sequence of the time-temperature episodes is of no importance except to calculate the amount of quality remaining up to that time, i.e. there is no history effect. If the rule of additivity is valid with reasonable accuracy, the use of time-temperature integrators (TTI) should provide reliable results with respect to prediction of shelf life remaining, which will be discussed later.

However, there are some cases where the total effect of various temperature experiences may not be independent of the order in which they occur or of the nature of temperature history. For example, widely fluctuating temperatures may cause freezer burn or in-package desiccation, which is not additive (July, 1984). Where the colloidal nature of a product is affected, the effect of time-temperature history may not be additive either, especially with a freeze/thaw cycles. This is also true when growth of microorganisms occurs (Fu et al., 1991). Certain chemical reactions, enzymatic as well as nonenzymatic, could even proceed more rapidly at temperatures below

freezing. This is called a negative effect of temperature (Singh and Wang, 1977), which could be caused by one or more of the following factors: (1) a freeze concentration effect; (2) the catalytic effect of ice crystals; (3) a greater mobility of protons in ice than in water; (4) a change in pH, up or down with freezing; (5) a favorable orientation of reactants in the partially frozen state; (6) a salting in or out of proteins; (7) decrease in dielectric constant; and (8) the development of antioxidants at higher temperatures. As has been shown by Fennema (1975), the freeze concentration effect can cause rates of chemical reactions to increase dramatically just below the freezing point (Figure 19.5), e.g. ascorbic acid loss at -3°C can be faster than at higher temperatures this one should not use data in the -4°C to 0°C range or above as part of an accelerated shelf life test to predict rates at lower temperatures. Fennema (1975), showed that the time to 50% loss of vitamin C in broccoli was 44 days at -5°C, 120 days at -2°C and 162 days at +2°C. This concentration effect is evident in the shelf life plot of frozen strawberries as shown in Fig. 19.6 using the data of Guadagni (1968). If the data collected only at 25 and 30°F (-3.9°C and -1.1°C) are used, the predicted shelf life at 0°F (-17.8°C) is over 27 years, if data are collected at only 20 and 25°F (-6.7 and 3.9°C), the shelf life predicted at 0°F is 40 days while data below 20∞F extrapolated to the true expected shelf life is about 280 days.

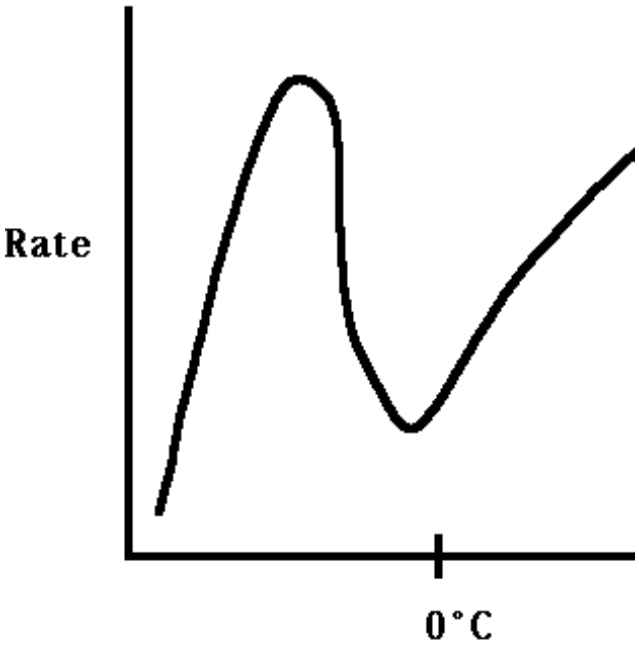

Figure 19.5 Rate of chemical reaction as a function of temperature above and below the freezing point of a food.

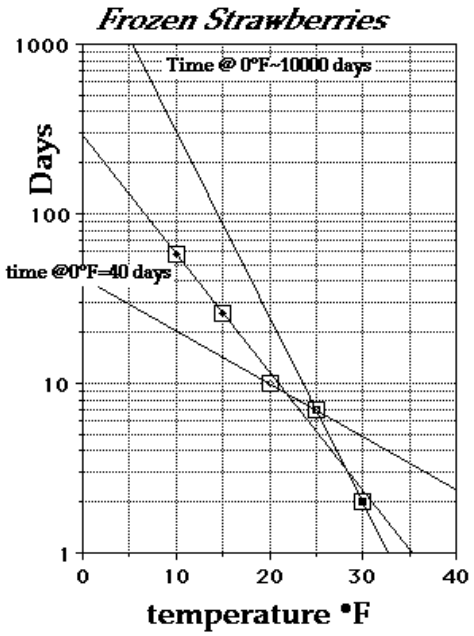

Figure 19.6. Shelf life plot of frozen strawberries showing the influence of the freeze concentration effect just below the freezing point on prediction of shelf life at 0°F . Data from Guadagni (1968). Each line represents a regression through a different selected set of temperatures.

The response ratio of the food to changes in environmental temperature (R_T) is dependent on the fluctuating temperature conditions as well as the heat transfer properties of the food as well as the package (Cairnes and Gordon, 1976; Dagerskog, 1974). In the analysis of food shelf life, an inherent assumption is made that the food is responding instantaneously to the environmental temperature changes, i.e., $R_T = 1$. This may be acceptable if a surface deterioration process is the deterministic factor for shelf life, e.g. mold growth in some foods. Freeze-defrost cycles generally can be considered as sinusoidal oscillations. The amplitude of the effect is reduced inside the package by some factor thus $R_T. < 1$. It can be expected that the shorter the period of the ambient variation the smaller the R_T, and hence the smaller the amplitude of the cyclic temperature variation in the package. Zuritz and Sastry (1986) also studied the effect of packaging materials on temperature fluctuations for frozen ice cream and found that packaging materials coupled with a layer of stagnant air were effective barriers against thermal fluctuations.

19.2.5 Hazard function

After the product is produced, it may fail at any point in time in accordance with its life distribution (Nelson, 1972). The hazard function h(t) of a distribution is defined for $t \geq 0$ by:

$$h(t) = f(t)/[1-F(t)] \qquad (19.14)$$

where f(t) is a probability density function and F(t) is a cumulative distribution function. The h(t) is the conditional probability of failure at time t, given that failure has not occurred before ..

The behavior of a hazard function for studying the shelf life of food products can be easily understood by examining the "bathtub" shaped curve in Fig. 19.7. Note that at time t_o, a frozen food product begins its journey to many distribution outlets for consumption. During the time between t_o and t_1, early failures may occur owing to a failure in the process itself, faulty packaging, extreme initial product abuse, and many other environmental stresses to which the product is subjected. Early failure should not be taken as a true failure relative to the shelf life of the product unless it represents the normal condition. From t_1 to t_2 one can expect, barring chance major temperature fluctuations, no failures. This interval represents the true period of the product's stability. The failure rate is almost constant and small during this time. The hazard or failure rate increases from time t_2 to the termination point t_3, owing to the true deteriorative changes occurring within the product. The concept of hazard function is important in the analysis and interpretation of the failure times of a product.

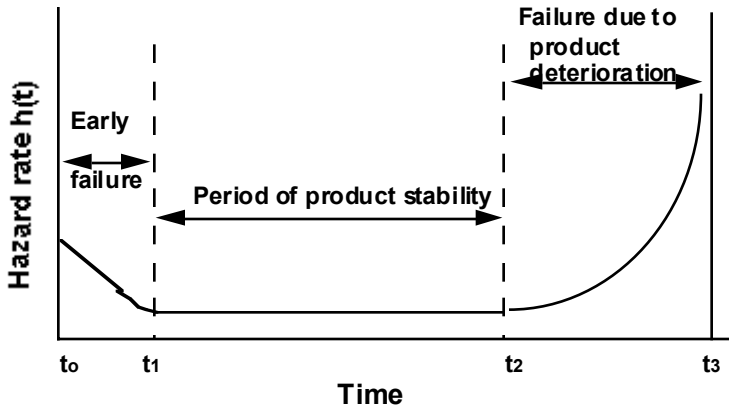

Figure 19.7 Failure rate as a function of time

A fundamental assumption underlying statistical analysis of shelf life testing is that the shelf life distribution of a food product belongs to a family of probability distributions and that observations are statistically independent. Parameters of a shelf life distribution are estimated by use of shelf life testing experimental data. Once the parameters of a shelf life model have been estimated, it can be used to predict the probabilities of various events, such as future failures (Nelson, 1972). Five statistical models, normal, log normal, exponential, Weibull and extreme-value distributions were tested for a few food products (Gacula and Kubala, 1975; Labuza and Schmidl, 1988) and it was found that the Weibull distribution fits best, which will be demonstrated later.

19.3 Shelf life testing — overall aspects

19.3.1 Purpose

In the development of any new food product including reformulating, change of packaging or storage/distribution condition (to penetrate into a new market), one important aspect is the knowledge of shelf life. The shelf life of a food product is vital to its success in the marketplace. This life must at least exceed the minimum distribution time required from the processor to the consumer. Shelf life testing can assess problems that the product has in the development stage, following a "fail small fail early" philosophy, thereby eliminating large disasters later. Marketing/brand managers also need reliable shelf life data to position the products and to establish the brand. Periodic determination of shelf life help to provide assurance that the product remains consistent over time with respect to quality.

Different shelf life testing strategies are necessary at different stages, as illustrated in Fig. 19.8. If the objective is to identify whether pathogens and spoilage microbes will grow in the case of temperature abuse, then a challenge study is necessary. If the objective is to quickly estimate the approximate shelf life of the product then an ASLT can be used, as long as the proper temperature range is chosen. A confirmatory shelf life test may be conducted at the last stage with simulated distribution chain conditions, although in today’s R & D environment, this may be skipped.

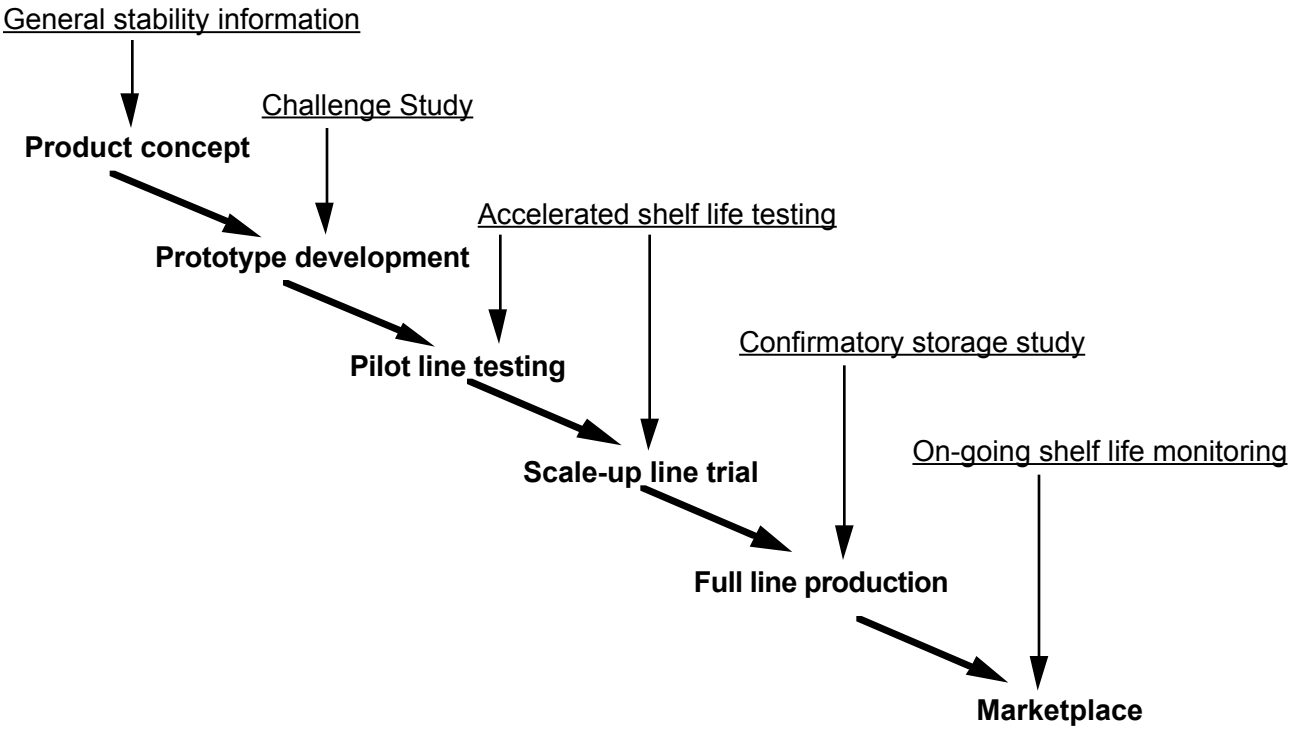

Figure 19.8 Shelf life testing strategy at different product development stages

19.3.2 Shelf life criteria

The criterion for the end of shelf life may be variable depending on the definition of product quality grade, so the shelf life of a product may also be variable. The shelf life of most perishable and semiperishable foods is almost solely based on sensory quality. For example, fresh meat degrades mainly by bacterial activity and rapid chemical oxidations that cause an off-flavor development and loss of color. This is readily recognizable by consumers. In contrast, many longer shelf-life foods including most frozen foods degrade mainly by slow chemical reactions such as loss of nutritional value. For example, the vitamin C content of some frozen fruits and vegetables, may fall below the required standard as listed on the label before sensory quality becomes inadequate.

The criteria for shelf life may also vary depending on the sensitivity of the consumer. For consumers, taste, odor, and appearance are the most obvious criteria; in academia and in the industry, sensory evaluation correlated with instrumental measurements of a given quality index (e.g., vitamin C level) are usually conducted. In general, the criteria level corresponding to the end of shelf life of a product depends

on: (i) any legal requirement, e.g. zero tolerance for botulinum toxin; (ii) consumer preferences or marketing requirements; and (iii) cost. In essence, the end of shelf life depends on the percentage of consumers a company is willing to displease. If 100% acceptance is required then high cost ingredients and absolute control of distribution up to point of consumption is necessary, otherwise there will always be some people who will get foods beyond shelf life. The aim is to keep this as small as possible.

19.3.2.1 Just noticeable difference (JND)

Sensory (organoleptic) examination of foods was a general procedure used by the human race to evaluate wholesomeness of foods long before the discovery of microorganisms. Sensory evaluation of foods by scientific methods can be used to evaluate such attributes as taste, odor, body, texture, color and appearance. Changes in these attributes may be brought out by microbial or non-microbial actions, usually the latter for frozen foods.

The methods used to evaluate sensory shelf life data include difference testing and hedonic scoring. Difference testing can involve paired comparisons, duo-trio tests, or triangle tests. The paired comparison procedure determines the time when a measurable difference in quality occurs between two test samples at a certain level of probability. When applied to frozen foods, this method is often referred to as the Just Noticeable Difference (JND) test or High Quality Life (HQL) test (July, 1984), which is usually based on flavor changes. Duo-trio testing compares two unknowns to an unabused control sample and asks the question of whether either of the unknowns are the same as or different from the identified control. Triangle testing determines the one different product among three test samples presented randomly to a set of judges (at least 10). Probability plots are used to predict shelf life at a given probability level. The difference method can result in finding a difference when none really exists (Type I error), or not finding one when indeed there is a true difference (Type II error). Labuza and Schmidl (1988) have discussed this topic more thoroughly in relationship to shelf life testing, which is not commonly found in sensory textbooks. Table 19.3 shows some data from Guadagni (1968) for HQL of frozen foods.

Table 19.3

Days of High Quality Life for fruits and vegetable (from Guadagni 1968)

Product	Type	0°F	10°F	20°F
apples	pie filling	360	250	60
blueberries	pie filling	175	77	18
cherries	pie filling	490	260	60
peaches	retail syrup	360	45	6
blackberries	bulk, no sugar	630	280	50
raspberries	bulk, no sugar	720	315	70
	retail, syrup	720	110	18
strawberries	bulk, sugar	630	90	18
	retail	360	60	10
green beans	retail	296	94	30
cauliflower	retail	291	61	13
peas	retail	305	90	27
spinach	retail	187	57	23
corn	retail	720	360	
corn on cob	retail	275	150	

19.3.2.2 Hedonic scoring

Hedonic scoring — which indicates acceptance on a numerical scale, e.g. a 1-9 point scale labeled from "dislike extremely" to "like extremely", is typically used for shelf-life evaluation. The test can be designed to not only evaluate the overall acceptance of the product, but that of specific characteristics such as flavor, texture, appearance, aftertaste, etc. Trained panels can also use this technique on a line scale, which can be converted to numerical equivalents.

If the hedonic method is used to evaluate shelf life, one can simply use the score as quality index A and plot the score vs. storage time, run a linear regression, and choose the end of shelf life as the time when the progressed value drops below a pre-set level (Waltzeko and Labuza, 1976; Gacula, 1975). The shelf life determined in this way is called the practical shelf life (PSL) for frozen foods (July, 1984), and is longer than the HQL or JND. The use of hedonic rating scales may be of limited use in shelf life testing, yet it is probably the most used method. Many food companies use a loss in hedonic score equal to Δ=0.5 for HQL and Δ=1.5 for PSL as the end of shelf life

(Labuza, 1982). Objective measurements and professional judgment are often required to determine the end point. Data in Table 19.4 from an report published by the former Refrigerated and Frozen Foods Institute (1973) Unfortunately there were no methods given, but the data suggests that the PSL is about 2 to 3 times longer than the HQL value. This in itself suggests that the HQL methods can be used to shorten shelf life testing times.

Table 19.4
Relationship between practical shelf life (PSL)
and High Quality Life for frozen foods.

Frozen Food	PSL/HQL Ratio
lean meat	1.9 - 2
fatty meat	2.0-2.4
lean fish	1.9-2.2
fatty fish	2.4-2.7
precooked foods	2.8-3.0
fruit	2.8-3.1
vegetables	3.1-3.5

19.3.2.3 Instrumental analysis

Chemical or instrumental analysis, such as moisture, nutrient loss, free-fatty acids or color measurement that closely correlate to sensory attributes, can supplement sensory techniques. They are usually less expensive and less time-consuming than sensory approaches. A correlation between a physical or chemical test can increase the confidence level of the sensory results. For example, the following constituents or properties can be considered for monitoring chemical changes of pizza quality during frozen storage: total free fatty acids, specific volatile free fatty acids by HPLC, peroxides, oxidative volatiles (e.g., hexanal) by GC, spice volatiles by GC, lysine, color (decrease in red color or increase in brown), in addition to sensory evaluation of taste and flavor (Labuza, 1986). Most sensory experts agree that analytical methods should complement the sensory tests. Vice versa, the endpoint determined by objective measurements should be confirmed by sensory techniques as well.

19.3.2.4 Weibull Hazard analysis

The Weibull Hazard procedure requires one to first make an estimation of the time to the end of shelf life. This becomes the initial estimated time limit for the study. The time limit is then divided into several segments at which points panelists grade the product. Additional panelists are added at a constant number for each subsequent time period to maximize the number of testers near the end of the test. The panelist is asked to grade the food as good (acceptable) or bad (unacceptable), i.e. no ranking on a hedonic score. When the product is identified as unacceptable by 50% of the panelists, the number of testers for the next period is increased by the number of failed samples plus the constant number. The interval between sample times is also shortened as the end of shelf life gets closer. The test ends when no more samples or panelists are available. The scores are ranked and the cumulative hazard calculated. The critical probability of failure P_c, can then be calculated from the following equation:

$$P_c = 100\ (1 - \exp(-\Sigma(H/100))) \qquad (19.15)$$

where H is the hazard value equal to 100/Rank. Choosing P_c = 50%, corresponds to an accumulated hazard value of 69.3%.

The relationship between the logarithm of storage time (log t) and the logarithm of hazard value (log H) is linear:

$$\log t = (1/\beta) \log H + \log \alpha \qquad (19.16)$$

where β is the shape parameter and α is the scale parameter. The shelf life can then be determined based on the desired probability level allowed for product failure. The lower this probability, the shorter the shelf life. This plot then allows one to make a management decision with respect to the probability of displeasing a certain fraction of consumers. It is hoped that the distribution time is such that greater than 99 percent of the product is consumed before the end of shelf life based on displeasing less than X% of consumers where X is the economic value. An detailed example was given by Labuza and Schmidl (1988). It should be noted that this process can also be used for simple analytical tests such as plate counts or vitamin C. In these cases the number of panelists are replaced with the number of samples tested. Some criterion such as 20% vitamin C loss is used as the negative response. Figure 19.9 shows an example of Weibull plot for a frozen food based on assumed data. A shelf life of 16 months is

found at P_c = 50% from the graph. From this graph then, if 95% of the food were distributed and consumed in 3 weeks, only 1% of the consumers would be displeased

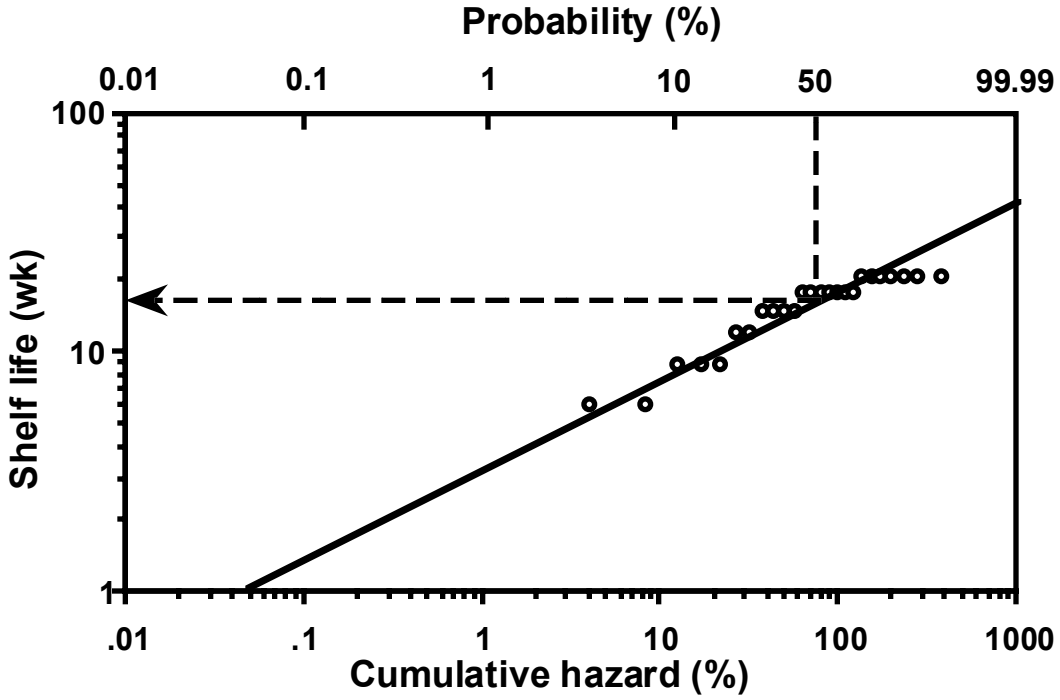

Figure 19.9 An example of Weibull plot for a frozen food. A shelf life of 16 wk was determined at Pc = 50%.

(or 0.95% of the product is out of compliance). If the rest were held and consumed at 10.5 weeks, 50% of those eating it would have out of quality food or another 0.5 x 5% = 2.5% of product. Thus in this distribution model about 3.5% of the product is unacceptable. To improve on this, the product must either move faster or one must distribute it at a lower temperature. Wittinger and Smith (1986) used this approach to determine sensory shelf life of ice cream based on iciness and found a shelf life of 5 weeks at 0°F (-15.5) which fits the general data for iciness in ice cream as shown in Figure 19.10 (Labuza, 1982). It should be noted that this gives a Q_{10} of about 12.

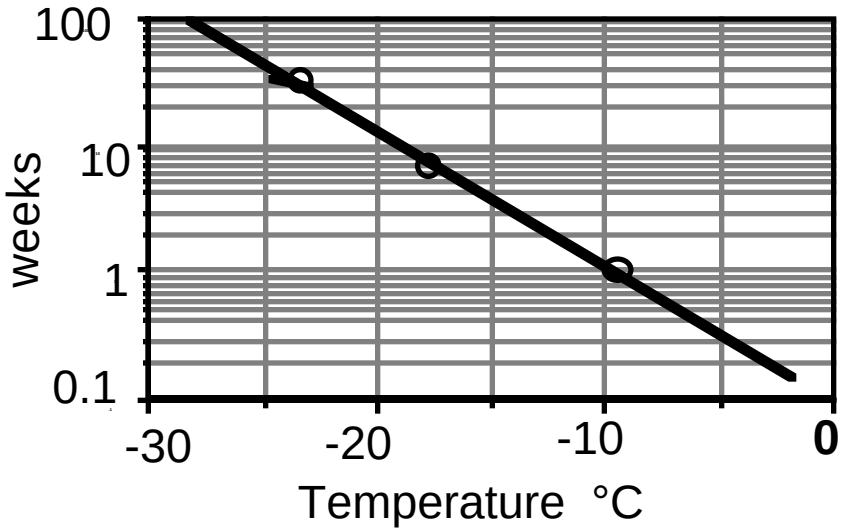

Figure 19.10 Shelf life plot for ice cream based on icyness perception from data of Labuza (1992)

19.3.3 General procedures

Shelf life testing experiments are designed to measure the average shelf-life of a product under given conditions. General procedures for shelf life testing of foods were proposed by Labuza and Schmidl (1985), which include:

Step 1: Develop testing protocol — The protocol should consist of: i) specific objective; ii) detailed test design in terms of product, package, and storage condition; iii) execution procedures in terms of time, space and resource availability; iv) cost estimation.

Step 2: Identify key quality indicator — Any previous shelf life data and kinetic parameters of food deterioration available in the literature (Labuza, 1982; Man and Jones, 1994) or the distribution turnover time of a similar or a competitive product in the market place, if any, would be very helpful in this preliminary identification or in determining the shelf life requirement.

Step 3: Estimate product sample and control needs — The number of samples and controls required should be based on the detailed experimental design. If sufficient product is available, extra samples should be placed into each storage

condition. Now and then it may be necessary to recheck a sample, especially if a value is not in line with other data. It would be disastrous to be out of sample before failure has occurred or the predetermined termination of the test is reached. Extra controls should also be prepared and stored. When the samples are placed into storage rooms, they should be positioned so that the complete package is exposed to the external atmosphere, unless otherwise specified. The specific location of the test sample should be recorded. Temperature controllers should be checked for accuracy, periodically. In addition, removal of all unused samples from the storage room to make space for future studies is a must.

There are various thoughts when it comes to using a control product. Some sensory experts prefer an actual physical control; others are satisfied to just use the numbers obtained in the zero time evaluation. There are three alternatives when using a physical example as a control: (i) making the control from scratch each time using the same ingredients, procedures, etc.; (ii) deep-freezing the control (e.g. pizza held at -70 °C) and accepting that it might have changed slightly, but minimally compared to the product in shelf life; (iii) using a fresh batch of product which may not be identical.

Step 4: Select proper package materials and package size — This is largely dependent on shelf life requirements, packaging costs and availability, and consumer information. Factors such as vacuum packaging, nitrogen flushing, or use of antioxidants are often considered in combination with packaging materials.

Step 5: Choose storage conditions — Storage conditions are chosen based on the type of shelf life testing. For example, the intended commercial storage/distribution temperature range should be used in confirmatory shelf life testing. Elevated temperatures are often used in accelerated shelf life testing to obtain data for prediction of shelf life at lower temperature or for prediction of shelf life under variable time-temperature distributions. Humidity control and/or monitoring is less important for frozen foods as compared to other foods (e.g., snacks, cakes, pies, and pastries). Light in the room should be properly controlled depending on the package.

Step 6: Estimate sampling frequency and duration of testing — The sampling frequency is generally an estimation based upon experience from prior studies with similar foods. However, once one knows an interval at one temperature, then the intervals at other temperatures can be estimated using a Q_{10} value i.e., if the Q_{10} is 3 then for a 10°C lower temperature the sampling times can be 3 times longer. If the interval between sampling is too long, the risk of under- or over-estimating shelf life increases. The more analyses that are completed, the more accurate will be the shelf life determination.

The question as to when one should end the experiment must be based on some pre-set criteria for failure. One criterion could be the minimum shelf life requirement driven by product category, distribution chain, and the benchmark's product stability. If there is an accompanying sensory test, the end time can be based on some organoleptic inferior quality criteria from which one then can get a microbial or chemical index limit. For frozen products, several weeks to months are usually needed. If the shelf life can be estimated with any accuracy, the test intervals can be lengthened and clustered around the expected failure period. Most of the experts only require about six evaluations to provide reliable results.

Step 7: Schedule for execution — Before scheduling the starting date for a shelf life test, one must check for the availability of ingredients, packaging materials, and storage space, and the time and resource available in the pilot plant or in the processing plant to prepare the samples. One should also check for the time and resources available in the microbial lab, the analytical lab and/or the sensory support staff throughout the test period. A copy of the test request and schedule should be sent in advance to those who will be doing the work. The courtesy of providing those involved with this advance information always pays dividends. Holidays should be marked on the scheduling calendar, since scheduling too many evaluations near major holidays or Friday afternoon is not recommended. However, once scheduled, sample observations on weekends and holidays should not be skipped over, since important data points could be missed.

Step 8: Take sample and evaluate quality — Samples should be taken and evaluated following pre-determined schedules. Sampling plans should be administratively and economically feasible, taking into account the heterogeneity of the food. Maxcy and Wallen (1983) pointed out the problem of heterogeneity of samples in shelf life prediction. Multiple subsamples (≥ 3) should be done for non-homogenous samples. A single package is usually used as an experimental unit. Replication of 3 or 4 units are desired for each measurement. For frozen foods, a thawing process is often involved in the sampling procedure. Proper thawing or microwave heating is critical to the product quality. All samples should be thawed or microwaved in the same way to minimize any biases.

The intended analyses should be based on the specific mode of deterioration, which was discussed earlier. Whatever the choice, the tests should be reasonable and logical. The key is to make sure that one is measuring the right thing. If the wrong quality factor is measured, the test starts out a failure. Unfortunately, in many cases this cannot be established initially, so sensory evaluation is a must in almost all shelf life

tests. Key sensory evaluation techniques for frozen foods have been discussed before.

At the time of each pull, one unit of the sample should be evaluated (informally by a minimum of 2-3 people) for changes in flavor and texture. This should be done in addition to the final tasting prior to a consumer sensory test. This is necessary since it helps the developer know approximately how the product is doing during the progress of the shelf-life, helping to avoid any surprises in the results. Control samples may need to be prepared fresh.

Step 9: Analyze data — Shelf life is the predicted day at which the stored product (test pull) is X% less than the control at day zero (Reference). The data should be plotted and regressed to determine that point using the proper model (zero or first). All too often the data are not analyzed until the experiment is over and then the scientist finds that nothing can be concluded because of lack of points or a poor fit or some surprises. Statistical curve fitting should be consistent with the chosen model based on a theoretical mechanism. The amount of change and number of data points are related to the coefficient of variation (CV) of the test. A weighting factor may be used in estimating the rate constant and its statistical limits. When the data for an attribute does not fit the regression model well (adjusted R^2 of < 0.8), scientific judgment should be used to decide whether the data are applicable.

When in doubt, a rerun on retention samples might help understand or clarify the results. Error analysis could be performed before experiments are run by first finding inherent errors in time, temperature, and quality index measurements, then calculating an expected standard deviation for the plot being used to determine a rate constant. If the experimental data have a standard deviation much higher than the expected value, either the functional form of the rate expression is incorrect or the data contain errors from unanticipated sources.

Step 10: Prepare shelf life report — Depending on the type of shelf life determination, the results should either throw light on the technical viability of the product or provide answers to the questions about the maximum safe shelf life as well as the maximum quality shelf life of the product. Before a shelf life is finally set, factors in the scale-up of shelf life data will need to be taken into consideration. Based on results from ASLT, the provisional shelf life will be set for the product. There is no government regulation which defines the product end point except for that related to nutrient levels (vitamin C and vitamin A) in 21 CFR 101.9(g)(1)(ii) which states that for the vitamins listed, the analysis level cannot be below 80% of the label value if it is a natural food with no added nutrients or cannot be below 100% (21 CFR 101.9(g)(1)(i))

if the product has any added vitamin or nutrient whether or not it is the nutrient under test. Thus one must base the label value on some predicted initial variability and some predicted loss during distribution and storage. The FDA usually takes samples at the supermarket level (where they can purchase them) for compliance testing, not from the end of the process line so distribution losses must be factored in.

The end point of shelf life is thus dependent on your corporate objectives and how much risk the company is willing to take with the brand. No shelf life test is completed until a termination summary has been written. All termination summaries should include the objective of the test, product description, package description, conditions and length of storage, methods of evaluation, results (in the form of graphs, shelf life plots and Q_{10} values) and conclusions. Termination summaries should become a permanent record in the company library for future reference and preferably indexed well on a computer data base for later retrieval when needed. The final shelf life should also be set to give a clear margin of safety. In any case, the shelf life of a new product, particularly of the high risk category, should be set based on data that relate to the worst case manufacturing and storage scenario. The shelf life can then be reviewed and if necessary re-set in the light of further experience in manufacturing and control after the product has been launched.

Step 11: Implementation — One should get top management's approval of the test results so that they can be implemented. Management must believe and support those test results. It is important for production, sales, distribution, purchasing and quality control to work together to be sure that the production is properly handled from the time of manufacture until this product is consumed.

19.4 Challenge study

19.4.1 Basis

Freezing reduces the microbial population of foods but considerable numbers usually survive even prolonged frozen storage. A challenge study is often used in the laboratory to study the factors and factor interactions as they affect the shelf life of the product. Such simulated experiments enable the researcher to better control the study. A challenge study is necessary for frozen foods for two reasons: (i) to predict microbial growth and potential risk of the product upon temperature abuse in a distribution chain; and (ii) to assess the relative stability and the relative risk of different formula, different processes or different packaging materials, which is a must in new product development. A challenge study may also be considered as a preliminary shelf life determination in terms of microbiological safety. It is often used in the early stage of

development since if microbial safety is a concern at this stage, then reformulating can be done quickly.

19.4.2 Microbial abuse procedures

Step 1: Identify barriers — A composition/ingredient analysis should be done to identify any barrier(s) against spoilage microbes and pathogens in case of temperature abuse.

Step 2: Choose types of organisms/strains and inoculation level — One principle is to use an organism or a strain that has been isolated previously from the product or similar foods which is responsible for spoilage or risk. The more isolates in the study, the greater is the confidence in the accuracy of the shelf life assessment. An inoculation level must also be determined, which is generally much higher than the normal contamination level in a product. If the average contamination level for a particular product is known, then the inoculation level should be as close to that level as possible. Sometimes several inoculation levels are used.

Step 3: Determine temperature abuse conditions — After inoculation, products should be packaged using the desired commercial packaging conditions, and subjected to temperature abuse. Factorial design and response surface methodology are often used in designing a challenge study. A typical temperature abuse condition used by some food companies is provided in Table 19.5. It starts out with five sets of test packages placed at -18 °C to begin the cycle. At the end of the first 24 hr, one set of packages is removed and tested for microbiological indicators to establish a zero-time level. All the other packages are kept at -18 °C for the next 20 hr, then removed and abused by placing them at 38 °C for 4 hr. Another set of packages is then removed for microbiological testing, and the cycle is repeated for the remaining packages, i.e. they are all returned to -18 °C for at least 20 hr, then abused at 38 °C for 4 hr. This procedure is repeated so that one set goes through at least four freeze-thaw cycles. If there is no significant increase in spoilage organisms or pathogenic organisms after the fourth cycle, the food is deemed safe microbiologically.

Table 19.5 A typical temperature abuse test sequence for microbial challenge studies

Day	Abuse temperature cycle	Number of package sets remaining
1	24 hr at -18 °C	5
2	20 hr at -18 °C 4 hr at 38 °C	4
3	20 hr at -18 °C 4 hr at 38 °C	3
4	20 hr at -18 °C 4 hr at 38 °C	2
5	20 hr at -18 °C 4 hr at 38 °C	1

Source: Labuza and Schmidl (1985)

Step 4: Do microbial survival analysis — This is to find out if there are any microbial growth upon temperature abuse or if the inoculated microbes survived the process. Appropriate detection and enumeration techniques should be used.

19.4.3 Applicability

The use of inoculated pack studies conducted by independent laboratories allows a food processor to assess the relative risks that can occur under conditions of temperature abuse of the food product in question. Taking frozen pizza as an example, both the cheese and sausage, if naturally fermented, will have high total counts of bacteria. Since the product is usually partially pre-baked and then frozen, the numbers of vegetative microorganisms will decrease until thawing occurs. Unfortunately, pathogens such as *Staphylococcus aureus* will not be totally inactivated by these treatments. If the product is abused during distribution so severally that the temperature near the surface reaches about 7 °C, pathogens may grow. A challenge study with *Staphylococcus aureus* will verify the microbial safety of the product.

It should be noted that inoculated pack studies with pathogens should not be conducted in food industry laboratories that are located close to the food processing facilities because of the possible transfer of pathogens to food products. No sensory

panel can be applied to evaluate the inoculated samples other than visual observation.

19.5 Accelerated shelf life testing

19.5.1 Basis

During product development, preliminary shelf life knowledge is often needed in addition to microbiological safety. Shelf life testing experiments at this stage are often accelerated to evaluate the effects of various formulation and processing parameters on shelf life stability of the product being developed periodically since one can not afford the relatively long shelf life period for a frozen food stored under normal freezing conditions. In addition, temperature fluctuations may occur in distribution and retail holding for frozen storage. Thus kinetic studies at several temperatures within that range are necessary to predict its shelf life. Accelerated shelf life testing conducted at elevated isothermal temperatures and/or with freeze/thaw cycles for frozen products have been used extensively for several decades by industry and government agencies (Labuza and Schmidl, 1985). The Arrhenius relation and the Q_{10} approach are used to extrapolate the results to the expected lower storage temperature. Acceleration factors other than temperature have also been studied for some other deterioration modes, such as moisture gain or loss and lipid oxidation (Labuza, 1984), but rarely done for frozen foods.

19.5.2 Unique procedures

Step 1: Clarify test objectives — In general there are two occasions where ASLT applies: i) estimate approximate shelf life quickly during development stage; ii) collect kinetic parameters for actual shelf life prediction as in the marketplace, which is conducted generally near the launch phase.

Step 2: Select accelerating temperature conditions — Suggested isothermal accelerating conditions for frozen foods are -15, -10, and -5 °C with a control stored at < -40 °C (Labuza and Schmidl, 1985). The inherent assumption is that the deterioration mechanism is the same across the temperature range although as noted earlier, there is concern about how close to freezing one can go.

Moisture migration from the food into the surrounding air with resulting desiccation of the food and ice crystal formation in the package is a major mode of deterioration of frozen foods under fluctuation temperature conditions. Cycling temperature storage is used to test for this, i.e. from 0 °F or 10 °F up to 20 °F with one day at each temperature and then repeated several times. A freeze-thaw cycling study is also needed to determine its effect on sensory quality. Usually, the high temperature

can be much lower than that used in a microbial challenge study unless microbial survival is still a concern. Typically, cycling temperature/time can be three to five 24 hour cycles between -18 °C and -7 °C, or between - 18 °C and 7 °C, depending on the product.

Step 3: Estimate testing time and sampling frequency— Testing times are dependent on a desired shelf life at target storage conditions. For example, given that a shelf life of 12 months at -18 °C is desired, a shelf life plot can be constructed. Figure 19.11 indicates the test time at -4 °C that equates to 12 months at -18 °C for various Q_{10} values. Sampling times at -4 °C should thus be 1 wk, 2 wk, 1 month, 3 months, and 4.5 -5 months. Most published results suggest that Q_{10} values for vitamin C loss and quality loss in frozen vegetables range from 2 to 20 and that the shelf life of vegetables is only 6-8 months at -18 °C (Labuza, 1982). Considering these Q_{10} values, a product that does not retain good quality for 4.5 months at -4 °C may not retain good quality for 12 months at -18 °C. This also suggests the sampling frequency shown in Table 19.6. All simple tests should be conducted at each sampling time, while sensory testing should be concentrated mainly toward the end of the test sequence with a few near the beginning.

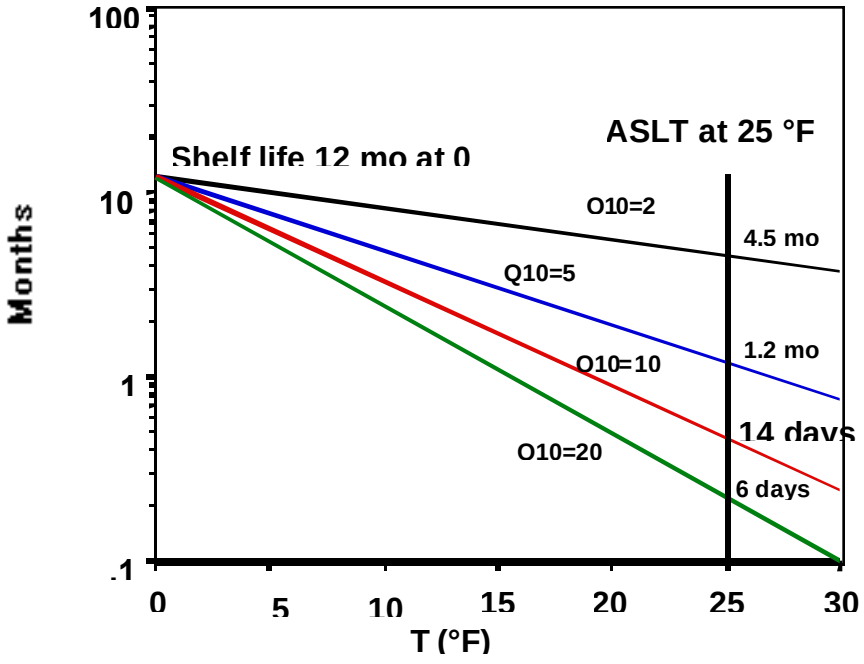

Figure 19.11 Shelf life testing times at 25 °F equivalent to 12 mo at 0 °F for various Q_{10} values.

Table 19.6 Sampling frequency for frozen pizza ASLT

Temperature (°C)	Sampling times (wk)
- 4	1, 2*, 3, 4, 5, 8, 12, 14, 16*, 20*
- 7	2, 4*, 10, 15*, 20*
- 10	4*, 10, 15*, 20*

* Sensory test times Source: Labuza (1986)

Step 4: Determine end point — Figure 19.12 shows a comparison of times to various levels for the loss of vitamin C in frozen spinach as a function of temperature (Kramer, 1974). The dotted line represents the 80/80 rule, i.e., from a legal standpoint, for natural products, 80% of the tested sample must have no more than a loss of 20% (i.e. 80% of the label value). Consumer sensory testing will not always give such a clear shelf-life result since different shelf life times can result using different quality attributes. Often professional judgment has to be made to decide what factor to use as the base for the end of shelf-life of the product. When shelf life is unacceptably short, adjustments should be made to the food, its environment, packaging, process and hygienic conditions, until a suitable extension of shelf life can be achieved. For some products, the test results may demonstrate that the target shelf life is not attainable. At this point, the question of whether to launch the new product with a shorter shelf life or to abandon the entire project becomes a marketing decision.

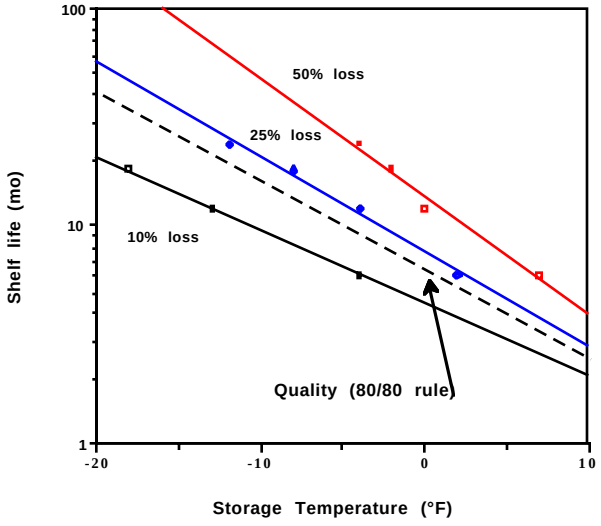

Figure 19.12
Shelf life of frozen spinach as a function of vitamin loss level

Step 5: Estimate kinetic parameters — From each test storage condition, estimation of k or q is needed to make the appropriate shelf life plot. From this one can then estimate the potential shelf life and confidence interval for the storage condition. Then parameters for the Arrhenius relation and the shelf life plot are determined by linear regression, which are used for shelf life prediction.

Step 6: Extrapolate to normal freezing storage condition — The most useful shelf life information is obtained for product kept at its intended storage temperature, which is about -18°C for retail frozen products and -23°C for distribution of frozen foods. Figure 19.13 demonstrates how the shelf life plot is used for extrapolation. It is always a good practice to compare a model's prediction against actual experimental results because of the potential for errors from using the higher temperature data as noted earlier besides the other errors suggested by Labuza and Riboh (1982). In addition, the existence of a glass transition at a temperature between the test temperature and the prediction temperature would lead to error as shown by Nelson and Labuza (1994). In the case of frozen foods, most likely the error would be an under prediction of the shelf life.

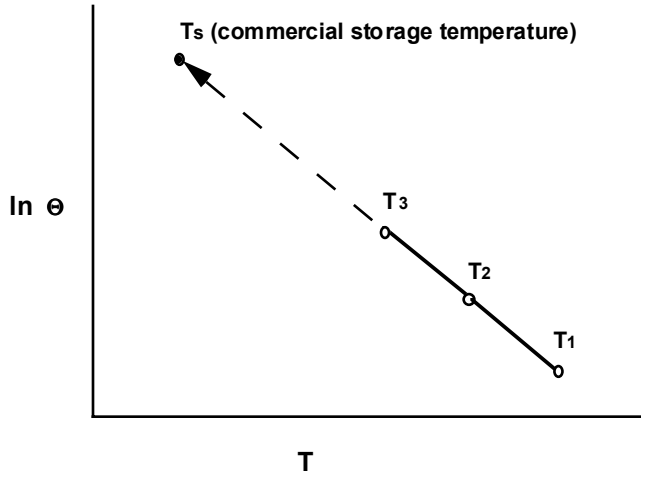

Figure 19.13 Extrapolation from ASLT

Step 7: Predict quality loss for a fluctuating time-temperature distribution — The prediction is based on two assumptions: (1) that there is no history effect from the time-temperature variation and (2) that the key deterioration mode does not change as a function of temperature. The frozen spinach data shown in Figure 19.12 is used in the following example in Table 19.7 for a time-temperature distribution. The line

equivalent to 20% loss is set as the end of shelf life limit i.e., if A_o = 36 mg/100 g then A at the end of shelf life is 0.8 x 36 or 28.8 mg or 7.2 mg of vitamin C can be lost. For each temperature of exposure, the time on the 80/80 line is the time for 20% loss, thus at -10°F, the 20% loss (equivalent to 100% shelf life) time is 16.5 months. Thus for 6 months storage at -10°F, there is 6/16.5 or 36.3% of the shelf life lost and the amount left is 36 - 6.36 x 7.2 = 33.4 mg.

Table 19.7 Estimation of quality remaining of frozen spinach after exposed to a variable time-temperature history with A_o = 36 mg/100g spinach.

Temperature (°F)	**Time t** (months)	**θ** shelf life (months)	**fcon (t/θ)**	**Σf_{con}**	**$A_{remaining}$** (mg/100g)
-10	6	16.5	0.363	0.363	33.4
+3	1	4.5	0.256	0.619	31.5
+12	0.25	1.6	0.156	0.775	30.4

Since as noted 80% of A_o is equal to 28.8 mg/100g at end of shelf life, this product is still acceptable at the end of the set of three different time/temperature exposures. In fact, the shelf life left @ 5 °F = (1-0.775) x 3.3 = 0.74 months = 22 days.

19.5.3 Applicability

Because of relatively long shelf life for frozen foods and the unique feature of freezing, the degree of temperature elevation is largely limited. Prediction of actual shelf life from ASLT may be severely limited except in very simple food systems. Frozen foods such as frozen pizzas, may present problems with moisture migration. The moisture may diffuse from the pizza sauce which has a higher a_W into the crust containing a lower a_w, creating a pizza crust that is limp and soggy. Product development scientists should only use the results as a guideline and must use as many storage conditions as possible to minimize prediction errors.

ASLT is just a quick method, which can not replace the normal storage tests discussed next. Once it is verified that the extrapolation may be wrong, i.e., too large an error, then a careful look should be taken at the deterioration mode, the experiment design and procedure, the data collected and the model developed. If the extrapolation under predicts the true shelf life, then it becomes an economic concern, it is over predicted, then reformulating may be necessary. If the shelf life prediction indicates that the product meets the stability expectation, then the product has a chance of performing satisfactorily in the marketplace.

19.6 Confirmatory storage study

19.6.1 Basis

The difference in potential shelf life should be considered when scaling up from experimental test batches to pilot plant and then to full scale production. Experience has shown that results of small-scale experiments in the laboratory may not be of much use for large-scale production (Graf and Saguy, 1991). Scale-up not only affects the processability and quality of a food product, but it often alters its shelf life. Depending on the mode of failure and the food scientist's approach to inhibiting microbial growth and chemical reactions leading to deterioration, scale-up may increase or decrease shelf life. Because of the difficulty in predicting the precise effect of scale-up on food stability, a confirmatory shelf life study is often needed (Graf and Saguy, 1991) simulating real distribution conditions (if available) using fluctuating temperatures or freeze/thaw cycles. Ideally the products to be tested are produced by the plant under typical steady-state manufacturing conditions. In this way the test can confirm that one can achieve an acceptable product quality and acceptable shelf life of a realistic food (not hand-picked showcase samples) at its intended commercial storage temperature conditions.

19.6.2 Unique procedures

Step 1: Identify real distribution chain conditions — The final decision as to the likely shelf life of a given frozen product must taken account of the fact that the product is not going to be stored at a steady temperature of -18°C (0°F) from the end of freezing until thawing for consumption or during end cooking. A typical frozen product will spend part of its shelf life in a bulk cold store, a refrigerated vehicle or container, a distribution store, a retail display cabinet or institutional frozen food storage cabinet, a period out of refrigeration during the journey from the retail outlet to home and time in a home freezer or star-marked frozen food storage compartment, before being

consumed in the frozen state, thawed or end cooked. An essential part of any shelf life determination in markets where the 'marked' frozen food storage compartments are in use is to assess the shelf life at the relevant temperatures for '1-Star', -6°C (21°F) and '2-Star', -12 °C (10°F) conditions (Symons, 1995).

Step 2: Determine shelf life — Shelf life determination can be done either using a kinetic modeling study or by sensory evaluation. Figure 19.14 is the same shelf life plot for frozen strawberries using all the data as was presented by Guadagni (1968). Based on that data, a demonstrative calculation for a known time-temperature distribution can be conducted and is summarized in Table 19.8. The calculations are based on the following. It is assumed that the order is zero and that at time zero the quality is 100%. At end of shelf life the quality level is set to zero percent. Thus the rate of loss is 100% divided by the shelf life time as found in Figure 19.14. Thus for the first period, which is holding in the producers locker for 250 days at -22°C, the rate of loss is 100/660 or 0.15% per day based on a shelf life of 660 days at the temperature. Given that the holding time was 250 days the accumulated loss is 250 x 0.15 or 38% loss. As seen there is a total of 66% loss up to the point of the consumers freezer. If consumed within three weeks there is still 22% of the shelf life left. Another way of looking at it, at -13°C with a loss of 66% of the total shelf life, there is 0.34 x 180 or about two months of shelf life left. The question would be “is this enough”.

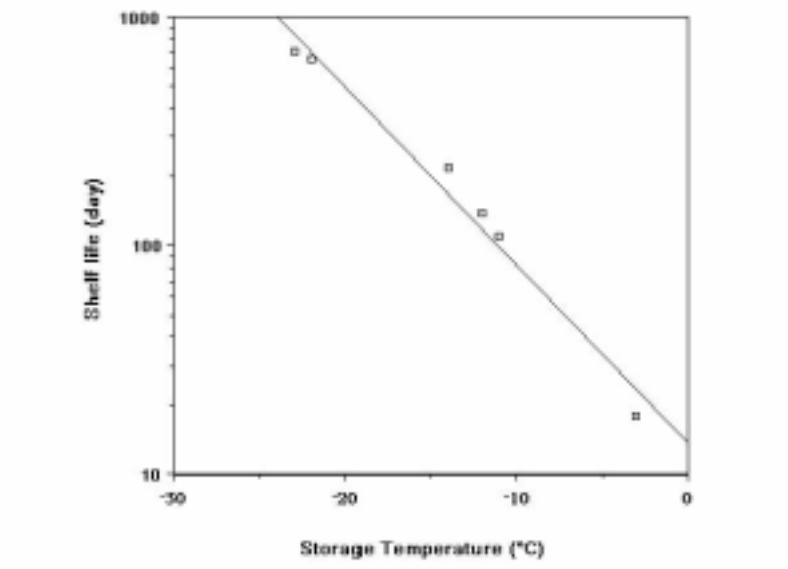

Figure 19.14 Shelf life plot for frozen strawberries from Guadagni (1968)

Table 19.5 Loss of shelf life of frozen strawberries Source: Jul (1984).

Stage	Time (day)	Temperature (°C)	Shelf life θs	Loss rate per day = 100/θs	% Loss
Producer	250	-22	660	0.15	38
Transport	2	-14	220	0.45	1
Wholesale	50	-23	710	0.14	7
Transport	1	-12	140	0.71	0.7
Retail	21	-11	110	0.91	19
Transport	0.1	-3	18	5.56	1
Subtotal	324				66
Home freezer	21	-13	180	0.56	12
Total	344				78

Step 3: Correlate sensory with instrumental analysis — Since at this stage effective methods intended for routine quality control must be established, a correlation to some simply measured objective index must be made.

19.6.3 Applicability

This type of test is usually carried out towards the end of the product development process, most certainly using product samples obtained during pre-launch runs. This will finally enable the shelf life to be set and the material, product, process, and packaging specifications to be finalized prior to a full scale product launch. However, for frozen products, it is likely that product launch may occur before any confirmatory shelf life determination is completed. In this case, an ASLT for products manufactured on the scale-up line can be employed simultaneously. Without ASLT, no prediction can be made for other variable storage conditions (e.g. another distribution chain as the market expands). In many cases sensory techniques must be used simultaneously to determine the practical shelf life. These tests require a lot of samples, a lot of panelists and are thus relatively expensive and may be passed over.

As discussed earlier, individual packages will experience a series of different environments corresponding to different locations during distribution and storage. Other packages from the same consignment will meet conditions which differ both in duration of storage and by traversing different parts of the distribution system, different temperatures and humidities. Thus, one cannot assume that all packages will be stored for the same period under identical conditions, or will react in the same way,

that is why methods or devices that integrate time-temperature exposure are required. In 1996 the USDA and FDA both began to examine the potential for using such devices for monitoring safety during distribution. (Anon, 1996)

19.7 On-going shelf life monitoring

19.7.1 Basis

It is very important that the shelf life of every existing product should be monitored on a regular basis and in such a way that is compatible with the shelf life in question, production volume and environmental conditions to which the product is exposed (or even abused) up to the point of consumption. During storage and transportation distribution phases, temperature conditions are often less than ideal and temperature abuses can occur. Reaction rates that are related to quality loss characteristics are strongly temperature dependent, even when all the other factors are controlled through effective packaging and maintained at the desirable levels. Ideally, what would be needed is a cost effective way to either maintain temperature or to individually monitor the temperature conditions of food products throughout distribution in order to indicate their real quality state. If either one is achieved, it could lead to an effective quality control of the distribution, optimized stock rotation and reduction of waste, as well as give some meaningful information on the remaining shelf life of the products. This calls for control over pallets, control over the cases of product and, finally, over the individual packages at every step in distribution. Control over pallets is generally achieved by the computerized recording system of the producer in concert with the bulk storage facility. Control over cases is achieved in Europe by printing the mandatory open shelf life date which is demanded on individual packets on the outer case in standard format. Time-temperature indicators were developed to monitor individual packages but could also serve in both other functions. (Labuza et al., 1991).

19.7.2 Time-temperature indicators

19.7.2.1 Principle

Broadly speaking, a time-temperature integrator/indicator is a device or tag that can keep track of an accumulated time-temperature distribution function to which a perishable product is subjected from the point of manufacture to the display shelf of the retail outlet, or even to the consumer. The operation of a TTI is based on

mechanical, chemical or enzymatic systems that change irreversibly from the time of their activation. The rate increases at higher temperatures in a manner similar to most chemical reactions. The change is usually expressed as a visible response in the form of a mechanical deformation, color development and color movement.

TTIs can be classified into: i) temperature indicators — which would indicate if the package is below a predetermined temperature (Billet, 1983); ii) temperature abuse indicators — For guidance on whether a frozen product has been exposed to temperature abuse, a small ball or cube of ice has often been placed in the frozen food pack. If it disappeared or has lost its shape, one would know that the product has been above 0·C.; iii) temperature abuse integrators — a device which integrates the time and the severity of the abuse, i.e. the sum of the multiples of degrees in degrees Fahrenheit over a critical point and the time of excess in minutes. In the frozen food range, these devices are available for 0°F, 10°F, and 20°F, but may be supplied with other melting points (July, 1984); iv) electronic or mechanical time-temperature integrators —Much effort has been devoted to developing sophisticated electronic time-temperature integrators from which data could be downloaded and the accumulated effect on shelf life calculated for a certain food after its passage through the freezer chain (Olley, 1976). Many companies supply such instruments (Ryan, Cox, Sensitech) but no company presently has put together spread sheets to do the analysis as we have shown previously. In fact some talk about the area under the T-t curve as being important (i.e. number of °C/days) when in fact it is not. One company (Remosys, UK) has an electronic integrator that displays the days of shelf life left for fresh fish. The square root temperature dependence model is used.; (v) chemical time-temperature integrators. These devices show the physical or color changes as noted earlier and respond with the same temperature sensitivity (Q_{10} or E_a) as the food they are monitoring. Companies that supply such devices are 3M (St. Paul, MN), Lifelines (Morristown, NJ) and VITSAB (Malmo, Sweden). Note this latter company marketed TTIs formerly as the i-Point Company. A general review has been done by Labuza et al. (1991).

19.7.2.2 Correlation of a TTI with a food

Figure 19.15 shows a schematic of how a TTI can be used to predict the shelf life of a food product based on mathematical modeling. The left side of this scheme indicates that tag kinetics are required. As noted, these kinetics have been determined for several types of TTI's. The tag basically predicts an effective temperature for a variable distribution which is then used to predict shelf life of the food using the right side of the scheme. This effective temperature is defined as that constant temperature, which if the tag was always at, would give the same measured response as for the variable temperature exposure. The assumption is made that the effective temperature of the TTI response is equal to that for the food deterioration based on the Arrhenius equation, the TTI response can then be integrated for a variable time-temperature distribution. The rate constant for a TTI response must also follows the Arrhenius theory. The $E_{A(TTI)}$ values of the commercially available indicators cover the range of the most important deteriorative reactions in foods including frozen foods (Fu and Labuza, 1992).

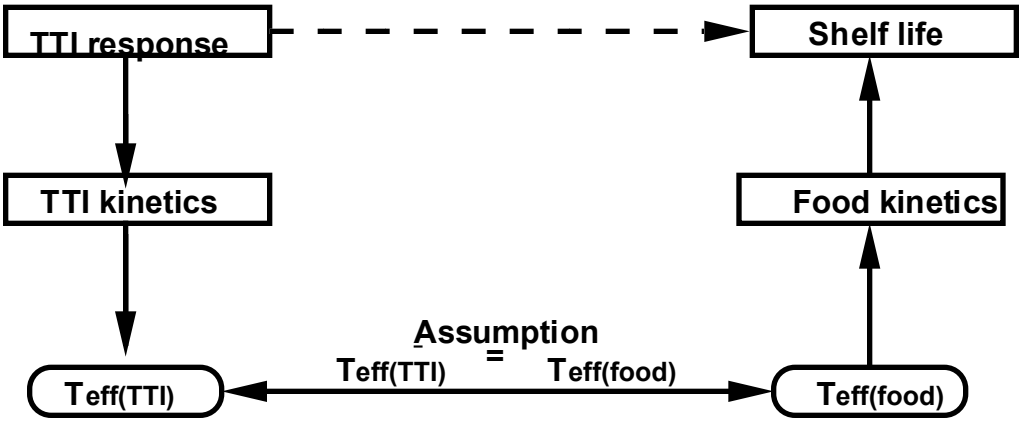

Figure 19.15 — Application scheme of time-temperature indicators

Other approaches such as equivalent time and equivalent point can also be used to correlate TTI response with food reaction based on the Arrhenius relation (Fu and Labuza, 1995). These approaches will be able to provide information on remaining quality or shelf life of the food at a targeted storage temperature after exposure to a variable time-temperature history. It can also be used to compare the effectiveness of different distribution chains.

19.7.2.5 Applicability

The usefulness of these sensors in monitoring quality depends on how well they can mimic changes in quality. It has been proven mathematically that the effective temperature of the TTI will not be equal to that of a food reaction unless the storage temperature is constant or the activation energy of the TTI is the same as that of the quality loss reaction. Thus, the device will only be able to indicate the shelf life remaining for reactions whose activation energy is the same as or close ($DE_a < 5$ Kcal/mol) to that of the TTI or some correction factor is needed (Taoukis et al., 1991). The reliability for TTI applications depends on the variability in responses within the temperature range encountered and the confidence on the determined kinetic parameters and the difference between $E_{A(food)}$ and $E_{A(TTI)}$. Table 19.9 lists some testing procedures for TTI to be used for monitoring frozen food distribution.

In the case of frozen strawberries, a decrease in firmness and a decrease in measured amount of ascorbic acid were well correlated with the response of the i-POINT model 2340 (Singh and Wells, 1987; Wells and Singh, 1988). They also studied the potential applications of TTI for monitoring quality changes during frozen storage of hamburger (Singh and Wells, 1985) and other frozen foods (Wells et al., 1987). It should be noted here that they did not use the tags to predict shelf life, they merely showed a correlation between quality change and tag change. In fact there are no published data giving a comparison of a TTI prediction to the actual quality change of a food. Despite this, some food companies are using the Lifeline type tags on some frozen foods such as turkey rolls (Labuza et al., 1991).

The applicability problem involves deviations from the Arrhenius relationship for both the TTI and food, the heat transfer problem since a TTI tag is usually applied on a package surface and does not reflect the temperature response in the center of a pallet load, and the chemical and light sensitivity of TTIs. The cost of a TTI also depends on the quantity required. All of these aspects and their potential solutions have been discussed in detail by Taoukis et al. (1991).

Table 19.9 — Suggested Technical Standards and Procedures of TTI for Frozen Foods

Test procedure	Technical standard
Temperature response test -25, -15, -10, -5, +5 C	Reproducible end point Maximum tolerance: ±6 days or 2.5% of the life span of TTI
Evaluation of kinetic parameters of TTI Make an Arrhenius plot based on the data collected in the above temperature response test	Arrhenius plot and the values of activation energy and pre-exponential factor
Temperature cycling test Cycling range: -25´ -15 C, -15 ´ -10 C, -10 ´ -5C, -25 ´ +5C Cycling period: 10 times the interned life span of a TTI	The predicted rate constant at the reference temperature from the Arrhenius equation differs from the actually measured value by < 10%.
Simulated field test	Applicable and reliable

From George and Shaw (1992)

19.7.3 Other devices

Alternatives to monitoring temperature during food distribution include the use of flexible, miniaturized electronic temperature recording devices (LeBlanc, 1988). They record time-temperature information that can be displayed and processed at the receiving end by interfacing with a microcomputer. In 1989 a satellite tracking system (Geostar Satellite Tracking Service, Geostar, Corp., Washington, DC, USA) was introduced (Labuza and Taoukis, 1990) but it is only used for drug shipments because of the cost. McMeekin and Olley (1986) gave a list of other time-temperature recording systems. This will provide a valuable record for future use as well as contribute towards evidence of due diligence.

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