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The measurement and character of caramel colour

D. W. GROVER

Summary. The colour of commercial caramels has been studied by measuring the extinction of monochromatic light (λ 0.4 to 0.7 μ) by solutions in water over a concentration range of 0.02-2.5 g/100 ml. The Lambert-Beer 'law' is proved to apply within the accuracy of measurement, and a straight line relationship between $\log E$ and λ is established. Similar observations are made for beverages containing caramel.

Based on these measurements, the colour characteristics of the materials are derived in terms of the Comité Internationale de l'Eclairage (C.I.E.) system, providing explanations for the differences and similarities between caramels, the difficulty of visual matching, and the changes in colour which occur on dilution.

A proposal is made for the assessment of the colouring power of caramel by measurement of extinction at wavelengths of 0.5 and 0.6 μ .

Symbols

λ = Wavelength in microns.	Es_{λ} = Specific extinction at wavelength λ .
I_0 = Intensity of incident light.	λd = Dominant wavelength.
I = Intensity of transmitted light.	P_e = Excitation purity (saturation).
T_{λ} = Transmission = I/I_0 , at wavelength λ .	F_0 = Luminous flux of incident light.
E_{λ} = Extinction = $-\log T_{\lambda}$.	F = Luminous flux of transmitted light.
l = Length of light path (cm).	E_f = Luminous extinction = $-\log F/F_0$.
c = Concentration (g/100 ml).	

Introduction

This paper is concerned with the physics of caramel colour. The chemical and technological aspects of the use of caramel as a colouring material are not considered. There are four main topics: (1) the spectral extinctions of caramel solutions, (2) the Lambert-Beer 'law', (3) caramel colour in the C.I.E. system, and (4) assessment of tinctorial power.

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Materials

Commercial caramels used for this investigation are listed in Table 1.

TABLE 1. Caramels investigated

Code	Application and type
A	Acid beverages; negative
B	Brewing; positive
C	Not known
E	Brewing; positive
F	Brewing
H*	Brewing
I	Spirits; alcohol compatible

*Probably deteriorated by age.

Part 1. Spectral extinctions of caramel solutions

Methods

Aqueous solutions of caramel containing 2.5 g/100 ml were prepared. In one instance where the solution was obviously turbid it was filtered clear. Solutions containing 0.5, 0.1 and 0.02 g caramel/100 ml were then made by successive dilution. Extinctions were measured from $\lambda = 0.38 \mu$ to $\lambda = 0.70 \mu$ with a Unicam 500 Spectrophotometer using 1-cm glass cells. In some instances readings were made at λ intervals of 0.01 μ or closer. However, after it was found that the $\log E$ vs λ plot is an almost straight line, readings were taken at wider intervals, e.g. 0.05 μ .

Results and discussion

Too many individual readings were taken for these to be conveniently tabulated. Instead they are shown graphically. The plots of E_λ against λ for all caramels tested are smooth curves without breaks, showing high extinctions at the blue end of the spectrum and low extinctions at the red end. This result is at variance with the observations of Truhaut *et al.* (1961) who reported maximum extinctions at $\lambda = 0.41 \mu$.

It will be established in Part 2 (p. 315) that, for monochromatic light, the extinction of caramel solutions is proportional to the optical path length and concentration (the Lambert-Beer 'law'). For the moment the validity of this will be assumed. This is expressed in the equations:

$$E_\lambda = E_{s\lambda} \times lc, \quad (1)$$

$$\log E_{s\lambda} = \log E_\lambda - \log lc. \quad (2)$$

Plots of $\log E_{s\lambda}$ against λ for caramels A, B, C, E, F and I are shown in Fig. 1.* It is seen that they approximate very closely to straight lines. By the usual mathematical procedure the best fitting straight lines have been found and are defined below by regression coefficients of $\log E_{s\lambda}$ on λ in the general equation:

$$\log E_{s\lambda} = a + b\lambda. \quad (3)$$

Values of a and b for the various caramels are tabulated in Table 2.

TABLE 2. Regression coefficients of $E_{s\lambda}$ on λ for commercial caramels in equation $\log E_{s\lambda} = a + b\lambda$

Caramel	a	b
A	3.07	-5.08
B	3.36	-5.11
C	3.23	-5.12
E	3.20	-5.22
F	3.28	-5.21
H	3.06	-4.76
I	3.84	-6.56

Similar plots for a pale ale, an Australian whisky and a caramel-coloured cola drink are shown in Fig. 2. The regression coefficients for these beverages are given in Table 3.

These figures can be compared with those for North American beers, calculated from extinction data published in 1962 (Stone & Miller, 1962). The λ vs $\log E_{s\lambda}$ relationships when plotted give almost straight lines for which the regression coefficients are given below:

	'a'	'b'
Canada (7)*	0.25	-5.82
New York (29)	0.10	-5.68
Wisconsin (13)	0.30	-6.27
New Hampshire (5)	-0.01	-5.74
New York (6)	-0.08	-5.69
Wisconsin (9)	0.16	-6.35

* The numbers are those in the original paper.

* For convenience of plotting the graph shows $\log E_{s\lambda} + 5\lambda$. This in no way affects the discussion.

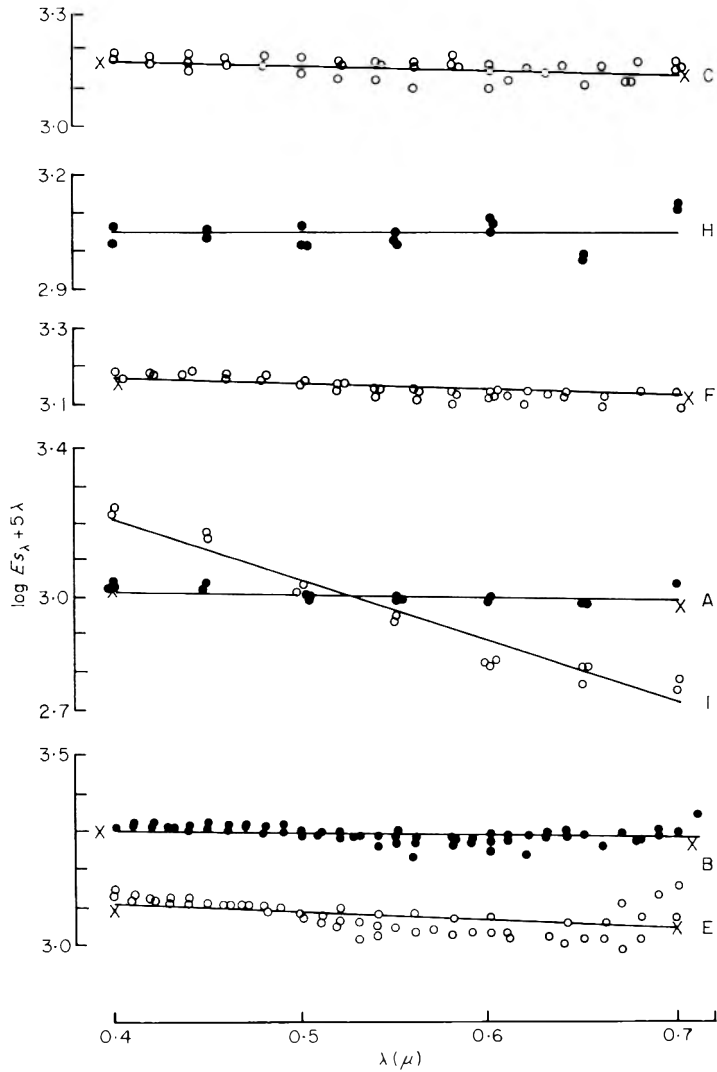


FIG. 1. Relationship between wavelength and specific extinction for some caramels.

TABLE 3. Regression coefficients of $\log E_{\lambda}$ on λ for beverages in equation $\log E_{\lambda} = a + b\lambda$

Beverage	<i>a</i>	<i>b</i>
Pale ale	0.10	-5.57
Whisky	0.28	-5.76
Cola drink	0.37	-5.10

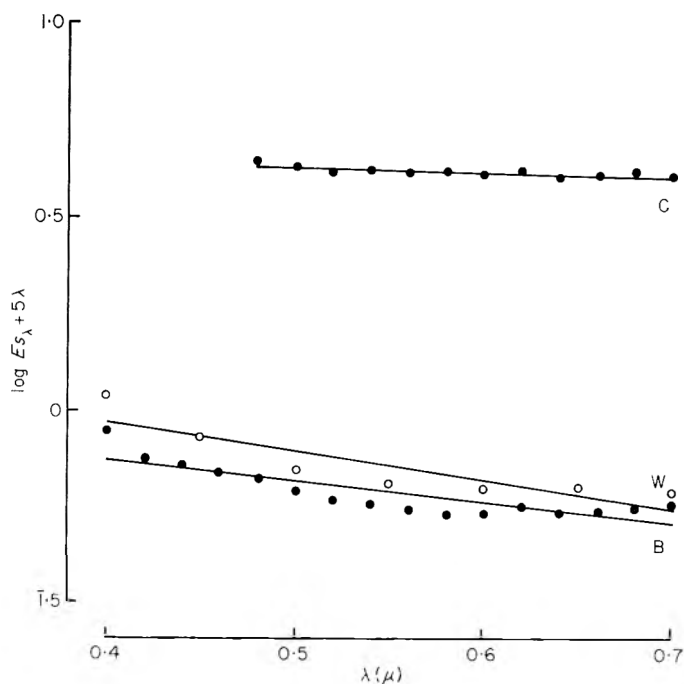


FIG. 2. Relationship between wavelength and specific extinction for some beverages. W, Whisky; B, beer; C, cola.

Discussion

The 'b' coefficients for caramels A, B, C, E, F and the cola drink are almost equal, with a mean of -5.15 . The lines of slope -5.15 which most closely correspond with experimental data fall between the points marked 'x' in Fig. 1. These are so close to the 'best' lines (as drawn in full) as to be almost indistinguishable. A consequence of identical values of 'b' is that a solution of one of these samples can be precisely matched in spectral transmission by a solution of any of the others by suitably adjusting the concentration.

Part 2. The Lambert-Beer (LB) law

This 'law', contained in two propositions propounded respectively by Lambert and Beer, applies only to homogeneous monochromatic radiation. It has been expressed verbally in several ways. Mathematically it is contained in equations (1) and (2). The validity of the LB law has been established for sugar house products—which are similar in many ways to caramel (Stone & Miller, 1962).

One test of the LB law is to find out whether $E_{s\lambda}$ changes as the concentration changes. It will be seen in Fig. 1 that for each caramel $\log E_{s\lambda}$ is almost independent of c . There is some scatter of the observations, notably at higher wavelengths where the use of a

logarithmic function exaggerates the error in measuring small values of E . To establish more formally that $E_{s\lambda}$ is independent of c , the divergence between observed values of $E_{s\lambda}$ and the best fitting line have been correlated with c , for three wavelength ranges. The results are given in Table 4.

TABLE 4. Correlation between deviations from best fitting straight line and concentration

Wavelength range	No. of observation	Correlation coefficient	Probability	Regression coefficient $\log E_{s\lambda}$ on c
0.4-0.49	52	0.223	0.1	—
0.5-0.59	70	0.269	0.023	0.021
0.6-0.71	75	0.209	0.1	—

Although there is a significant correlation in one range the regression coefficient of $\log E_{s\lambda}$ on c is very small, a concentration change of 0.1 being associated with a change of only 0.5% in the specific extinction. It seems likely that the observed correlation is due to a small systematic observational error, rather than to any real departure from the LB law.

The validity of the LB law has also been tested visually in white light. A Hellige colorimeter was used for this purpose. Solutions of different concentrations of the caramel were placed in the two cells. The length l_1 was varied and the length l_2 , required to obtain a match, was observed. Results are given in Table 5, where the observed values of l_2 are compared with values calculated on the basis of the LB law.

The small differences between observed and calculated values of l_2 are not systematic and can be assumed to be due to experimental error.

It is concluded that the LB law applies to solutions of commercial caramels within the limits of accuracy of the present experimental observations, and within the limits of practical importance for the use of caramels as colouring substances.

Part 3. Caramel colour in the C.I.E. system

The value of caramel lies in the character of light transmitted by the beverages and other foodstuffs in which it is used. This is obviously true for transparent materials viewed against the light. It is also true for coloured objects viewed by reflected light, when the coloured effect is produced by penetration of the light a small distance into the coloured material followed by internal reflection.

The visual colour depends on the distribution of intensity of the various radiations in the visual range. Although this can readily be measured with a spectrophotometer, interpretation in terms of visual effect is a complex and incompletely understood problem. In order to bring the relationship of distribution of radiation and visual colour into a form amenable to mathematical treatment, certain assumptions and conventions have to be accepted. The C.I.E. system of colour definition contains such

TABLE 5. The validity of the Lambert-Beer law with white light

Left-hand cell			Right hand cell		
c_1 (g/100 ml)	l_1 (cm)	c_2 (g/100 ml)	l_2 (cm)		
			Observed	Calculated	Difference
0.5	0.5	0.1	2.49	2.50	-0.01
		0.2	1.21	1.25	-0.04
		0.3	0.81	0.33	-0.02
		0.4	0.61	0.62	-0.01
		0.5	0.50	0.50	0.00
0.5	1.0	0.1	4.98	5.00	-0.02
		0.2	2.51	2.50	+0.01
		0.3	1.70	1.67	+0.03
		0.4	1.26	1.25	+0.01
		0.5	1.00	1.00	0.00
1.0	0.5	0.5	0.98	1.00	-0.02
		0.6	0.83	0.83	0.00
		0.8	0.62	0.62	0.00
		1.0	0.50	0.50	0.00
1.0	1.0	0.5	2.02	2.00	+0.02
		0.6	1.67	1.67	0.00
		0.8	1.29	1.25	+0.04
		1.0	1.10	1.00	+0.01

assumptions and conventions, but is well accepted as a good means of practical colour definition. In the C.I.E. system any mixed visible radiation is represented as a mixture of three specific monochromatic radiations (e.g. $\lambda_1 = 0.700 \mu$, $\lambda_2 = 0.546 \mu$, $\lambda_3 = 0.4358 \mu$). The respective amounts of these required to give a match, called the tristimulus (X, Y, Z) values of the mixed radiation, can be computed from the spectral transmission. Any mixed radiation can also be defined as a combination of white light and monochromatic light of wavelength λ_d , where λ_d is the dominant wavelength of the mixed radiation. The colour and intensity of the mixed radiation are then completely defined by three parameters:

F = Luminous flux derived from the tristimulus values.

λ_d = Dominant wavelength.

P_e = Excitation purity.

The light transmitting properties of a transparent coloured medium can similarly be defined by three characteristics, each equivalent to, or identical to one of the three

characteristics of the transmitted light discussed in the previous paragraph, and each independent of the intensity of the incident light.

These are:

E_f = Luminous extinction = $-\log F/F_0$ (for an equal energy source).

λd = Dominant wavelength.

P_e = Excitation purity.

The relationships between E_f and, respectively, λd and P_e for two hypothetical caramels are shown in Figs. 3 and 4. The caramels, marked respectively '5.15' and '6.56' are assumed to have the following $\log E_\lambda$ equations:

$$\text{'5.15' } \log E_\lambda = 3.09 - 5.15\lambda + \log lc,$$

$$\text{'6.56' } \log E_\lambda = 3.84 - 5.56\lambda + \log lc.$$

In the figure are also marked the values of P_e and λd for the three beverages included in Table 3.

Discussion

The fact that the majority of caramels have parallel straight lines for the $\log E_{s\lambda}$ vs λ plots (Fig. 1) means that any solution of one can be identically matched by a solution of the correct concentration of any of the others. Some materials, e.g. caramel I, covered by this paper are not alike in this way. Differences between them are illustrated in the graphs of Figs. 3, 4 and 5.

The dominant wavelength, λd , is of immediate interest. Fig. 3 demonstrates a familiar phenomenon—the change in colour from deep red to yellow which occurs when a caramel solution is progressively diluted. The dominant wavelength of light transmitted through dilute solutions of low extinction is about 0.575μ , while for concentrated solutions of high extinction it is about 0.658μ . The cola drink in a depth of 10 cm has $\lambda d = 0.619 \mu$. As an indication of the visual quality of these wavelengths the colour ranges are set out in Table 6.

TABLE 6. Visual appearance of monochromatic light of various wavelengths

Colour ranges = $0.57\text{--}0.62 \mu$ (Kelly, 1943)	
Above 0.618	Red
0.596–0.618	Reddish orange
0.586–0.597	Orange
0.580–0.586	Yellowish orange
0.575–0.580	Yellow
0.570–0.575	Greenish yellow

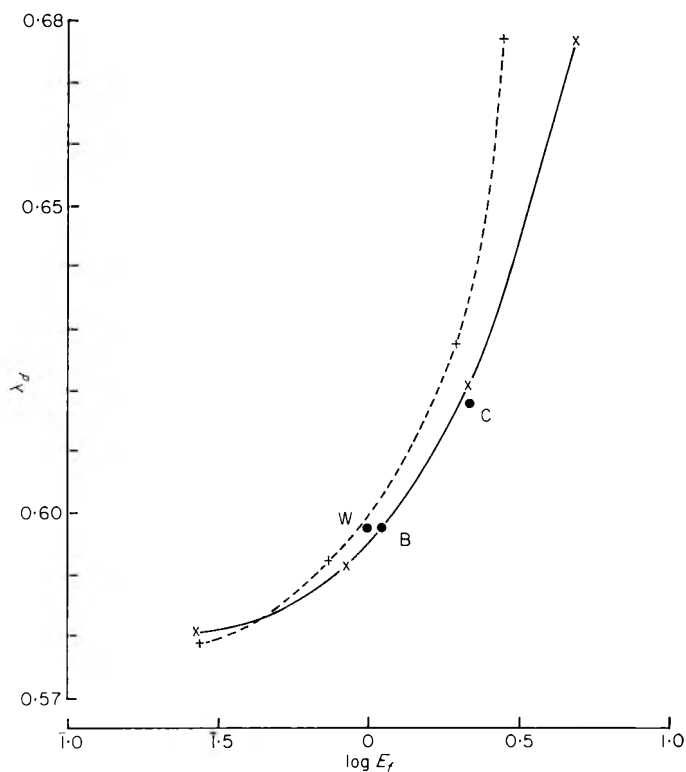


FIG. 3. Relationship of dominant wavelength to luminous extinction. +, Caramel '6.56'; x, caramel '5.15'; W, whisky; B, beer; C, cola beverage.

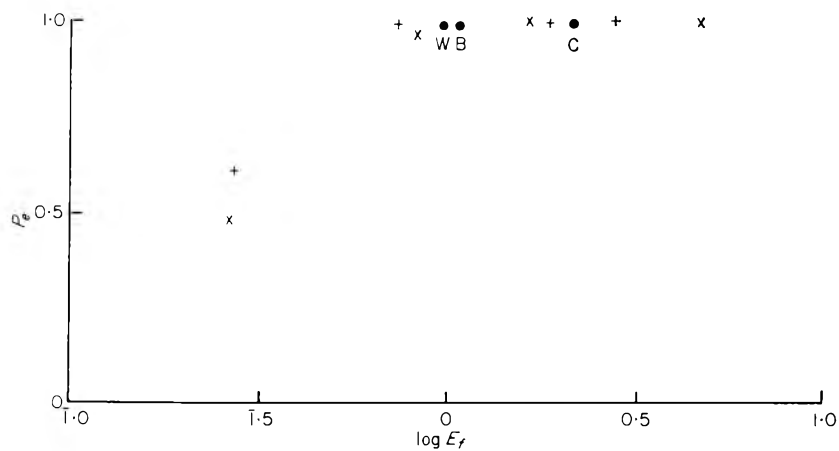


FIG. 4. Relationship of excitation purity to luminous extinction. +, Caramel '6.56'; x, caramel '5.15'; W, whisky; B, beer; C, cola beverage.

Changes in saturation or extinction purity (P_e) with increase of concentration are shown in Fig. 4. Dilute solutions have a relatively unsaturated colour. More concentrated solutions have higher P_e values. When $E_f > 0.9$ (λd about 0.59), $P_e = 1$. Fig. 4 reveals differences between caramels '5.15' and '6.56'. The latter has greater P_e values when E_f is less than 0.

The LB law provides that E_λ is proportional to both the light path and the concentration, and it has already been established that this relationship holds for caramel solutions. However the luminous extinction E_f of caramel solutions is not proportional to $l \times c$. The luminous extinction increases less as concentration is increased than proportionality requires (see Fig. 5).

Part 4. Assessment of tinctorial power of caramels

Several methods of assessing caramel colour are in current use, and the caramel manufacturer is frequently required to specify tinctorial power in different ways for different buyers. The commonly employed methods are of three types:

(a) Visual comparison with some standard reference such as Lovibond (Salamon & Goldie, 1900) or European Brewing Convention (1950, 1952, 1953) glasses.

(b) Comparison with some standard (Truhaut, 1961) reference by means of an absorptiometer.

(c) Measurement of the extinction coefficient of a solution in monochromatic light at one or more wavelengths by means of a spectrophotometer (Gillette & Heath, 1954; Deitz, 1956).

The limitations of visual comparisons are well known. Nevertheless, for routine testing of caramel for a specific purpose reasonably consistent results are obtained. Lovibond glasses have been in use for over 65 years. The E.B.C. comparator has been introduced more recently and is widely applied to caramels for the brewing trade.

Instrumental techniques give more definite and objective, but not necessarily more useful, information. From what has been learned about the light transmitting properties of caramel solutions it is obvious that a single measurement cannot provide a complete colour assessment.

Spectrophotometric measurement

Various suggestions have been made for the best wavelength for measuring the light extinction of caramel and caramel-like solutions (Gillette & Heath, 1954; Deitz, 1956). The International Commission for Uniform Methods of Sugar Analysis (1958) has proposed 0.42, 0.56 and 0.72 μ ; 0.42 μ for lightly coloured material, 0.56 μ for dark materials and 0.72 μ as a basis for correcting for turbidity. In the absence of a spectrophotometer similar readings can be obtained on an absorptiometer using Ilford filters having appropriate transmission bands. The transmittance results however are not exactly equal to those obtained with a spectrophotometer.

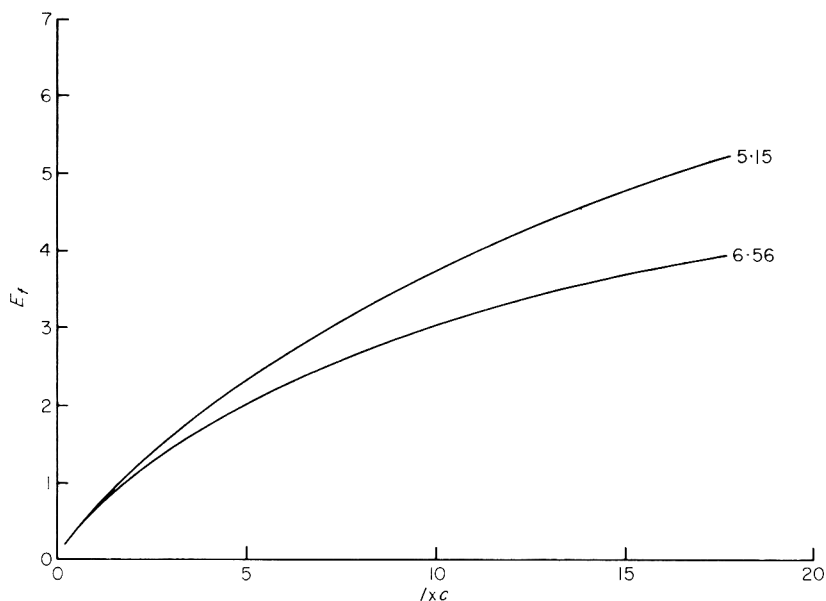


FIG. 5. Relationship of luminous extinction to optical path (l) by concentration (c) for two caramels.

Some theoretical basis for selecting the wavelength is provided as follows:

(a) If two caramels have the same values of ' b ' in the equation (3), their relative colouring power will be the same whatever wavelength light is used for the comparison. If the ' b ' values are different, their relative colouring powers will depend on the wavelength of the light selected.

(b) As an index of the colouring power of a substance it is reasonable to use the extinction of light of a wavelength which is significant in determining the colour of the transmitted light. This criterion can be applied in selecting a wavelength of light for extinction measurement. The light which contributes the most to the luminous flux of the transmitted light is that for which $T_\lambda V_\lambda$ is maximum (where V_λ is the luminous efficiency of the light of wavelength λ).^{*} This has been calculated for solutions of caramel '5.15' for values of $lc = 0.5$ and 2.0 . The maximum values of $T_\lambda V_\lambda$ for these solutions occur at $\lambda = 0.60$ and $\lambda = 0.63$, respectively. A proportional change in transmission of light of these wavelengths therefore has a greater effect on the quality of the transmitted light than the same proportional change in the transmission of light of any other wavelength.

^{*} V_λ is the relative spectral luminous efficiency of monochromatic radiation of wavelength λ for photopic vision as standardized in 1924 by the C.I.E. Consider two radiations of equal luminous flux, and wavelengths λ and 0.555μ (the wavelength of maximum luminous efficiency), and relative energies respectively ϵ_λ and $\epsilon_{0.555}$. Then $V_\lambda = \epsilon_{0.555}/\epsilon_\lambda$.

(c) The light absorbing properties of any of the caramels included in this study are completely defined by the two parameters 'a' and 'b' in the equation (3). Both 'a' and 'b' can be evaluated following the measurement of E_λ at two different wavelengths. The second wavelength is selected to be: (i) sufficiently close to 0.6μ for good measurements of E_λ to be made without changing the concentration of the caramel solution, and (ii) sufficiently far from 0.6μ to provide an accurate base for calculation. A wavelength of 0.5μ satisfies these conditions.

Proposed spectrometric procedure

It is proposed that the specific extinction $E_{s\lambda}$ be measured at wavelengths 0.6 and 0.5μ , the values so obtained to be designated, respectively $E_s(0.6)$ and $E_s(0.5)$.

The colouring power of the caramel is then defined by two figures:

- (i) Specific extinction ($\lambda = 0.6$) i.e. $E_s(0.6)$.
- (ii) The value of b in the equation $\log E_{s\lambda} = a + b\lambda$.

This is derived from the equation:

$$b = -10 [\log E_s(0.5) - \log E_s(0.6)].$$

$E_s(0.6)$ gives a primary measure of the colouring power while b gives an indication of the change in the colouring power on dilution. $E_s(0.6)$ and 'b' figures of some of the commercial caramels included in Table 1 are listed below in Table 7.

TABLE 7. Tinctorial power of commercial caramels

	$E_s(0.6)$	b
A	1.05	-5.08
B	1.97	-5.11
C	1.44	-5.12
E	1.17	-5.22
F	1.43	-5.21
I	0.80	-6.56

These figures are calculated from the data presented in Fig. 1, namely the best straight line through the plot of $\log E_{s\lambda}$ against λ .

Acknowledgments

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References

- DEITZ, V. (1956) *J. Res. natn. Bur. Stand.* **57**, 159.
- EUROPEAN BREWING CONVENTION (1950) *J. Inst. Brew.* **56**, 373.
- EUROPEAN BREWING CONVENTION (1952) *J. Inst. Brew.* **58**, 247.
- EUROPEAN BREWING CONVENTION (1953) *Analytica*, 304E.
- GILLETTE, T.R. & HEATH, W.D. (1954) *Analyt. Chem.* **26**, 1780.
- INTERNATIONAL COMMISSION FOR UNIFORM METHODS OF SUGAR ANALYSIS (1958) *Proceedings*, 12th session (subject 13), p. 51.
- KELLY, K.J. (1943) *J. Res. natn. Bur. Stand.* **31**, 274.
- SALAMON, A.G. & GOLDIE, E.N. (1900) *J. Soc. chem. Ind., Lond.* 301.
- STONE, I. & MILLER, M.C. (1962) *Wallerstein Labs Commun.* **25**, 143.
- TRUHAUT, R., CASTAGNOU, R., LARCEBAU, S. & LASALLE-SAINT JEAN, V. (1961) *Bull. Soc. Pharm. Bordeaux*, **100**, 145.

Ozone treatment of chilled beef

I. Effect of low concentrations of ozone on microbial spoilage and surface colour of beef

G. KAESS AND J. F. WEIDEMANN

Summary. The effects of continuous ozone treatment at 0.3°C on the growth of psychrophilic meat spoilage organisms, grown on muscle slices with equilibrium relative humidities (EH) of 99.3, 98.5 and 98.0% were investigated. Ozone concentrations ranged from 0.15 to 5.0 mg/m³ in air and the micro-organisms included pigmented and non-pigmented *Pseudomonas*, the yeast *Candida scottii*, and the moulds *Thamnidium* and *Penicillium*. A treatment at EH 99.3% with air containing carbon dioxide (11%) and ozone (0.6 mg/m³) was included.

Small but significant inhibitory effects on non-pigmented *Pseudomonas* species and on the yeast *Candida scottii* were obtained with ozone concentrations ≥ 2 mg/m³ (EH 99.3%). A concentration of 0.6 mg/m³ was not always significantly effective with non-pigmented *Pseudomonas* species, but significant decreases were recorded for the slower growing pigmented *Pseudomonas* species.

With ozone concentrations ≥ 0.6 mg/m³, the population density of 10⁸ cells/cm², at which bacterial colonies first become manifest in air (slime point), was increased to about 10⁹/cm². Lowering the EH or introducing 11% carbon dioxide into the storage further delayed the appearance of the 'slime point' of the bacterial population owing to ozone treatment.

In the presence of ozone, the lag phase of *Thamnidium* and *Penicillium* was longer but the growth rate was about the same as that of the controls. No aerial mycelium appeared with ozone concentrations ≥ 0.6 mg/m³.

The colour of the treated muscle surface did not differ from that of controls when the ozone concentration of the storage atmosphere was ≤ 0.6 mg/m³.

Introduction

High concentrations of ozone (about 1000 mg/m³) were needed to prevent growth of bacteria in solutions of organic nutrients (Ingram & Haines, 1949). The application of high concentrations of ozone to obtain a reduction of the microbial population on the surface of meat is, however, restricted by the high sensitivity of meat pigments

to oxidation. A daily ozonization with 10 mg/m^3 for 3 hr (3°C , 90% relative humidity) extended the lag phase of bacteria, but it could be applied only for about 3 days if discoloration was to be avoided (Kaess, 1936). Under the same experimental conditions, the germination of mould spores (*Mucor*, *Penicillium*) was retarded and no aerial mycelium appeared in the period of treatment. Kefford (1948) found a concentration of 10 mg/m^3 (3 hr/day at 1°C , 5°C) effective only when the application started during the lag phase of the bacteria and when the moisture content of the surface was considerably reduced. According to Ewell (1941) the Bunsen-Roscoe reciprocity law holds approximately for ozone concentrations up to 6 mg/m^3 . This allows the effect of ozone to be maintained at a reduced concentration if the time of treatment is increased correspondingly. It is not known whether such a relationship holds for the colour changes of meat.

In the present experiments a continuous ozone concentration was decreased in steps to find the effect on growth of micro-organisms with concentrations at which colour changes of chilled meat remained negligible during storage.

Methods

(1) Preparation of samples

Meat samples were obtained from fresh semitendinosus muscles under sterile conditions. Circular discs with a diameter of 7.6 cm were cut with a cutting cylinder from samples 0.12 cm thick, sliced from the muscle perpendicular to the fibre direction, with a mechanical cutter (Kaess, 1961). The slices fitted sterile, stainless steel sample holders (Kaess & Weidemann, 1962) which exposed a meat area of $35 \text{ cm}^2/\text{side}$ to the ozone.

Equilibrium relative humidities (EH) of 98.5 and 98.0% over the slices were obtained by exposing them, in a duct, to an air stream flowing at a rate of 3.5 m/sec at 2°C and 80% RH, to reduce the moisture content to the values given by Scott (1936) for a definite EH.

(2) Apparatus

The experimental setup described before (Kaess, 1956) was modified by using stainless steel containers for storing samples in an ozone atmosphere. The containers (Fig. 1) did not appreciably affect ozone decomposition. A continuous stream of air (30 l/hr) with EH of 99.3, 98.5 or 98.0% and a constant temperature of 0.3°C was drawn through the containers. The ozone concentrations ranged from 0.15 to 5 mg/m^3 . In two experiments the effect of a concentration of 0.6 mg/m^3 ozone in a mixture of air with 11% CO_2 was tested. The EH was maintained by passing the dry air through distilled water in a gas washbottle and then through a glass coil (20 ft long, $\frac{1}{2}$ in. inner diameter) filled with sulphuric acid by submerging it in a desiccator with the solution having the concentration to establish the EH. The gas was bubbled into the

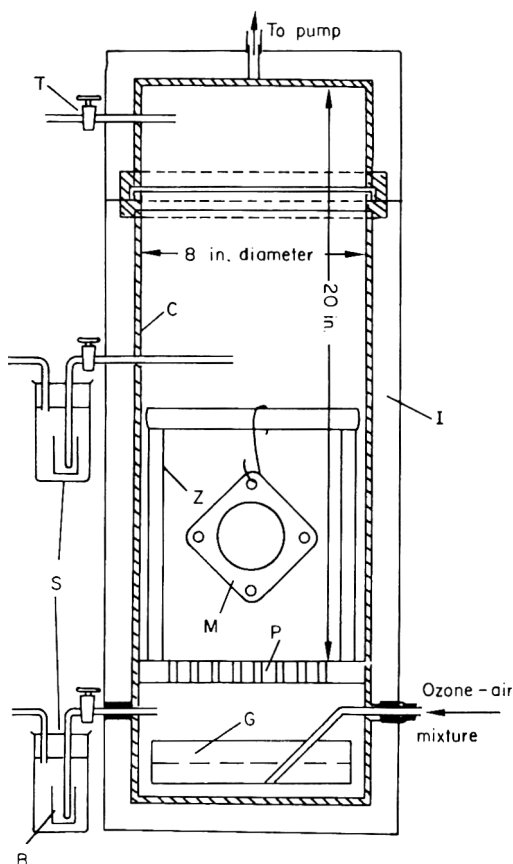


FIG. 1. Stainless steel container for the determination of the effects of ozone on growth of micro-organisms and the uptake of ozone by the meat. Container C with lid. G, Glass dish with sulphuric acid solution with a relative humidity in equilibrium with the meat surface. P, Glass plate with twenty-five drilled holes of $\frac{1}{8}$ in. diameter for uniform air flow. Z, Glass cylinder with sample holder, M, with meat sample. S, Samplers for ozone, with small weighing bottle, B. T, Tap for sampling CO_2 . I, Insulation.

lower end of the coil from a tube entering the desiccator through the inlet of the lid, and left it through a second glass tube.

(3) Ozone generation and gas analysis

Ozone, free of nitrogen oxides, was produced with 'Oliphant' ultraviolet tubes. The ozone concentration was controlled linearly with the primary voltage and with the length of the tube. To obtain the lower concentrations ($\leq 0.6 \text{ mg/m}^3$), the air-ozone mixture was passed through a proportional dividing device, where part of the ozone was destroyed by directing a measured rate of flow of the gas mixture through a flask containing activated charcoal.

Ozone concentrations were determined iodometrically with a micro-method. The liberated iodine was titrated amperometrically (for details see Part II, Kaess & Weidemann, 1968). The carbon dioxide concentration was measured with the Haldane apparatus.

(4) *Microbiological procedures*

Diluted cultures of typical meat spoilage, non-pigmented (Nos. 1482, 131, 39 and A2) and pigmented (Nos. 221, 91 and 41) *Pseudomonas* species, grown for 3 days in nutrient broth at 23°C, were sprayed on the meat samples in the holders with the equipment described elsewhere (Kaess & Weidemann, 1962). Suspensions of the yeast *Candida scottii*, grown in Wickerham's Y.M. broth, were blended in the Buehler homogenizer (E. Buehler, Tuebingen, W. Germany) for 1 min, diluted in 0.5% peptone water and filtered before spraying. Samples of 1.0 cm² were taken with sterile cutting cylinders from each of the two slices used for an experiment and the population determined by the poured plate method. Bacteria were plated on nutrient agar and yeasts on potato dextrose agar. Incubation was for 5 days at 20°C.

Spores were washed off slope cultures of *Thamnidium* or *Penicillium* species with a small quantity of physiological saline, to which a drop of non-ionic detergent had been added as wetting agent. This suspension of spores was used to inoculate meat samples in the centre with the ground end of a sterile glass rod.

(5) *Histological examination*

Pieces of about 1 cm² were cut from meat slices and fixed at room temperature by placing the side free of organisms on top of several layers of filter paper, the lower layers of which were submerged in 10% formalin solution. With this method losses of microbial colonies were avoided. After fixation the samples were dehydrated in an alcohol series, embedded in paraffin and sectioned at a thickness of 10 μ.

Sections of samples with bacterial colonies were stained with Gram stain; sections with mould were overstained with haematoxylin and eosin, and both examined with the light microscope.

(6) *Colour and odour*

At regular intervals the appearance of treated samples and controls was subjectively judged by two persons and the times were reported at which brown and grey components of treated samples could be distinguished from controls. Perceptible odours of bacterial volatiles were assessed subjectively by two persons.

(7) *Statistical analysis*

The significance of the effect of ozone on bacterial growth was tested by examining the difference between the logarithms of counts of treated and control samples at the middle of the time range and the extent to which the difference remained significant

when averaged over increasing ranges of time. The significance of differences was given in all cases for a probability level of 5%. Data were processed in the computer centre of the University of Queensland.

Results

(1) Effects of ozone on growth of bacteria

At 0.3°C and 99.3% EH, ozone concentrations of 0.15–5 mg/m³ delayed moderately the growth of non-pigmented *Pseudomonas* species (Fig. 2). In the presence of ozone

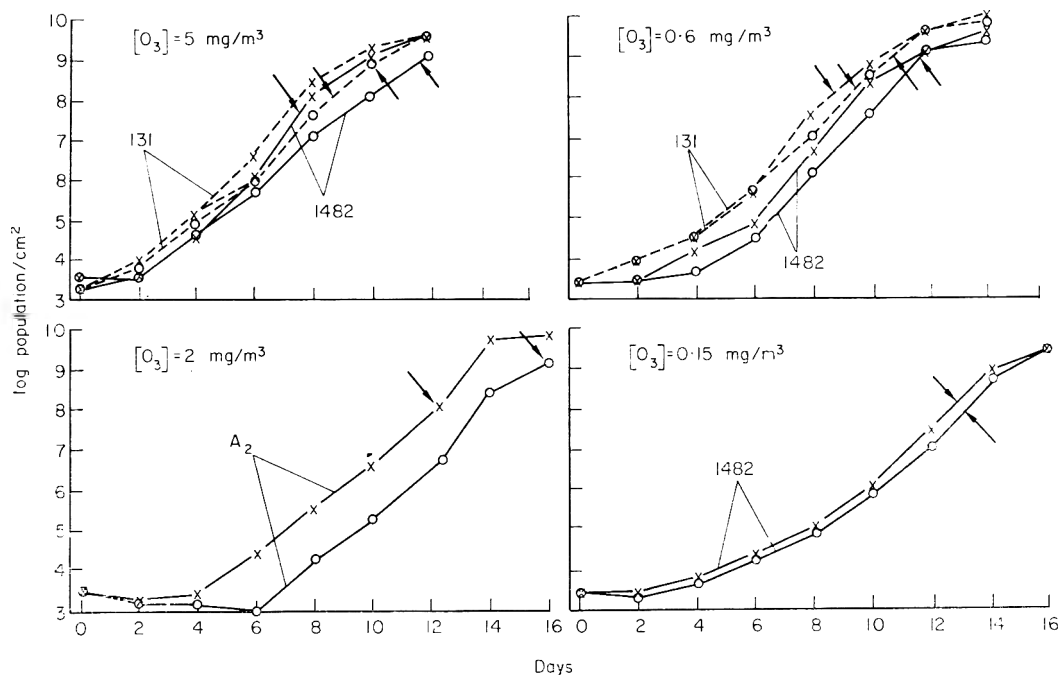


FIG. 2. Logarithm-time growth curves for non-pigmented *Pseudomonas* species 131, 1482 and A₂. Organisms growing on slices of *Musculus semitendinosus* in air or in air-ozone mixtures at 0.3°C and 99.3% EH. Ozone concentrations of 0.15–5 mg/m³. The appearance of slime point is indicated by arrows. O, Ozone; X, air.

the lag phase was increased but the growth rate was not affected. Differences in population density between *Pseudomonas* 131, 1482 and A₂ exposed to ozone concentrations 5 or 2 mg/m³ and controls, as shown in Fig. 2 were significant, except for organisms 1482, for which treatment (5 mg/m³) was significant only towards the end of the storage time. With the low concentration of 0.6 mg/m³ differences between treated and untreated samples were secured for organism 1482 but not for 131. The bactericidal effect practically disappeared with an ozone concentration of 0.15 mg/m³.

The retardation of growth of pigmented *Pseudomonas* species was significant, mainly due to an extended lag phase. In some cases the variance between ranges or within ranges of growth curves was significant, caused probably by the variation in composition of the muscle (connective tissue, fat content) and also by the variation of ozone absorption within and between slices.

The time at which bacterial colonies first became manifest in air at a population density of 10^8 organisms/cm² was delayed until the population increased to approximately 10^9 /cm² (Figs. 2 and 3) with concentrations of ozone ≥ 0.6 mg/m³.

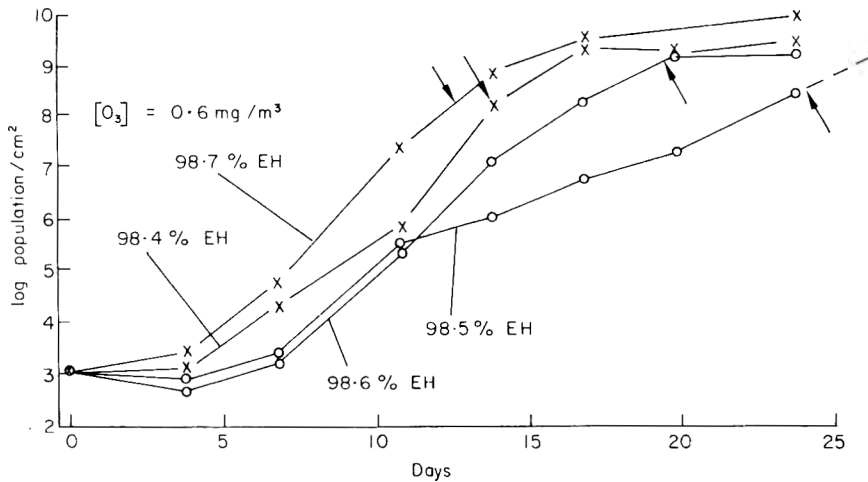


FIG. 3. Logarithm-time growth curves for *Pseudomonas* 1482 growing on slices of *M. semitendinosus* with an EH of approximately 98.5% (exact value indicated on curve) in air and air-ozone mixture of 98.5% EH at 0.3°C. Each curve represents growth on an individual slice. Slime point is indicated by arrows. O, Ozone; X, air.

Histological examination showed that at the time when colonies of bacteria growing in air had merged into a film (Plate 1a), the surface of muscle exposed to an ozone concentration of 5 mg/m³ was free of organisms and colonies were situated predominantly in crevices of the tissue and openings of vessels, where they were protected from the direct effects of ozone (Plate 1b). At lower concentrations of ozone, individual colonies developed leaving sterile areas of muscle between them (Plate 1c).

The distinct dark layer, observed on the muscle surface after storage for 12 days at an ozone concentration of 5 mg/m³ (Plate 1b) was absent on tissue stored at lower concentrations.

(2) Influence of equilibrium relative humidity

Fig. 3 shows the maximum inhibitory effect of ozone (0.6 mg/m³) obtained with the non-pigmented *Pseudomonas* 1482 at an EH of 98.5%. The retarding effects of the ozone

Microbial growth on ozonized meat

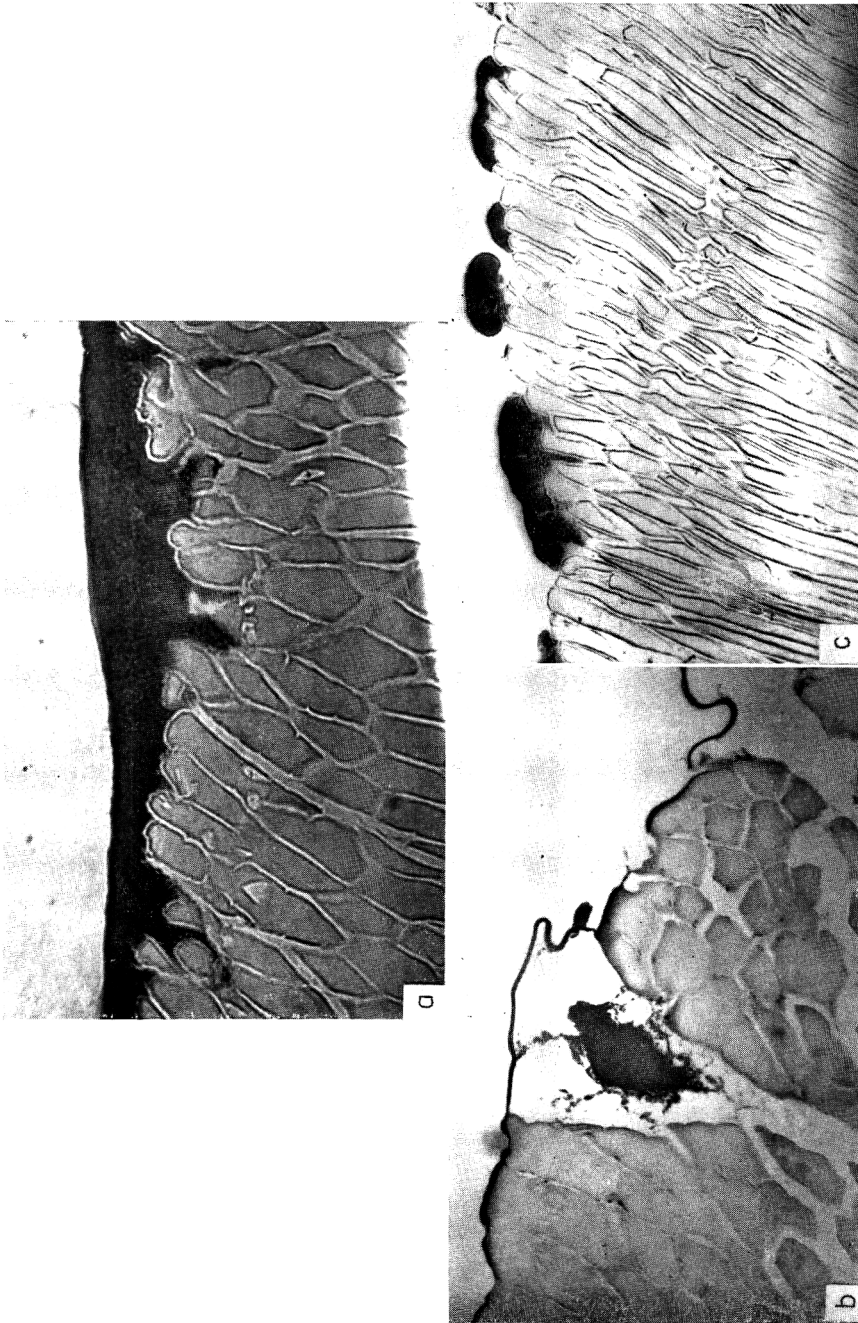


PLATE 1. Sections from *M. semitendinosus*. (a) With slime formation of *Pseudomonas* 1482 grown at 0.3°C and 99.3% EH in air. × 84. (b) With colonies of *Pseudomonas* 1482 grown in a mixture of air with 5 mg/m³ ozone (0.3°C, 99.3% EH), taken at the same time as sample in (a). × 84. Colonies grow in crevices of the tissue. Surface free of bacteria shows dark layer which is probably oxidized tissue. (c) With *Pseudomonas* 1482 grown in air with 2 mg/m³ ozone. Discrete colonies grow on the surface from which the dark layer is absent. × 34.

(Facing p. 330)

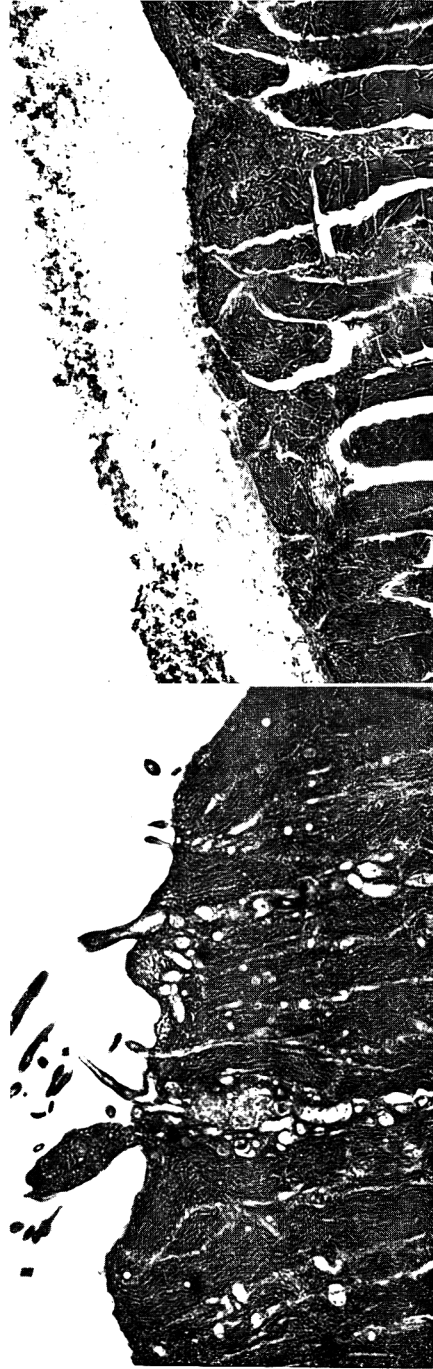
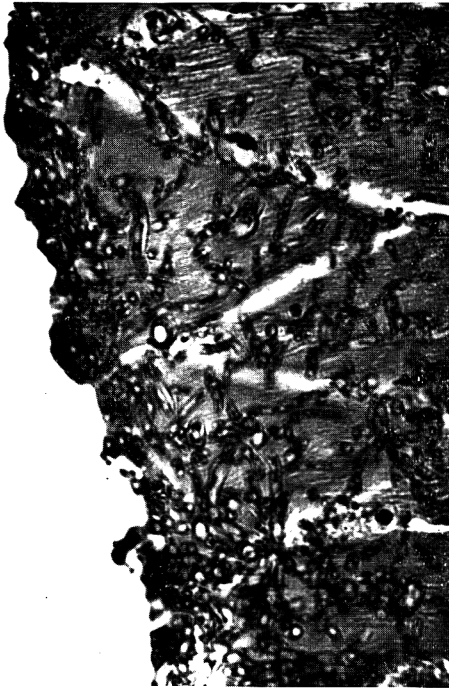


PLATE 2. Sections of muscle slices showing: (top) growth of *Penicillium* in air with 1.9 mg/m³ ozone; (right) in air; and (left) growth of *Thamnidium* in air with the same ozone concentration. Temperature 0.3°C, EH 99.3%. Magnifications, (top) $\times 340$; (right) and (left) $\times 84$.

were significant, but differences were smaller and insignificant in another experiment. The slime point was again delayed until the population increased to 10^9 cells/cm².

With an EH of 98.0% (moisture content 150% dry weight), growth of *Pseudomonas* 1482 in air was prevented; in a mixture of air and ozone (0.6 mg/m³) the population declined slowly and approximately linearly (0.035 log population/day).

(3) *Bacterial growth in air-carbon dioxide-ozone mixture*

Experiments with *Pseudomonas* species exposed to an atmosphere with 11% CO₂ at 0.3°C confirmed earlier results of Scott (1938) which showed that the growth rate of bacteria could be reduced by 50–60% by addition of 10% CO₂ to the air. *Pseudomonas* 131 was the most sensitive to CO₂ of the species tested. With this markedly reduced growth rate the effect of adding 0.6 mg/m³ ozone to the air-carbon dioxide mixture was significant only at the end of the experiment for *Pseudomonas* 1482 and there was no increased effect for *Pseudomonas* 131. Colonies were visible at a cell density of 10^8 /cm² when growing in air with 11% CO₂, but the slime point appeared at a density of about 10^9 /cm² in an atmosphere with 11% CO₂ and 0.6 mg/m³ ozone.

(4) *Odour produced by bacteria*

The colonies produced perceptible odour at a population density of 10^8 – 10^9 /cm², when growing in air. This odour was not perceptible at densities of 10^9 – 10^{10} cells/cm² when the bacteria grew in air-ozone mixtures with ozone concentrations of 0.6 mg/m³ or higher, but was detectable with a concentration of 0.15 mg/m³. With the ozone supply maintained constant in the containers, the ozone concentration close to the muscle also remained constant until a population density of about 10^9 /cm² bacteria was reached, after which a definite drop in the concentration was determined. This decrease was small with a concentration of 5 mg/m³ and largest when the average concentration before the decline was 0.15 mg/m³.

(5) *Effect of ozone on the growth of yeasts*

The growth of *Candida scottii* on muscle slices kept at 0.3°C and 99.3% EH was significantly retarded in air with 5 mg/m³ and 2 mg/m³ ozone, but the effect was insignificant in air with 0.6 mg/m³ ozone. Instead of the moist appearance (slime point) of control samples, a dry dull surface was apparent on treated samples.

(6) *Effect of ozone on the growth of moulds*

The lag phase of *Thamnidium* and of *Penicillium* was increased by about 2 days over that of the controls (Fig. 4) when the moulds developed on muscle stored in air with ozone concentrations of 0.16–5.0 mg/m³. The growth rate of *Thamnidium* was slightly but significantly smaller than controls with ozone concentrations of 5 and 1.9 mg/m³ but not with smaller concentrations. *Penicillium* had a tendency to grow at a slightly higher rate in ozone treated muscle than in controls.

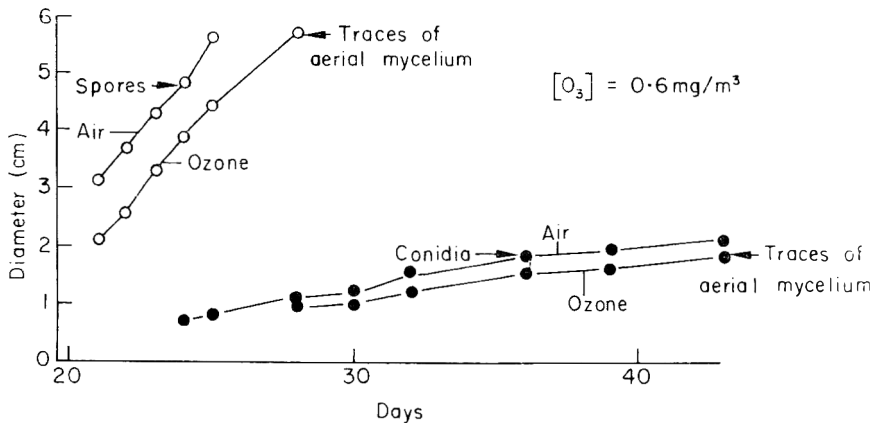


FIG. 4. Increase with time of colony diameter of *Thamnidium* (○) and *Penicillium* (●) growing on slices of *M. semitendinosus* in air and air with 0.6 mg/m³ ozone at 0.3°C and 99.3% EH.

Normal aerial mycelium did not develop on mould growing in air with ozone at concentrations ≥ 0.6 mg/m³. At a concentration of 0.6 mg/m³ traces of mycelium were just visible on the muscle slices at the end of experiment. At an ozone concentration of 0.16 mg/m³ thickened sterile aerial hyphae of *Thamnidium* grew to a length of 1–2 mm (controls about 10 mm.) *Penicillium* hyphae had a length of a fraction of 1 mm.

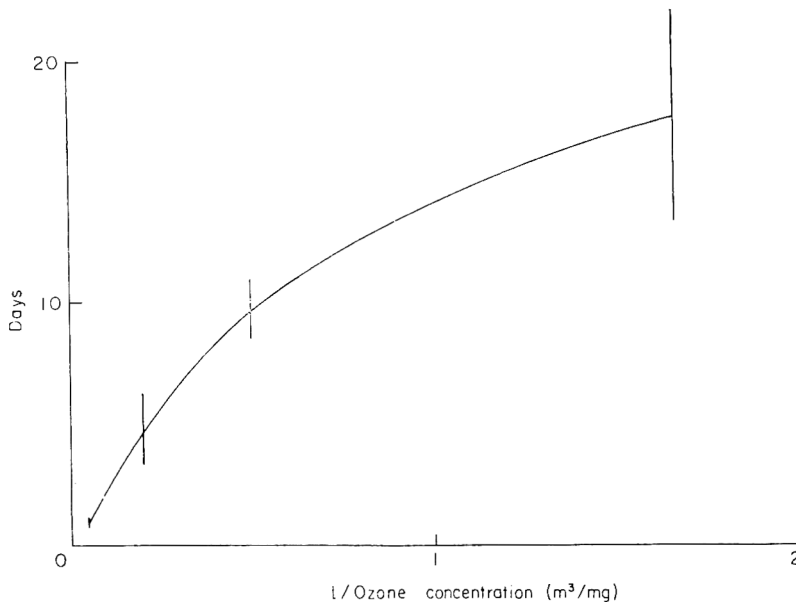


FIG. 5. Onset of discoloration (days) of the muscle surface due to treatment with continuous, constant ozone concentration at 0.3°C and 99.3% EH.

Histological examination showed that *Penicillium* exposed to 1.9 mg/m³ ozone developed freely between and within muscle fibres (Plate 2, top). Aerial mycelium with conidia appeared on the control sample (Plate 2, right). Similar results were obtained with *Thamnidium* but short, single, thickened aerial hyphae showed up on the muscle surface in an atmosphere with 1.9 mg/m³ ozone (Plate 2, left).

(7) *Effects of ozone on colour of meat*

The meat pigments myoglobin and haemoglobin were quickly oxidized to brown heme compounds in an ozone concentration of 2 mg/m³ or more (Fig. 5), but at a level of 0.6 mg/m³ there was no difference in colour between treated samples and controls when the storage life was terminated at population densities of 10⁹ and 10⁸ cells/cm² respectively. Short times between slaughter and the beginning of cooling and treatment, and also high pH values (close to 6) tended to prolong storage times for the onset of discoloration.

Discussion

The need to prevent discoloration of the muscle surface sets low limits to the permissible concentration of a continuous ozone treatment. The absence of a linear relationship between the duration of treatment to produce a brown discoloration and the reciprocal ozone concentration (Fig. 5) excludes using the Bunsen-Roscoe law for the calculation of the concentration-time relationship of discoloration.

The effect on growth of micro-organisms of ozone at concentrations which did not noticeably cause discoloration of the muscle varied with organisms. The reduction of growth was greatest with naturally slow growing pigmented *Pseudomonas* species, but only small when the growth rates of rapidly growing *Pseudomonas* 1482 and 131 were artificially reduced by decreasing the EH of the muscle or by using an atmosphere with 11% CO₂. The retarding effect of ozone (0.6 mg/m³) at an EH 98.5% on growth of *Pseudomonas* was of the same order as that obtained with control samples whose moisture content (% dry weight) was lowered by 20%, i.e. at an EH of 98.3%. The increased inhibition of bacterial growth due to ozone (up to 10 mg/m³, 3 hr/day) with decreasing EH of muscle, which Kefford (1948) found, was not observed with a continuously applied low ozone concentration (0.6 mg/m³).

Ozone was mainly effective on organisms directly exposed to concentrations \geq 0.6 mg/m³. Bacterial growth appeared to be strongly reduced in the distinct dark layer on muscle exposed to an ozone concentration of 5 mg/m³ (Plate 1b). This inhibition was probably due to oxidation of tissue close to the surface. As a consequence of this bacteria started growth in crevices under the surface, formed isolated colonies, and the appearance of the slime point was delayed. At ozone concentrations \geq 0.6 mg/m³ *Thamnidium* and *Penicillium* developed mycelium almost entirely in the substrate.

Optimal conditions for muscle stored at 0.3°C were obtained by applying a continuous ozone concentration of 0.6 mg/m³.

Acknowledgment

The authors gratefully acknowledge the programming of the statistical problems and the calculations with the computer carried out by Mr A. Howard and the skilful assistance of Mrs M. Shevill, Mr B. Mee and Mr R. Turner.

References

- EWELL, A.W. (1941) *Refring Engng*, **41**, 331.
INGRAM, M. & HAINES, R.B. (1949) *J. Hyg., Camb.* **47**, 146.
KAESS, G. (1936) *Z.ges.Kalteind.* **43**, 152.
KAESS, G. (1956) *Aust. J. appl. Sci.* **7**, 242.
KAESS, G. (1961) *Fd Technol., Chicago*, **15**, (3), 122.
KAESS, G. & WEIDEMANN, J.F. (1962) *J. appl. Bact.* **25**, 180.
KAESS, G. & WEIDEMANN, J.F. (1968) *J. Fd Technol.* **3**, 335.
KEFFORD, J.F. (1948) *J. Coun. scient. ind. Res. Aust.* **21**, 116.
SCOTT, W.J. (1936) *J. Coun. scient. ind. Res. Aust.* **9**, 117.
SCOTT, W.J. (1938) *J. Coun. scient. ind. Res. Aust.* **11**, 266.

Ozone treatment of chilled beef

II. Interaction between ozone and muscle

G. KAESS AND J. F. WEIDEMANN

Summary: The initial specific rate of ozone decomposition by muscle was 1.02 ± 0.23 m/hr, for tissue stored at 0.3°C and equilibrium relative humidities (EH) of 99.3, 98.5 or 98.0% in air with ozone concentrations between 0.15 and 38.3 mg/m³, or in air–ozone mixtures containing 11% CO₂. The value was not affected by the presence or absence on the muscle surface of meat spoilage bacteria, yeasts or moulds but considerably lower values were obtained for lean and high fat surface tissues (0.44 and 0.31, respectively).

The slope α of the linear course of the logarithm of reaction velocity–time curves was a power function $\alpha = -9.74 \cdot 10^{-4}[\text{O}_3]^{1.54}$ for ozone concentrations $[\text{O}_3] \leq 5$ mg/m³. The slope was not significantly different from zero when muscle of EH $\geq 98.5\%$ was exposed to 0.7 mg/m³ ozone, but significant values were determined when muscle had an EH of 98.0, and also when tissue (EH 99.3%) was exposed to ozone concentrations > 0.7 mg/m³. Values of α determined for surface tissue and muscle tissue were not significantly different when the same treatment was applied.

Introduction

Earlier experiments (Kaess, 1956) showed that the rate of ozone decomposition in the presence of muscle was in fairly good agreement with the first order law. The logarithm of the specific decomposition of ozone (mg ozone/hr/m²/unit of concentration) by the muscle surface decreased in an approximately linear way with storage time. A thin surface layer with much more intense Gram staining than the remaining tissue was found in histological sections (Kaess & Weidemann, 1968) taken from muscle stored in an atmosphere with an ozone concentration of 5 mg/m³ at 0.3°C and an equilibrium relative humidity (EH) of 99.3% but not with lower ozone levels. The appearance of visible bacterial colonies, which occur on muscle in air at a population density of $10^8/\text{cm}^2$, was delayed in the presence of ozone (≥ 0.6 mg/m³) until the density had increased to $10^9/\text{cm}^2$.

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It was assumed that α , the decline of the specific ozone decomposition with time, depends on ozone concentration and that this relationship may give further information about the development of the intensely stained surface layer and the formation of isolated bacterial colonies. Values of α were determined for ozone concentrations between 0.15 and 5 mg/m³ and for several higher concentrations.

Stoichiometrically equivalent amounts of iodine, i.e. O₃≡I₂, were liberated when high ozone concentrations reacted with neutral potassium iodide (Boelter, Putnam & Lash, 1950; Birdsall, Jenkins & Spadinger, 1952). The agreement of the iodometric method using low (1 mg/m³) or high concentrations was tested by the dilution method. The influence of pH of the potassium iodide solution on the reaction and the effect of concentration of the solution on ozone absorption were checked. The purity of ozone was tested by determination of nitrogen oxides.

Experimental

(1) *Generation and analysis of ozone*

A continuous air stream was drawn through the apparatus previously described (Kaess & Weidemann, 1968). Ozone was produced in few experiments with 'Electrozone' generating tubes and in the majority of experiments with 'Oliphant' ultraviolet tubes.

For the detection of nitrogen dioxide the method of Griess-Illosvay as modified by Salzman (1954) was used. Nitrogen pentoxide was determined with the diphenylamine test of Tillman as modified by Finnie & Yallop (1957).

For the measurement of ozone concentrations (0.15–5 mg/m³), gas samples of 500–8000 ml were aspirated from the storage containers with a Mariotte bottle arrangement at a rate of 10 l/hr. Complete iodine liberation was obtained by bubbling the gas mixture through 2 ml of potassium iodide solution in a 5 ml weighing bottle with a micro-bubbler which produced a slight froth over the surface of the absorbing liquid.

Potassium iodide was dissolved in 0.1 M Clark and Lubs buffers (Bower & Bates 1955), or in 0.05 N-sodium hydroxide when pH 12.6 was required. All solutions were prepared in glass distilled water with AR grade reagents. Potassium iodate was dried at 120°C.

(2) *Amperometric titration of iodine*

The amperometric method of Lindsey (1952), which uses a vibrating platinum electrode, was adapted for working with a small volume (1–2 ml) of solution. To obtain better stirring effects during vibration, the length of the platinum wire cathode immersed in the solution was increased by bending at a right angle a length of 5 mm at the end of the tube. Saturated potassium chloride in a thin plastic tube terminating in a sintered glass tip served as a salt bridge between calomel cell and titration vessel.

The volume of titration solutions was measured with 'Agla' syringes (capacity 0.5 ml), with the delivery needles dipping into the solution.

Iodine was titrated with 0.001 N-sodium thiosulphate or potassium iodate solutions. Bradbury & Hambly (1952) gave an accuracy of 0.1% for such determinations. Blank determinations were made. Potassium iodide solutions were acidified to 0.15 N in all titrations.

(3) Dilution studies

Dilution experiments were carried out by counter-current injection of an air-ozone mixture, with ozone concentrations up to about 4200 mg/m³, at a constant rate into the main air stream in a glass mixing tube. With a flow rate of 20 l/hr in the glass tube (90 cm long, 1 cm inner diameter) Reynold's number was 3100. Injecting dye in a stream of water with the same Reynold's number showed that mixing was complete within 2 cm of the injection nozzle.

Ozone concentrations of the injected and the diluted ozone-air mixtures were measured and the rate of flow determined in both cases with rotameters. The air was passed through a container with activated charcoal to remove volatile impurities. A cotton wool filter prevented dust from the charcoal moving into the mixing tube.

4. Reaction of ozone with muscle tissue

Two slices (1.2 cm thick) of beef semi tendinosus muscle held in stainless steel frames (Kaess & Weidemann, 1962), which exposed a muscle area $a = 0.014 \text{ m}^2$ (both sides of samples) to the ozone-air mixture ($r = 0.030 \text{ m}^3/\text{hr}$), were suspended in each of two storage containers. Ozone concentrations were measured immediately before the gas mixture entered the reaction space $[\text{O}_3]_e$ and immediately after it passed the meat slices $[\text{O}_3]$. The velocity constant (k_c in hr^{-1}) for the decomposition of ozone in the containers with stainless steel frames without meat was determined separately; the specific rate of ozone uptake by muscle A_m (Kaess, 1956) at any time was then calculated from the relation:

$$A_m = \left[r \left(\frac{[\text{O}_3]_e}{[\text{O}_3]} \right)^{n-1} - vk_c \right] / a$$

where v was the volume of the reaction space in m^3 ; A_m was determined for a number of storage times at 0.3°C, and EH of 99.3, 98.5 or 98.0%, for practically sterile muscle and for muscle populated with psychrophilic bacteria, yeasts or moulds. In some instances the storage atmosphere contained 11% carbon dioxide.

The regression of logarithm A_m on time is reported as a function of ozone concentration. In the presence of bacteria on the meat surface the determination of the regression was restricted to the period in which ozone decomposition was not affected by bacterial volatiles (Kaess & Weidemann, 1968).

Results

Ozone analysis

The 'Oliphant' tubes gave pure ozone. 'Electrozone' tubes produced small amounts of nitrogen pentoxide (0.4% w/w) at a high ozone concentration (3600 mg/m³).

Neutral phosphate buffer (0.1 M) solution containing 0.2 N-potassium iodide was required to obtain optimal ozone absorption (Fig. 1). Using potassium iodide solution

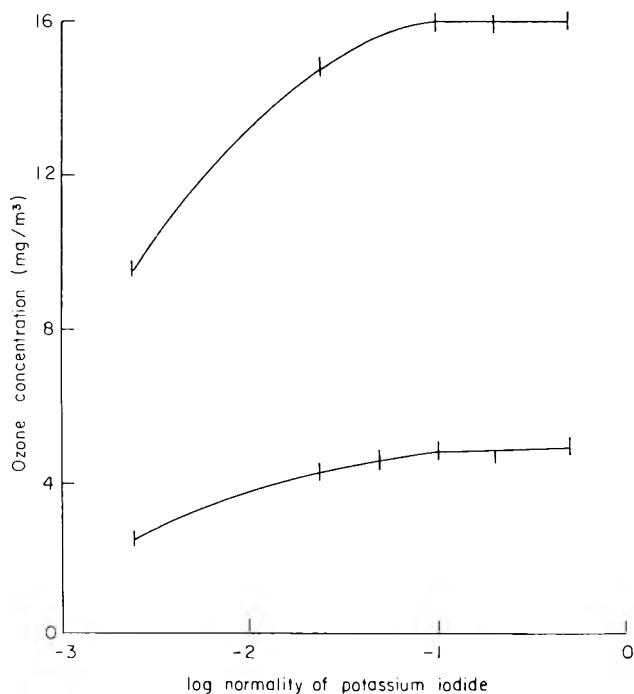


FIG. 1. Iodometrically measured ozone concentrations of two ozone-air mixtures. Vertical lines indicate fluctuation of two to four measurements.

(0.2 N) with varied pH values for the ozone analysis, three different levels of ozone concentrations were obtained (Fig. 2). The ratios between any two levels were practically the same for higher concentrations but the limits of the central 'neutral' range widened by about 1.5 pH units at both ends of the range at 10,000 mg/m³.

The iodine content of neutral potassium iodide solution, liberated by low ozone concentrations, did not increase significantly within 1.5 hr at room temperature.

When ozone concentrations up to 4272 mg/m³ were diluted with pure air in two steps to obtain an ozone-air mixture with an ozone concentration of about 1.2 mg/m³ dilution ratios and ratios of ozone concentrations were not statistically different. No significant amount of ozone was lost between sampling points when the rate of flow was the same in injection and mixing tube.

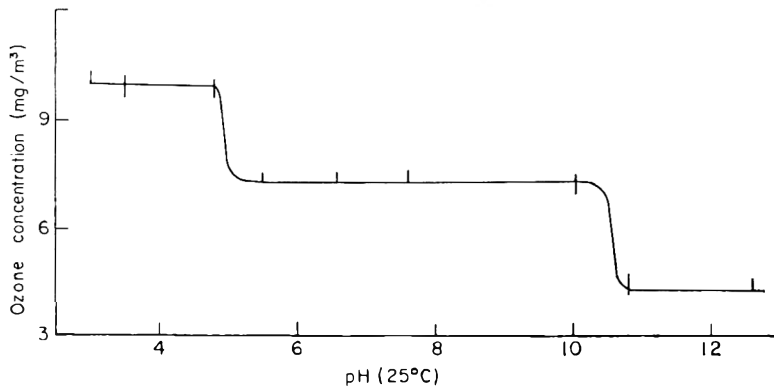


FIG. 2. Concentration of ozone in an ozone-air stream of constant composition as estimated iodometrically in buffers of varying pH and 0.2 N with respect to potassium iodide.

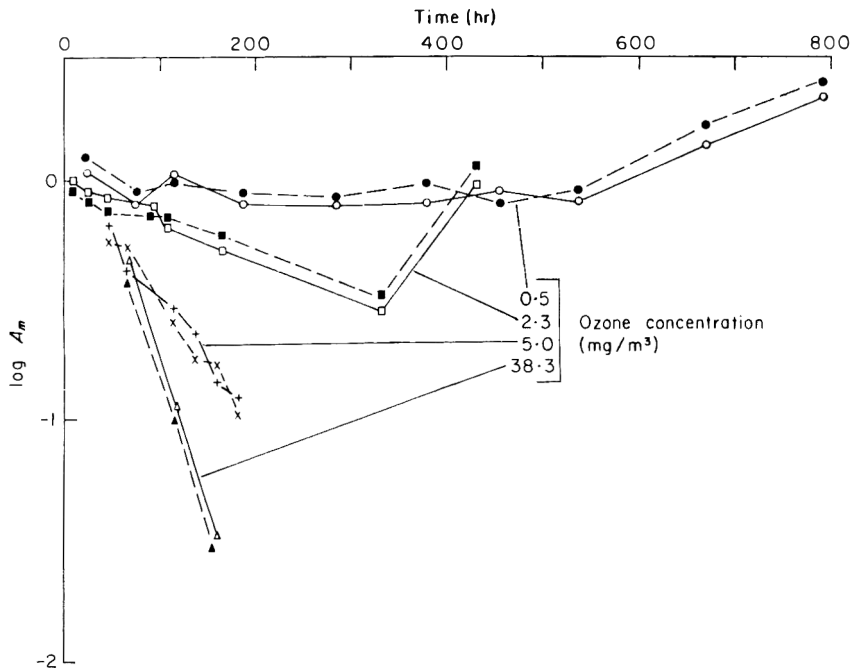


FIG. 3. Logarithm (base 10) of specific ozone uptake (A_m) of muscle as function of storage time, for various ozone concentrations. Muscle slices were populated with *Pseudomonas* species (ozone concentration 0.5, 2.3 and 38.3 mg/m³) or with moulds (ozone concentration 5.0 mg/m³).

Interaction of ozone with muscle

The average concentration of ozone entering the reaction space containing two meat slices was, at the beginning of an experiment, 1.46 times the concentration close to the muscle. After the reaction the ozone concentration seemed to equilibrate quickly: concentrations near the samples were about the same as those close to the wall of the container.

(1) Effect of time on rate of ozone uptake

The approximately linear decline with time of the logarithm of specific ozone consumption by the muscle (A_m) was very small for the low concentration of 0.5 mg/m³; it increased with ozone concentration up to 5 mg/m³, but a further increase in concentration had less effect (Fig. 3). Curves obtained for samples populated with micro-organisms remained in the range of scatter of curves for sterile meat, except when bacterial volatiles were evolved. At ozone concentrations ≤ 0.6 mg/m³, A_m then increased again to values higher than the initial values.

In fifty-eight experiments, an initial value $A_{m_0} = 1.02 \pm 0.23$ was obtained from the regression (S) of $\log^{10} A_m$ on time, for all ozone concentrations used, independent of the presence or absence of micro-organisms ($F = 0.73$, N.S.-D.F.: 1,56). Reductions of EH from 99.3 to 98.0% and the presence of 11% carbon dioxide in the atmosphere had no influence on A_{m_0} .

Significant S values were obtained for ozone concentrations of 1.8 mg/m³ and above ($P < 0.01$). With ozone concentrations of 0.7 mg/m³ and lower, the regression S was not different from zero when EH was 98.5 or higher, but a significant regression was obtained for EH 98.0% ($P < 0.02$).

(2) Effect of tissue on rate of ozone uptake

A considerably lower specific rate of ozone consumption was determined for high fat surface tissue ($A_{m_0} = 0.31$) and for lean surface tissue ($A_{m_0} \times 0.44$) taken from the flank. Regression values S were smaller than for muscle tissue, but not significantly different (fiducial limits are shown in Fig. 4).

(3) Effect of ozone concentration on ozone consumption

The slopes S as a function of the ozone concentrations are plotted on a log-log basis in Fig. 4. A significant regression ($F = 227^{xx} - D_f: 1.48$) for ozone concentrations between 0.15 and 5.4 mg/m³, omitting values for surface tissue and for muscle tissue with EH 98.0%. In this range the plotted values fit the regression line reasonably well. The regression line can be written $S = -4.23 \times 10^{-4} [O_3]^{1.54}$, where the power is the absolute value of the regression of $\log^{10} S$ on $\log^{10} [O_3]$.

The logarithm of A_m varies approximately linearly with time thus $A_m = A_{m_0} e^{-\alpha t}$. For ozone concentrations up to 5 mg/m³, α (hr⁻¹) is given by:

$$\alpha = 2.303S = -9.74 \times 10^{-4} [O_3]^{1.54}.$$

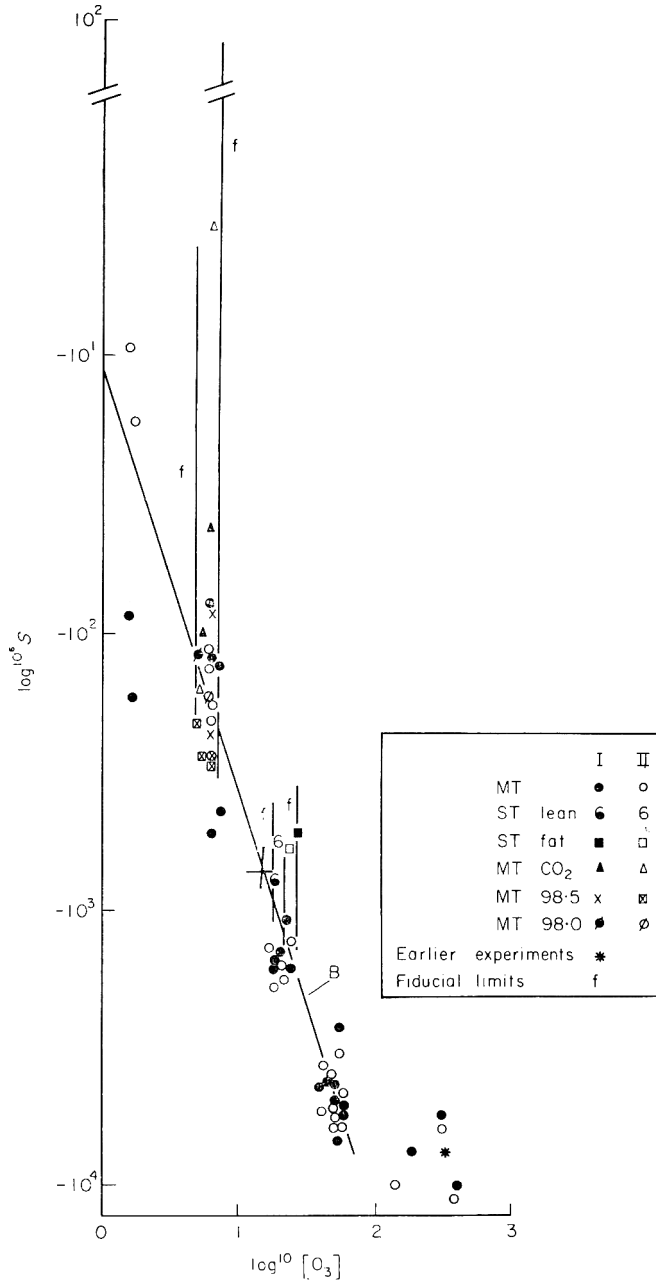


FIG. 4. \log^{10} of slope S of \log^{10} of specific rate of ozone decomposition (A_m)-time curves as a function of \log^{10} of ozone concentration $[O_3]$. Regression line B of $\log^{10} S$ on $\log^{10} [O_3]$ for ozone concentrations ≤ 5 mg/m³ using S -values for muscle slices, except those in equilibrium with EH 98.0%. MT, Muscle tissue; ST, surface tissue in containers I and II.

Discussion

(1) *Estimation of low ozone concentrations*

It was shown that the rate of ozone delivery was statistically not different in air-ozone mixtures with high ozone concentrations (up to 4200 mg/m³) entering the mixing tube and in gas mixtures leaving the tube after dilutions up to 3500 times with pure air. According to Salzman & Gilbert (1959) iodine equivalents changed in the same ratio as the dilutions for ozone concentrations between 2 and 80 ppm and nitrogen dioxide equivalents were equal to iodine equivalents when ozone (1–20 ppm) reacted with neutral potassium iodide buffered with phosphate.

It seems probable that stoichiometrically correct ozone estimations are obtained when gas mixtures with ozone concentrations of the order of 1 mg/m³ are analysed.

(2) *The specific rate of ozone decomposition*

The ozone consumption per unit area of fatty surface tissue was considerably smaller than that of muscle tissue. When ozonizing quarters of beef on which the surface of muscle unprotected by connective and adipose tissue amounts only to about 10% of the total surface, the ozone consumption is predominantly determined by the specific rate of decomposition of the surface tissue.

The velocity constant α for the decline of A_m with time plotted as a function of the ozone concentration on a log-log basis showed a linear course for concentrations up to 5 mg/m³. Beyond this concentration α followed an approximately hyperbolic course with increasing ozone levels. An increasing α seemed to indicate increased oxidative changes of the muscle surface, and the appearance in the meat pigments of brown components was a visible indication of these changes. Histological sections (Kaess & Weidemann, 1968) of muscle exposed to 5 mg/m³ ozone showed a thin, intensely stained surface layer, most probably due to oxidation effects. This oxidized layer had an inhibitory effect on microbial growth in storage periods where A_m was strongly diminished. The oxidized layer was absent in tissue exposed to ozone concentrations ≤ 2 mg/m³. The inhibitory effects of ozone at a concentration of 0.6 mg/m³ cannot, therefore, be due to an oxidized layer and appear to be associated with the uptake of ozone at a constant and high specific rate.

Acknowledgments

Mr A. Howard's programming and processing of statistical data with the computer and the technical assistance of Mr B. Mee and Mr R. Turner are gratefully acknowledged.

References

- BIRDSALL, C.M., JENKINS, A.C. & SPADINGER, E. (1952) *Analyt. Chem.* **24**, 662.
BOELTER, E.D., PUTNAM, G.L. & LASH, E.I. (1950) *Analyt. Chem.* **22**, 1533.
BOWER, V.E. & BATES, R.G. (1955), *J. Res. natn. Bur. Stand.* **55**, 197.
BRADBURY, J.H. & HAMBLY, A.N. (1952) *Aust. J. scient. Res. A.* **5**, 541.
FINNIE, T.M. & YALLOP, H. J. (1957) *Analyst, Lond.* **82**, 653.
KAESS, G. (1956) *Aust. J. appl. Sci.* **7**, 242.
KAESS, G. & WEIDEMANN, J.F. (1962) *J. appl. Bact.* **25**, 180.
KAESS, G. & WEIDEMANN, J.F. (1968) *J. Fd. Technol.* **3**, 325.
LINDSEY, A.J. (1952) *J. Phys. Chem., Wash.* **56**, 439.
SALTZMAN, B.E. (1954) *Analyt. Chem.* **26**, 1949.
SALTZMAN, B.E. & GILBERT, N. (1959) *Analyt. Chem.* **31**, 1914.

A method for collection and concentration of fish volatiles

J. N. KEAY AND A. S. MCGILL

Summary. A method is described for the collection of volatiles from fish by high vacuum distillation and the subsequent concentration of the frozen aqueous distillates by zone refining. Concentrations of up to 35-fold have been obtained. The enhanced GLC profiles thus derived greatly facilitate analysis of the mixtures. The method employs mild conditions which minimize loss or alteration and is particularly suited to the analysis of numbers of samples.

Introduction

The collection of traces of complex mixtures of volatiles from foodstuffs without loss or chemical alteration of labile constituents and in sufficiently high concentration to permit complete analysis (e.g. by GLC) is a major problem in flavour chemistry. Methods described for achieving this include sampling of head-space vapour, vacuum distillation and extraction with low boiling solvents (Mendelsohn, Steinberg & Merritt, 1966; Lea, Swoboda & Hobson-Frohock, 1967; Wick, Underriner & Paneras, 1967). The concentration of a solvent extract by zone refining minimizes flavour loss and thermal decomposition and this process has been successfully exploited by Huckle (1966) for the analysis of raspberry flavours. An even more attractive proposition is the direct zone refining of aqueous solutions of volatiles and the technology of this process has been studied by Swoboda (1967).

The present communication describes a simple high vacuum distillation technique for the extraction of fish volatiles and their concentration by zone refining of the frozen aqueous distillate.

Apparatus, materials and methods

Distillation vessel

This is shown in Fig. 1. It consists of a two-limbed glass vessel; the limbs A and B are fitted with 24/29 and 14/23 standard sockets, respectively. The vessel shown is of a size used routinely for distilling 50 g of fish. Other sizes have been used to distil up to 200 g.

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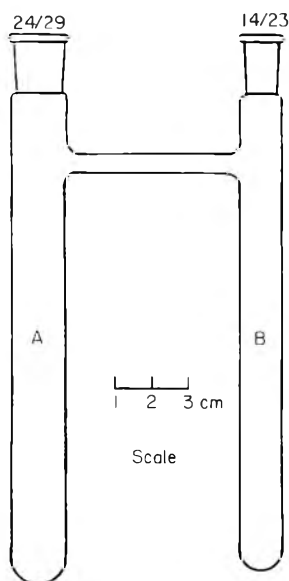


FIG. 1. High vacuum distillation vessel.

High vacuum system

This consisted of an all-glass manifold exhausted by a mechanical backing pump and silicone oil diffusion pump connected in series. Liquid air traps were fitted at suitable points to prevent contamination of the pumps with vapours from the system and conversely to protect experimental systems from contamination with pump oil vapour.

Vacuum distillation of f. h

The fish muscle (50 g) was cut into small pieces with a pair of scissors and placed in limb A of the distillation vessel. Mincing the fish, which was the method adopted in early experiments, is to be avoided since the fine mince conglomerates and impedes the devolatilization of material in the bottom of the limb. The fish was frozen by immersion of limb A in liquid air contained in a wide-neck Dewar flask. The limb was then stoppered, limb B immersed in liquid air and the vessel attached to the vacuum manifold by the 14/23 socket. With both limbs still refrigerated by immersion in liquid air the stopcock on the manifold was opened and the vessel evacuated to the required pressure (< 0.001 torr). *The stopcock was then closed.* Limb A and its contents were thawed; this was assisted by cautious immersion of the limb in a tall beaker of warm water ($< 40^{\circ}\text{C}$). When thawing was complete and bubbling had subsided limb A was again frozen, the vessel re-evacuated and the thawing process repeated. Several such cycles had the effect of removing entrapped air from the fish, thus preventing excessive frothing during the distillation. The vessel was then finally evacuated

with both limbs frozen, the stopcock closed and limb A immersed in warm water as before. Distillation was allowed to continue for several hours (conveniently overnight). By using a Dewar flask which fitted neatly around limb B, the need for replenishment of liquid air during the period of distillation was avoided. When distillation was complete (the dry appearance of the fish and the volume of water collected were guides to this, 50 g of fish providing approximately 40 ml water) the vessel was removed from the manifold and the distillate transferred to a flask for frozen storage.

The efficiency of the distillation procedure was checked by distillation of dilute aqueous solutions of organic compounds of a wide range of volatility. These included water (50 ml) containing a mixture of trimethylamine and n-butanol (0.001% of each) and water (50 ml) saturated with n-decanol. Chromatographic analysis of the original aqueous solutions and the distillates (the conditions were identical to those described later under 'Analysis of concentrates' p. 348) showed that complete recovery of the solutes was obtained in each case.

In order to eliminate the possibility of contamination of distillates with materials originating in the distillation system (e.g. volatiles from high vacuum grease), distilled water (50 ml) was re-distilled in the system. Gas chromatographic analysis of the original distilled water and the re-distilled sample was carried out as before. The chromatograms displayed minor peaks due to traces of volatiles present in the water. The profiles of both samples were, however, identical indicating the absence of contamination from the distillation system. Further, both samples of water were subjected to zone refining as described later and the concentrates obtained were analysed by GLC. The profiles were once again identical thus reinforcing the evidence against contamination.

Zone refining of frozen distillates

The zone refiner used was a commercial apparatus, the 'BTL Multi Zone Refiner' marketed by Baird and Tatlock (London). The apparatus is equipped with five zone heaters mounted vertically in series, three speeds of travel and continuously variable temperature control. The movement of the zones is from top to bottom, the impurities (in this case the volatiles) thus being located at the bottom of the tube.

In order to effect zone refining of frozen aqueous distillates a number of simple modifications had to be carried out and departures from conventional technique adopted. The apparatus was operated in a cold room at -20°C . An ambient temperature of -30°C used in early experiments frequently resulted in tube fracture after one pass. In the absence of such cold room facilities the apparatus can be operated in a refrigerated cabinet. In order to obtain zones of uniform length, convection currents in the apparatus had to be minimized by sealing round the window and the space at the bottom of the cabinet with adhesive PVC tape. Best results were obtained using a tapered tube but not of the type supplied by the manufacturer where the taper is sufficiently remote from the lower heater to prevent melting. The tube used is shown

in Fig. 2. By providing an internal taper but preserving the external diameter of the tube, melting at the lower heater is effective. The tube was conveniently filled with distillate using a hypodermic syringe with a needle sufficiently long to reach the bottom of the taper. In order to prevent fracture of the filled tube when first inserting it into the apparatus it was necessary to freeze the contents by fairly rapid immersion of the tube in liquid air contained in a tall Dewar flask. The opaque ice thus obtained also provided a suitable contrast with the molten zones which aided heater adjustment at the beginning of a run. The precise setting of the heater control required varied from instrument to instrument but in general lay between 3 and 4. The maximum speed of travel was used, i.e. 2.5 cm/hr or one cycle every 1.6 hr. After a few passes the entire contents of the tube, molten zones and ice, became transparent but it was noticed that a slight cloudiness appeared to correspond with the position of concentrated solutes. The passage of this cloudiness to the bottom of the tube was used as a guide to the process of concentration. Forty-eight to 72 hr zoning was normally required to send this cloudiness to the bottom. A run was terminated when the tube was at the lowest point of travel and, thus, when the contents of the tip were solid. A short length near the extremity of the taper was then cut off and placed in a stoppered tube for frozen storage pending GLC analysis.

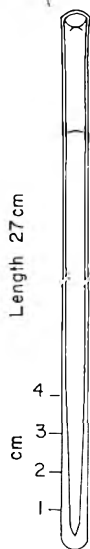


FIG. 2. Zone refining tube.

Analysis of concentrates

(a) Solutions of volatile materials were analysed by gas chromatography of 5- μ l samples in a Perkin Elmer F.11 chromatograph equipped with a Carbowax 1540

column (8% on Chromosorb W) of length 2 m and internal diameter 2.2 mm using a flame ionization detector. Quantitation was made by peak area measurement.

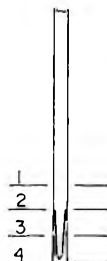
(b) Evan's Blue dye (British Drug Houses), molecular weight approximately 960, was used in one experiment to monitor the progress of zone melting. Concentration of the dye solutions was determined by measurement of absorbance at 600 m μ .

Results and discussion

(1) In order to test the behaviour of typical individual fish volatile substances in the system, dilute aqueous solutions (0.001%) of acetone and trimethylamine were zone refined for 72 hr. The concentrations of the solutions so obtained were compared with those of the original as determined by GLC. In a straight zone refining tube concentrations of ten-fold were obtained for both substances. In the tapered tube described above, acetone and trimethylamine were concentrated twenty and 28-fold, respectively.

(2) Water containing acetone (0.001%) and Evan's Blue (0.001%) was zone refined. The presence of the dye provided a means of monitoring the progress of zone refining and also of comparing the behaviour of two substances of widely different molecular weight in the system. After 72 hr, concentration of the dye in the lower part of the tube was almost complete although a slight blue tinge persisted at the lower end of each of the liquid zones. Continued refining for 7 days did not markedly improve this situation. The lower part of the tube was cut into sections, the concentration of dye in each determined spectrophotometrically and the acetone concentration determined by GLC. The volume of liquid contained in each section was also measured. The results are shown in Table 1. Acetone and dye concentration correspond, indicating that in this system there was no preferential concentration on the basis of molecular weight. Calculations based on the figures in the table, the concentration of the original solution and the total volume of the tube (3.3 ml) indicate total recovery of solutes in the sections cut.

TABLE I



Fraction	Volume (ml)	Acetone concentration factor (times)	Dye concentration factor (times)
1	0.230	1.25	1.3
2	0.250	2.30	2.3
3	0.140	11.0	10.8
4	0.035	25.8	25.0

(3) Distillates from a number of species of fish were zone refined and the GLC profiles of the concentrates, together with profiles for the original solutions are shown in Fig. 3. The results show that the extent of concentration varied from run to run, ranging from about ten-fold to over 35-fold. This appears to be associated with the length of the tapered portion which is cut for the sample. The higher concentrations were obtained when no more than 2 cm was cut. More intractable is the disparity which sometimes exists in the degree of concentration of different compounds in a concentrate. The dilutions are such that ideal behaviour would have been expected from the solutes and the acetone-dye experiment bears this out.

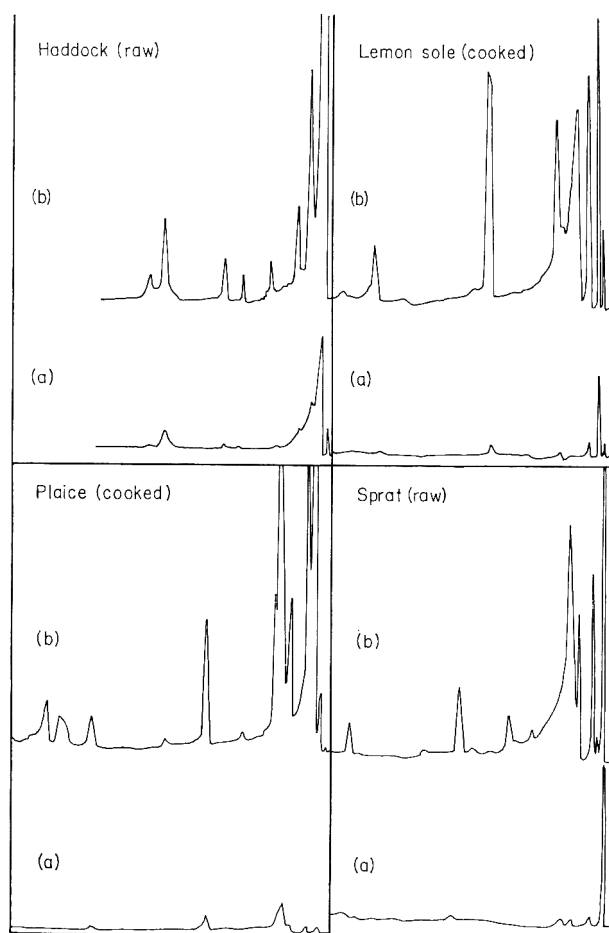


FIG. 3. GLC profiles of distillates from various species of fish, (a) before and (b) after zone refining (5- μ l injections on Carbowax 1540 column, temperature programmed 30–125°C). All the analyses were carried out at the same detector sensitivity ('Range' 20×1).

Conclusions

The high vacuum distillation procedure effects almost quantitative recovery of water from fish tissue. Since low temperatures and few manipulations are employed, recovery of volatiles without alteration may also be expected to be good. Zone melting of the aqueous distillate effects concentration of volatiles up to 35-fold which greatly facilitates their complete analysis by GLC. The volumes of solution so far used for zone refining have been small (3.3 ml). The construction of an apparatus to deal with even larger volumes and the repeated zone refining of concentrates so obtained is an attractive proposition for future work.

Acknowledgments

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References

- HUCKLE, M.T. (1966) *Chem. Ind.* 1490.
LEA, C.H., SWOBODA, P.A.T. & HOBSON-FROHOCK, A. (1967) *J. Sci. Fd Agric.* **18**, 245.
MENDELSON, J.M., STEINBERG, M.A. & MERRITT, C. (1966). *J. Fd Sci.* **31**, 389.
SWOBODA, P.A.T. (1967) Gas Chromatography 1966, *Proceedings of the Sixth International Symposium on Gas Chromatography and Associated Techniques*, p. 344, Institute of Petroleum London.
WICK, E.L., UNDERRINER, E. & PANERAS, E. (1967) *J. Fd Sci.* **32**, 365.

A comparison of the 'glass' electrode and flame photometric methods for determining sodium in food materials

A. G. CAMERON AND R. A. DELANEY

Summary. The 'glass' electrode technique and its application to environments likely to be encountered in food analysis is described. It is shown that the technique offers a rapid method of determining sodium concentration in the range 1 ppm to 2% in samples containing little or no protein. The 'glass' electrode and flame photometer methods, as carried out with the types of instrument likely to be available in control laboratories, are shown to be of comparable accuracy. Results obtained with the two techniques agree to 1–3%. The 'glass' electrode is shown to have certain advantages over the flame photometric technique, notably its freedom from interference by solid matter and chemical substances, resulting in simple and rapid sampling and calibrating procedures.

Introduction

The flame photometric technique for the measurement of sodium in foods is well established and results for a wide range of foods have been published by Bills *et al.* (1949) among others. The method, nevertheless, suffers from certain limitations, particularly if a simple filter instrument is used. Although such instruments are easy and rapid to use for the measurement of sodium, they are subject to interference effects due to the presence of other ions and organic food constituents in the sample. Sample preparation may be somewhat lengthy and involves the removal of any solid matter and interfering substances; the latter requirement may necessitate ashing the sample. A careful calibration procedure over the range of sodium concentration to be measured is also required as the calibration plot is non-linear except at very low sodium levels.

The increasing use of low sodium foods has created a need for rapid routine methods of determining sodium and in view of the limitations of the flame photometric technique it is of interest to compare it with the newer glass electrode technique. Much effort has been expended in developing glass electrodes which respond to specific metal ions in the same way that conventional glass electrodes respond to hydrogen ions. Eisenman, Rudin & Casby (1957) in the U.S.A., and Mattock (1962) in the U.K., have developed sodium-ion responsive glass electrodes and such electrodes are now commercially available.

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The rapidity and simple nature of the technique, together with the extreme selectivity of modern electrodes, offers certain advantages over flame photometric procedures, and has resulted in their application to a wide range of analytical problems. Nevertheless insufficient is known about the effect of environment on the performance of sodium-responsive electrodes to make it possible to apply the technique directly to foods. Only one such application has been reported—in a paper by Halliday & Wood (1966)—and this represents a special case in which the salt content of bacon was measured by direct contact of the electrode with the solid sample. In these circumstances environment proved unimportant.

The present work was undertaken to assess the conditions necessary for the application of sodium-responsive glass electrodes to the measurement of sodium in foods and to compare the method with the flame photometer technique.

Sodium-ion responsive glass electrodes, referred to in what follows as 'glass' electrodes, respond to cations in a way which is determined by the concentration and size and valency of the cations in relation to the composition of glass. The response of the 'glass' electrode was, therefore, measured under different conditions of pH, in the presence of cations likely to affect electrode behaviour, particularly potassium, and also in the presence of common organic food constituents. The technique was then applied to a selected range of simulated foods and the results obtained were compared with those from flame photometer measurements.

The potential E of a 'glass' electrode varies with sodium ion¹ activity A_{Na} according to the equation:

$$E = \text{constant} + 2.303 \frac{RT}{F} \log_{10} A_{\text{Na}},$$

where R is the universal gas constant, T the absolute temperature and F the Faraday. Provided that the activity coefficient of sodium ions f_{Na} is constant, the above equation may be re-written in terms of sodium ion concentration C_{Na} :

$$E = \text{constant}^1 + 2.303 \frac{RT}{F} \log_{10} C_{\text{Na}},$$

where $\text{constant}^1 = \text{constant} + 2.303 \frac{RT}{F} \log_{10} f_{\text{Na}}$.

Sodium ion concentration, by analogy with pH, may be expressed in terms of $p\text{Na}$ where

$$p\text{Na} = -\log_{10} C_{\text{Na}}.$$

Hence,

$$E = \text{constant}^1 - 2.303 \frac{RT}{F} p\text{Na}.$$

Provided that ionic strength is maintained constant by the addition of a relatively high concentration of a suitable buffer, measurement of the potential E of a 'glass' electrode measures C_{Na} (or $p\text{Na}$) in the same way that an ordinary glass electrode measures pH. This potential can be measured in terms of the EMF of a cell consisting of a 'glass' electrode, a reference calomel electrode and the solution under test, using a high impedance voltmeter (pH meter).

Materials and methods

An Electronic Instruments (E.I.L.) direct reading pH meter, model 23A, was used for $p\text{Na}$ measurement, the Δ pH scale of the meter, with a range of 7 pH (or 7 $p\text{Na}$) units, being used throughout. $p\text{Na}$ values were measured in the same way as pH values, an E.I.L. saturated calomel electrode, type GRJ 23, containing a potassium chloride salt bridge, being used in conjunction with an E.I.L. sodium-responsive electrode, type GEA 33, made from BH104 glass. The pH meter was standardized by setting the meter at 3 with the electrodes in a 10^{-3} M-sodium chloride solution ($p\text{Na} = 3.00$), at a temperature of $21 \pm 1^\circ\text{C}$.

Measurements with the 'glass' electrode were made in static conditions, though agitating the solution was found to produce no permanent displacement of the meter reading. The response time, i.e. time taken for the meter to reach an equilibrium value was 1–3 min. The technique of electrode usage produced a significant effect on response time. For example, if the electrode dried out or was washed with water between sample measurements, response time increased. Thus, provided that the concentration change between successive measurements was less than a factor of ten, the electrode was simply wiped with a dry tissue between readings. After a series of measurements, and during storage, the electrode was immersed in a solution of 10^{-1} M-sodium chloride.

The ionic strength of all solutions was maintained constant by the addition of 0.5 M-triethanolamine and the pH was adjusted to the required value by the addition of M-hydrochloric acid. pH values were measured with a Pye, model 79, pH meter. Solutions were prepared from analytical reagent grade materials, and all measurements were made at $21 \pm 1^\circ\text{C}$.

Flame Photometer measurements were made with an Evans Electro-selenium (E.E.L.) instrument using a coal gas-air flame and a gelatin sodium filter. The instrument was operated according to standard procedure, and calibrated using standard solutions containing analytical reagent grade sodium chloride.

Results and discussion

Effect of pH and interfering ions on 'glass' electrode response

Sodium response of the 'glass' electrode was measured at different pH values using

sodium chloride solutions in the range 10^{-1} – 10^{-6} M. The pH of each solution was adjusted to a specific value in the range 3.8–10.2 using triethanolamine plus hydrochloric acid. The optimum pH value for the measurement of pNa was found to be 10.2. At this pH the 'glass' electrode was found to give a theoretical response in the pNa range 0–4, that is within this range there is a linear relationship between electrode potential expressed as pNa and sodium ion concentration most conveniently expressed as $-\log C_{Na}$. This indicates that, provided the pH is maintained at about 10, sodium concentration may be accurately measured down to about 1 ppm corresponding to pNa 4.4. Provided that pNa values below 3.5 are to be measured, i.e. sodium concentration above 11 ppm, it is sufficient that pH be in the range 8–10. Thus the effective range of the 'glass' electrode is large being pNa 0–3.5, that is molar to 11 ppm sodium at pH 8, extended down to 1 ppm sodium at pH 10. These conclusions accord with those previously reported by Mattock (1962).

The effect of certain cations on 'glass' electrode response, particularly potassium, was investigated in view of the close association of sodium and potassium in foods. Mattock (1962) found that using BH 104 glass, potassium interferes if the potassium to sodium ratio is greater than 50. However, as the work using this glass was carried out at only one pH level and one potassium concentration, further work was done to determine the effect of varying these parameters. It was found that a 100-fold excess of potassium over sodium did not affect the response of the 'glass' electrode in the pNa region 0–4 at pH 10 at the 0.02 pNa unit level of precision achieved with the pH meter used, but that if the pH was reduced to 7, pNa measurements were only unaffected in pNa region 0–3. It was also found that a 100:1 ratio of potassium to sodium produced a zero shift of 0.1 pNa units. Further additions of potassium gave no further permanent shift.

Measurements of interference by ammonium and of mixtures of ammonium and potassium both produced results similar to those of potassium; namely that at the optimum pH of 10 a 100:1 ratio of potassium to sodium produced no change in the measured pNa value in the pNa range 0–4. Mattock (1962) obtained similar results with calcium and magnesium.

It may be concluded that pNa measurements at the 0.02 pNa level of precision and optimum pH value of 10 may be made in the 0–4 pNa region without interference from other cations provided that the interfering ion to sodium ratio does not exceed 100:1.

Effects of organic food constituents on electrode response

Moore & Wilson (1963) found that glucose, when added to urine in the range 2–6%, increased pNa response somewhat. No indication is given, however, of the pNa range studied nor of the level of discrimination at which this effect was observed. A more precise investigation of the effect of glucose on pNa response was, therefore, undertaken, and the effect of other soluble carbohydrates was also measured.

pNa response was measured in the presence of glucose, fructose and sucrose used

singly and also in mixtures. The results for sucrose are shown in Fig. 1, from which it

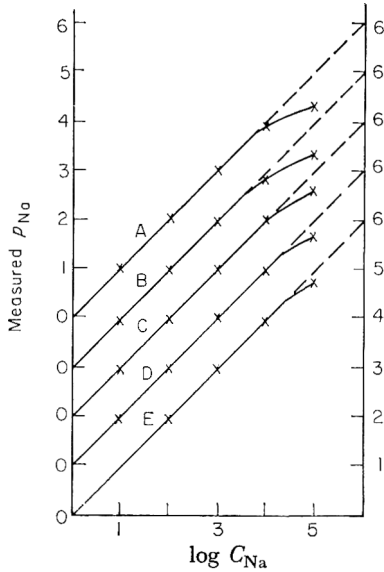


FIG. 1

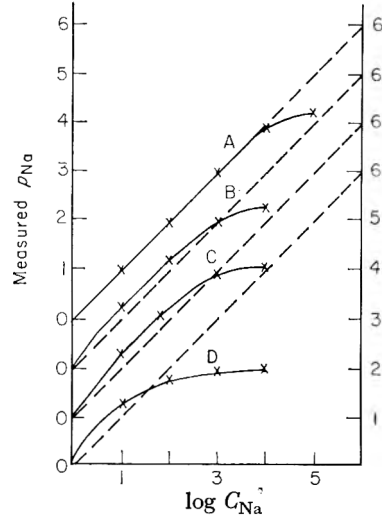


FIG. 2

FIG. 1. The effect of sucrose on sodium response. All solutions were made up in 0.5 M-triethanolamine and have pH 10.2. Theoretical response is shown by the broken diagonal lines, the curves being displaced vertically to aid comparison. Curves: A, 0.5 M-triethanolamine only; B, 1% w/v sucrose; C, 3% w/v sucrose; D, 6% w/v sucrose; E, 12% w/v sucrose.

FIG. 2. The effect of casein on sodium response. All solutions were made up in 0.5 M-triethanolamine and have pH 10.2. Curves: A, 0.5 M-triethanolamine only; B, 0.1% w/v casein; C, 0.5% w/v casein; D, 8% w/v casein.

can be seen that below pNa 4, sucrose in the range 1–12% does not affect 'glass' electrode response. At pNa 4 a sucrose concentration at or above 3% w/v increases the pNa reading by nearly 0.2 pNa units, and by about 0.4 pNa units at pNa 5 at the 0.02–0.05 unit level of discrimination. Similar results were obtained with glucose and fructose and with mixtures of these sugars.

In measuring the sodium content of urines Moore & Wilson (1963) report that the 'glass' electrode is unaffected by the presence of protein and they conclude that protein interference in general is negligible. In the present work, however, protein has been found to have a pronounced effect. Fig. 2 shows how 'glass' electrode response is affected by casein. Even at the low casein level of 0.1% w/v electrode response was non-linear over the pNa range 0–4, and as casein concentration was increased there was an increasing effect on pNa response. Eight per cent casein gave a measured pNa value of 1.9 at pNa 4. Approximately similar results were obtained using albumin and gelatin.

As the present study was concerned with developing a rapid direct method of sodium measurement, it was concluded that the use of the 'glass' electrode should be confined to food systems containing little or no protein.

'Glass' electrode results using simulated food systems

A growing interest in high calorie-low electrolyte-low protein drinks is reflected in their increasing commercial availability. Such foods are usually based on the use of liquid glucose and in the present study two such products A and B were prepared from demineralized liquid glucose. They both contain sodium benzoate as preservative and are protein-free fruit-flavoured products, A having a higher liquid glucose and lower sodium content than B. C is made from sucrose and artificial sweeteners and simulates a commercial fruit drink containing traces of protein.

The accuracy of the 'glass' electrode method was determined by measuring the sodium content of liquid glucose and of A, B and C before and after standard additions of sodium. The values obtained are shown in Table 1 which indicates recoveries in the

TABLE 1. Recovery of added sodium (expressed as ppm)

Sample	Sodium present	Sodium added	Sodium found: 'Glass' electrode	% recovery of added sodium	Sodium found: flame photometer	% recovery of added sodium	Difference between methods (% of total sodium)
Demineralized liquid glucose	39	200	238	99.5	236	98.5	0.84
With added sodium	239	100	334	95.0	339	100	1.5
With added sodium	339	10	349	100	350	110	0.29
A							
Alone	240	200	437	98.5	438	99.0	0.23
With added sodium	440	100	530	90.0	534	94.0	0.76
With added sodium	540	10	549	90.0	549	90.0	0.00
B							
Alone	480	200	670	95.0	671	95.5	0.15
With added sodium	680	100	772	92.0	782	102	1.3
With added sodium	780	10	790	100	789	90.0	0.12
C							
Alone	244	200	433	94.5	439	97.5	1.4
With added sodium	444	100	540	96.0	544	100	0.74
With added sodium	544	10	553	90.0	554	100	0.18

range 90–100%. The lower recovery values all apply to sodium concentrations above 500 ppm; below this value recoveries are in the range 95–100%.

Comparison of 'glass' electrode and flame photometer measurements

Mattock (1963), in summarizing the results of several workers, reports that in most cases there is good agreement between 'glass' electrode and flame photometer results for sodium concentration. Agreement of 1–5% is achieved in many cases and where significant differences arise this is usually attributable to failure to provide a constant ionic strength medium for 'glass' electrode measurements. In the present study, using standard sodium solutions, in the range 50–200 ppm sodium, and the optimum pH value of 10 for 'glass' electrode measurements, the difference between the methods was 1–3%. 'Glass' electrode results were 2–5% low and flame photometer results were 0–3% low.

In Table 2 the results of sodium measurements on liquid glucose and the simulated food systems A, B and C are compared. It can be seen that, as with pure solutions, the two methods differ by 1–3%, suggesting that interference effects are absent. The recovery of added sodium to the same systems as determined by the two methods is shown in Table 1. The correlation between the two sets of results is good, the difference between the two methods expressed as per cent of total sodium being in the range 0.0–1.5%.

TABLE 2. Sodium content of simulated food systems
(results given in ppm)

Sample	'Glass' electrode	Flame photometer	% difference
Demineralized liquid glucose	38	39	2.6
A	225	230	2.2
B	466	480	3.0
C	240	244	1.7

Conclusions

'Glass' electrodes allow rapid direct measurement of sodium concentration of food systems containing very little or no protein over the range pNa 0–4, corresponding to a sodium concentration range of 2% to 1 ppm. Over this range the 'glass' electrode shows the theoretical response and the accuracy achieved depends on the suitability of the pH meter used and on environmental conditions. pH should be maintained at or above 8 and ionic strength should be sufficiently high to swamp any variations in sodium activity coefficients resulting from the presence of other cations. The presence of cations other than sodium does not cause interference provided that the ratio of interfering ions to sodium does not exceed 100 : 1. In the low protein samples used organic food constituents did not cause interference.

At levels of pNa above 4 (less than 2 ppm sodium) electrode response curves deviate

from the theoretical and conditions should be more carefully controlled. In particular pH should be maintained at its optimum value of 10. At such high $p\text{Na}$ levels interference effects are increased and the accuracy of the technique reduced.

The pH meter used was of a type normally available in control laboratories and had a discrimination of 0.02 $p\text{Na}$ units. At this relatively low level of discrimination the corresponding sodium concentration discrimination in the $p\text{Na}$ range 0–4 was found to be 2–5% for pure solutions. In spite of the relatively low level of accuracy obtained, it is comparable with that obtainable from the type of simple flame photometer likely to be available in control laboratories. The difference between 'glass' electrode and flame photometer measurements of sodium concentration was in the range 1–3% for both pure solutions and simulated food systems.

In some circumstances 'glass' electrodes offer advantages over flame photometers. 'Glass' electrodes are highly selective, and consequently interference effects are less pronounced than with flame photometers. Potassium, calcium, sugars, proteins and organic solvents are some of the more important materials that interfere with flame photometer measurements. To avoid such interference ashing is frequently required, whereas with 'glass' electrodes sample preparation is simple except where protein interferes. Samples with a wide range of physical characteristics can be tolerated by 'glass' electrodes. Thus a variation in viscosity, which would affect the flame photometer reading, does not affect the 'glass' electrode. Suspensions, and other samples containing solid matter, can be used without the filtration required in the flame technique. The chemical inertness of 'glass' electrodes enables them to be used in most environments encountered in foods, and continuous monitoring is possible.

The technique of using 'glass' electrodes is simple and rapid. 'Glass' electrodes show a fairly rapid response; the BH 104 electrode reached equilibrium in 1–3 min, though this is inevitably slower than the few seconds required to make a flame photometer reading. Calibration of the pH scale in the $p\text{Na}$ range 0–4, in which electrode response is linear with concentration, is also rapid. In routine working it is sufficient to calibrate the scale with a single standard, whereas in flame photometry a complete calibration curve over the region to be used must normally be prepared. Moreover, the 'glass' electrode is sensitive to such a wide range of sodium concentration that the necessity of knowing the approximate sodium content of the sample before measurement, as required in the flame photometric technique, is avoided.

References

- BILLS, C.E., McDONALD, F.G., NIEDERMEIER, W. & SCHWARTZ, M.C. (1949) *J. Am. Dietet. Ass.* **25**, 304.
EISENMAN, G., RUDIN, D.O. & CASBY, J.U. (1957) *Science, N.Y.* **126**, 831.
HALLIDAY, J.H. & WOOD, F.W. (1966) *Analyst*, **91**, 802.
MATTOCK, G. (1962) *Analyst*, **87**, 930.
MATTOCK, G. (1963) *Analytical Chemistry 1962: Proceedings of the International Symposium held at Birmingham University*, 1962, p. 253. Elsevier, London.
MOORE, E.W. & WILSON, D.W. (1963) *J. clin. Invest.* **42**, 293.

The effect of storage prior to processing on the quality of canned herring

R. McLAY

Summary. Herring were kept in ice for up to 10 days and then canned. Total carbonyls were determined as a measure of rancidity before and after canning. The carbonyl content increased with storage prior to canning but did not show a corresponding increase after canning.

A taste panel could not distinguish between canned herring stored at various times in ice before canning.

Introduction

It has been stated (Hughes, 1963) that rancid fish lose their rancid flavour after canning. It was the purpose of the following experiment to test this statement. Hughes (1963) studied this phenomenon using volatile carbonyls as a measure of rancidity, however no taste panel assessments were carried out. The carbonyls measured by Hughes were trapped by a vacuum distillation procedure. This involved evacuating a slurry of herring for 4 hr at 40°C. Preliminary experiments showed that carbonyls were produced by this heating. The experiments described below used a 10% trichloroacetic acid extraction to avoid generation of carbonyls by further heating. It was found that this method extracted twice the amount of carbonyls than the distillation method produced but that their composition was similar.

Experimental

Storage and canning

Two boxes of fresh herring were stored in a chill room at 4°C and samples of fish were removed at intervals up to 10 days. The boxes contained a layer of ice on the bottom, the fish being laid over this and then covered with ice. Twenty-four fish were removed on each of the 1st, 3rd, 4th, 6th, 7th and 10th days for analyses and twelve of these were nobbed, brined for 20 min, placed in cans, 350 g to each, and steamed for 20 min with the lids clinched. The cans were immediately sealed and heated at 115°C for 70 min. The remaining twelve fish were nobbed, minced and the two 20-g samples taken and immediately analysed for carbonyls.

Six weeks after the last samples had been canned, random pairs of cans containing

herring stored for different periods in ice were compared for flavour and analysed for carbonyl content.

Extraction of carbonyls

Twenty grams of minced fish were homogenized with 50 ml of 10% trichloroacetic acid (TCA) and centrifuged at 3000 rev/min for 10 min. The supernatant liquid was removed and the extraction with 10% TCA was repeated twice on the residue. The three TCA extracts were combined, saturated with salt, and then filtered into 50 ml of 2 N-HCl saturated with 2,4-nitrophenyl hydrazine (DNPH). The resulting solution, after standing at room temperature for 24 hr, was extracted with four 50-ml portions of carbon tetrachloride and then with four 50-ml portions of benzene.

The two extracts were combined, washed with 2 N-HCl until the acid layer was colourless and then with distilled water until the aqueous layer contained no acid. The carbon tetrachloride-benzene extracts were filtered through anhydrous sodium sulphate and washed with benzene until colourless. The extract was then evaporated to dryness on a rotary evaporator and 3 ml of benzene was added to dissolve the DNPH derivatives.

Separation of mono- and dicarbonyls (Gaddis, El'is & Currie, 1959)

A $\frac{3}{4}$ -in. diameter column was packed to a height of 3 in. with 5% hydrated alumina. A layer $\frac{1}{4}$ in. thick of sodium sulphate was added to the top of the column. The DNPH derivatives in benzene were added to the column and the monocarbonyls were eluted with 500 ml benzene. The dicarbonyls were developed with ether and eluted with 250 ml absolute ethanol, leaving the excess reagent on the column. The absorbances of the mono- and dicarbonyl derivatives were measured at 430 and 480 m μ , respectively.

Each fraction was evaporated to dryness and dissolved in 2 ml benzene in readiness for further chromatographic separation.

Paper chromatography of mono- and dicarbonyls (Byske *et al.*, 1956)

Fifty microlitres of each sample was spotted on Whatman No. 3 MM chromatography papers. The papers were dipped in dimethyl formamide up to 1 in. from the spots and dried for 30 min in a current of warm air. The papers containing monocarbonyls were allowed to equilibrate for at least 8 hr in a sealed tank containing a layer of n-hexane saturated with dimethyl formamide at the bottom. The monocarbonyl derivatives were developed for 6 hr by a descending solution of n-hexane saturated with dimethyl formamide. When spectrophotometric determinations were required, the paper was run in a strip and the spots cut out and extracted with 3 ml EtOH.

Papers containing dicarbonyls were not allowed to equilibrate but were developed for 48 hr with n-hexane saturated with dimethyl formamide.

Taste acceptance of the canned herring

A panel of five members of staff was asked to assess the canned herring for palatability. Marks from 0 to 5 were scored according to the degree of rancid flavour present; 0 = very rancid, 5 = not rancid. Two different cans were tasted at a time, the nature of the cans being unknown to the tasters. As an independent check on the staff panel, the Torry Research Station taste panel assessed three different cans for palatability. Their marking system was: 4 = like very much, 3 = like moderately, 2 = no opinion, 1 = disliked. They were unaware of the nature of the experiments. The fresh herring were not tasted because after 24 hr storage they were too spoiled.

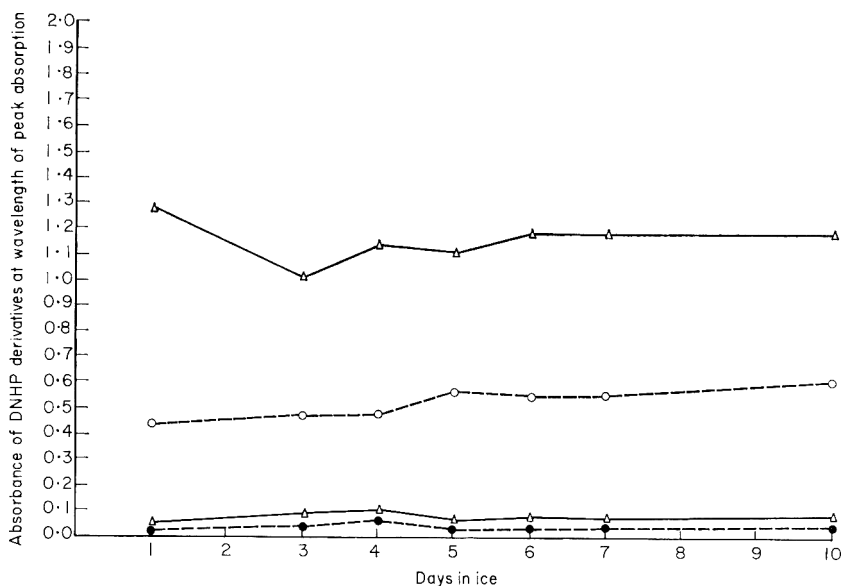


FIG. 1. Changes in carbonyl content of herring while being stored in ice and after canning and subsequent storage of cans for at least 6 weeks at ambient temperature. Δ , Canned herring monocarbonyls; \circ , raw herring monocarbonyls; \blacktriangle , canned herring dicarbonyls; \bullet , raw herring dicarbonyls.

Results

Carbonyls

Fig. 1 illustrated the changes in carbonyl content of the fish while it was being stored in ice and after canning and subsequent storage of the cans for at least 6 weeks at ambient temperature.

The monocarbonyl content of raw ageing fish increased by 30% during the 10 days storage, while the dicarbonyl content remained more or less constant. On canning, the monocarbonyls approximately trebled in amount and the dicarbonyls doubled in amount. Apart from the fish stored 1 day in ice, the carbonyl content of the herring

after canning appeared to be almost independent of the time of storage in ice prior to canning.

Chromatographic separation of the dicarbonyls was poor and provided no useful information.

Paper chromatography of the monocarbonyls gave a satisfactory separation of compounds according to molecular weight but not into classes. Eleven spots were obtained which corresponded to C₁ to C₁₁ monocarbonyls. Formaldehyde, acetaldehyde, acetone, propionaldehyde, iso-butyraldehyde and methyl-butyraldehyde were definitely identified. The remainder are yet to be identified. Since most spots contained more than one carbonyl it was not possible to quantify the results other than by a measurement of absorbance.

The relative concentrations of the monocarbonyl derivatives varies little with the time of storage of the fish in ice, but there was a striking difference between the monocarbonyl fractions before and after canning. Before canning C₄-C₁₁ carbonyls were present in the highest concentration but after canning, the C₃ carbonyls predominated.

Taste

The taste assessment of the canned herring by B.F.M.I.R.A. staff and Torry staff was as follows:

Days in ice	Average score	
	B.F.M.I.R.A.	Torry
1	3.5	2.2
3	4.2	
4	3.2	
5	3.8	2.7
6	4.5	
7	3.2	
10	3.5	3.0

The results of the B.F.M.I.R.A. panel, which was for rancidity, indicate that there was no significant difference between the samples. The results of the Torry panel show a very slight preference for fish stored 10 days in ice over fish stored for shorter periods, but the result is of doubtful significance.

Discussion

From the results it appears that the increase in carbonyl content when herring are stored from 1 to 10 days in ice is not accompanied by a corresponding increase when the fish are canned and subsequently stored for up to 6 weeks at ambient temperature. After the 5th day in ice the fish smelled very rancid, but this was not accompanied by any marked increase in water extractable carbonyls. The smell was in fact somewhat

suggestive of bacterial decomposition and after 10 days the fish broke up on handling and were regarded as rotten and unfit for use. After canning the amount of total carbonyls trebled and their composition changed, the canning process having removed most of the C₄-C₁₁ carbonyls, leaving a greatly increased amount of mainly C₃ carbonyls. The C₄-C₁₂ carbonyls have been associated with rancid flavours in herring so that the diminution in these might account for the improvement which apparently occurred when these fish were canned.

It was rather surprising that one taste panel should show a slight preference for fish canned after more than 5 days in ice. The herring canned after 10 days in ice was said to have a salt/meaty type of flavour somewhat similar to canned salmon.

Conclusions

From the information obtained it would appear that the limiting storage time in ice prior to canning depends mainly on the handling quality of the herring. The total carbonyl content increases with time of storage in raw fish, but remains constant in canned fish irrespective of storage time prior to processing.

Acknowledgments

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References

- BUYSKE, D.A., OWEN, H.L., WILDER, P. & HOBBS, M.E. (1956) *Analyt. Chem.* **28**, 910.
GADDIS, A.M., ELLIS, R. & CURRIE, G.T. (1959) *Fd Res.* **24**, 283.
HUGHES, R.B. (1963) *J. Sci. Fd Agric.* **14**, 893.

Effect of solutes and pH on the structure and firmness of cooked carrot

C. STERLING

Summary

Xylary segments of carrot root were cooked in solutions of different hydrogen-ion concentration (pH 3-8) and different solute composition. 'Firmness' was measured as resistance of the cooked segments to compression, and histological sections were prepared. The segments were softer, the higher the pH of the solution. This softening was attended by increasing cellular separation and eventual cellular collapse at higher values of pH. Firmness increased (over that in water) in sucrose solutions and solutions with divalent cations, and it decreased in solutions with monovalent cations. Explanations are considered for these effects and for the relatively minor increase in firmness in solutions of AlCl_3 .

Introduction

The firmness of cooked vegetables must ultimately be a function of the strength of the cell walls which compose their skeletal framework. The residual mechanical properties that survive the loss of turgor should then depend upon the structure and arrangement and the chemical composition of the cell walls. The classical use of 0.2 N concentrations of such salts as CaCl_2 and MgCl_2 (Personius & Sharp, 1939) illustrates the effect of solutes in strengthening the tissues of vegetables and fruits. The strengthening occurs presumably through cross-linking of molecules of pectinic acid by the introduced divalent cations to form a more rigid matrix in the middle lamella and primary cell wall (Kertesz, 1951; Matz, 1962; Sterling, 1963). Hence, the chlorides of monovalent cations have the opposite effect (Personius & Sharp, 1939).

However, results of other experiments with salts supported the possibility that the role of ions in cooking plant tissues might be more complex. With starch solutions (Morsi & Sterling, 1963) there are evidently lyotropic effects as well, and salts might cause a similar dispersal or coagulation of the polysaccharides of the cell wall, according to the polarizability and concentration of the ambient ions. For example, higher concentrations of CaCl_2 (to 1 M) generally resulted in greater solubility of starch than lower concentrations. The anomalously greater effect of (1 M) Na^+ over Mg^{2+} in firming of apple tissues at low temperature (Sterling, 1968) should also be noted.

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A testing of the role of the cation in chloride salts of similarly high concentration on structure and firmness in a cooked vegetable therefore was essayed. In addition, other types of solution served as controls: sucrose solutions for a non-ionic control of the concentration of the solute and various buffered solutions of different pH values for control of the hydrogen-ion concentration. Personius & Sharp (1939) had shown decrease in tensile strength in plant tissue as the pH of holding solutions of lactic acid was lowered from 4 to 2.5, and Doesburg (1961) had indicated also a decrease in firmness with a rise in pH, from pH 4 to 6.5.

Materials and methods

Imperator carrots were peeled, decorticated with a cork borer to produce cylinders of internal xylary tissue 1 cm in diameter, and cut into segments 1 cm long. The structure of the xylary region of the root has been described by Havis (1939) and Esau (1940). Essentially it may be envisioned as a mass of parenchymatous cells, in which are embedded small, widely scattered groups of tracheary elements, shown already in an earlier study (Sterling, 1955).

Fifty-five segments were added to 500 ml of a boiling solution and cooked 5 min after the solution again reached the boiling point. They were drained, placed in 500 ml of distilled water at room temperature, and then fifty were tested for firmness in the General Foods Texturometer (Friedman, Whitney & Szczesniak, 1963). Firmness was expressed as the maximum force required to compress the cylindrical segment from a height of 1 cm to 0.5 cm, expressed in arbitrary 'G.F. units' (Sterling, 1968). The five remaining segments were cut into thin disks, which were fixed in Randolph's fluid (Johansen, 1940) and then dehydrated with a series of graded solutions of polyethylene glycol 400, water and butanol. After being transferred to xylene, they were embedded in paraffin, cut, mounted on slides, and stained with methylene blue or Conant's quadruple stain (Conant, 1950).

The cooking solutions were made up as follows: nominal pH buffers, ranging from pH 3 to 8 by one-unit steps, were mixed from stock solutions of the McIlvaine buffer system (Hodgman, Weast & Selby, 1956), 20 ml of a buffer mixture being added to 480 ml of distilled water. The measured pH values after dilution were 3.04, 3.95, 4.97, 6.00, 7.00 and 8.00. Salt solutions of 1 M concentration were prepared from NaCl, KCl, CaCl₂, MgCl₂, ZnCl₂ and AlCl₃, with distilled water, and a 2 M solution of NaCl was also made up. Sucrose solutions were 0.5 M and 1.0 M. The pH values of these solutions are given in Table 2.

Results

The effects of different values of pH on firmness of cooked carrots are given in Table 1, which shows a progressive decrease in firmness as the pH increases from 3 to 8. A plateau seems to exist between pH 4 and 6. An analysis of variance shows that the variance ratio (F) is 9.8, as compared with a ratio of 3.1 for $F_{0.99}$. The probability

Firmness of cooked carrot

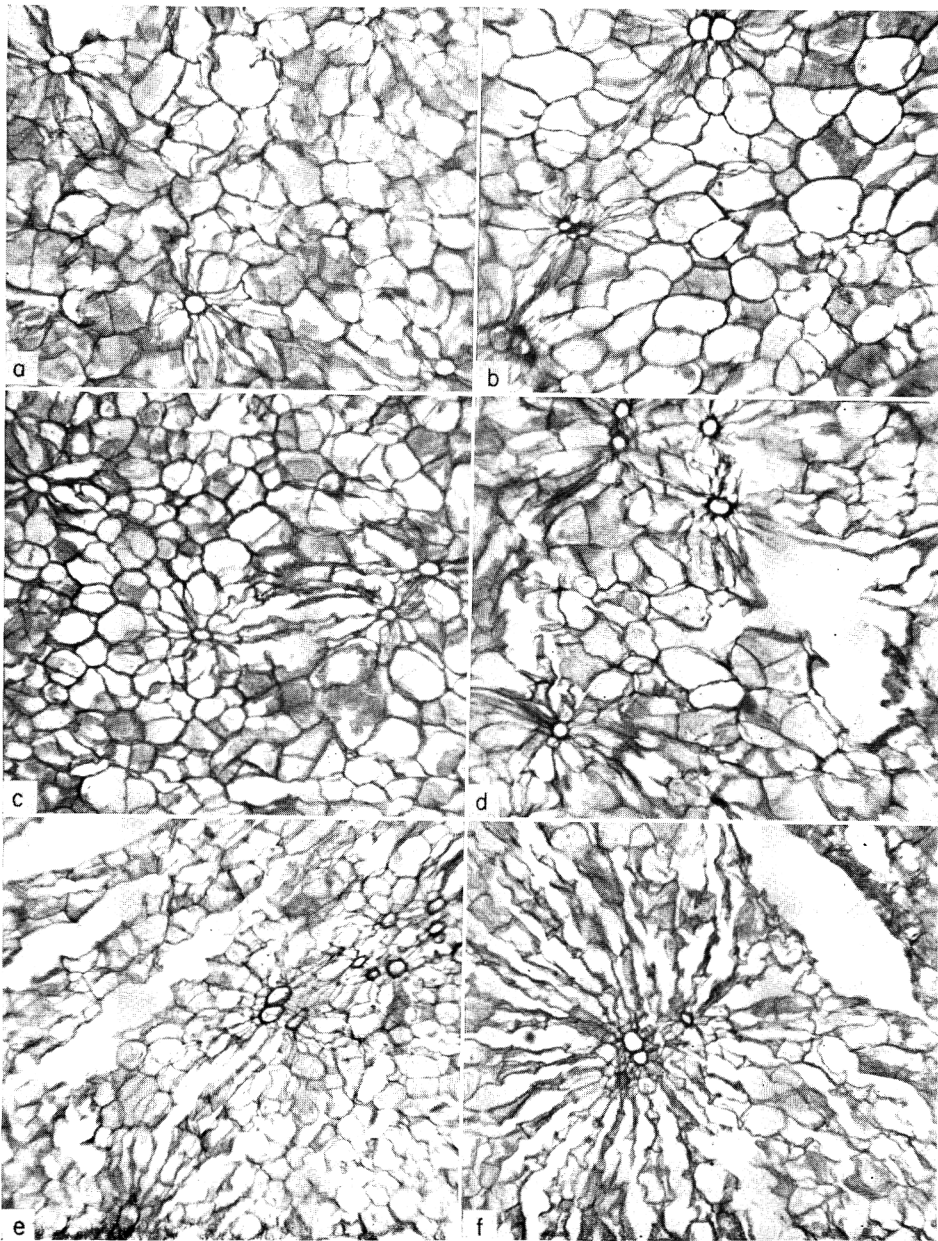


PLATE 1. Cross-sections of xylary region of carrot cooked in solutions of different pH values. Smaller, thick-walled cells from which larger, thin-walled parenchyma cells appear to radiate are tracheary elements. All figures $\times 61$. (a) pH 3; (b) pH 4; (c) pH 5; (d) pH 6; (e) pH 7; (f) pH 8. Note progressive separation and collapse of cells with increasing pH.

(Facing p. 368)

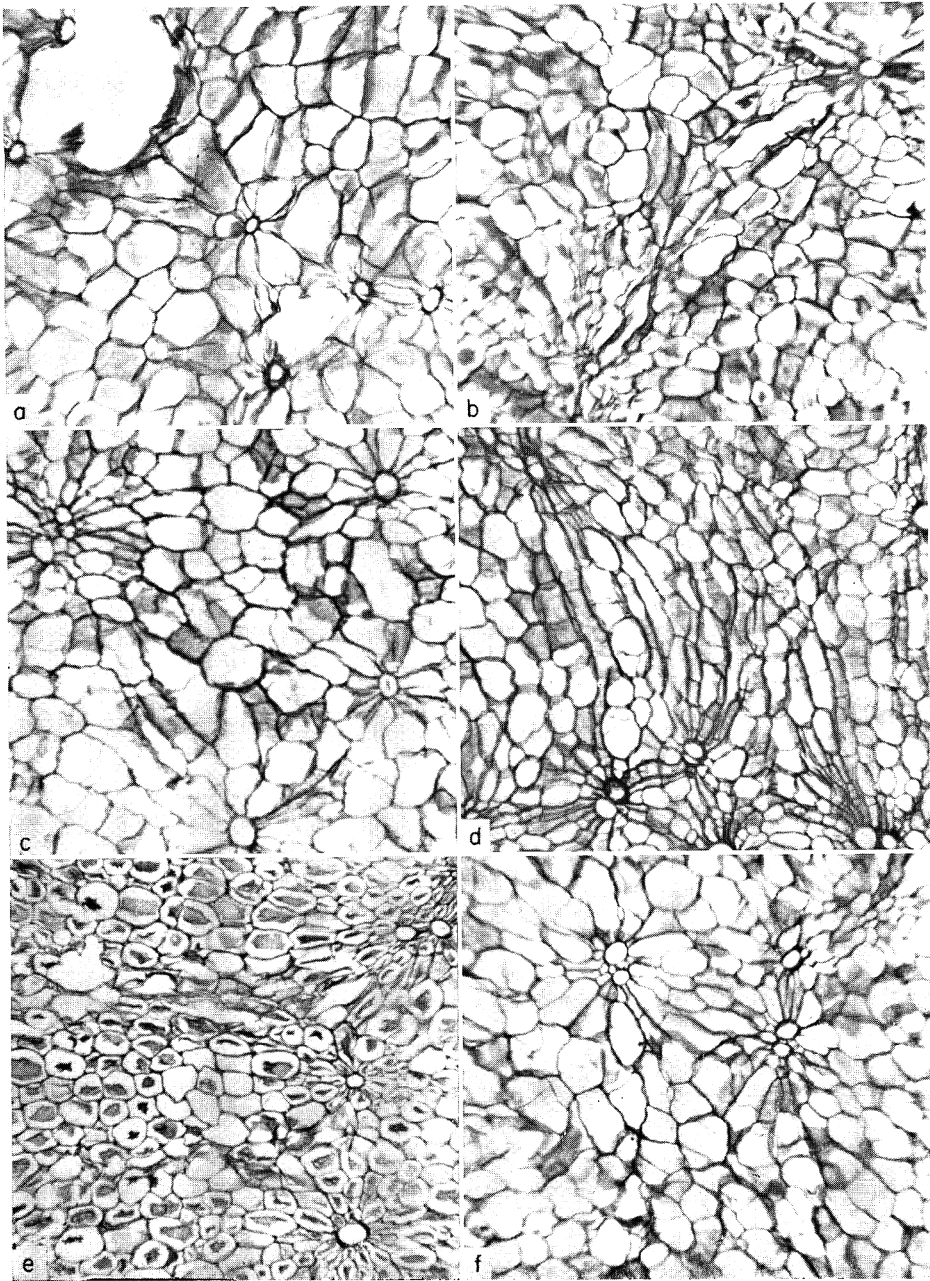


PLATE 2. Cross-sections of xylary region of carrot cooked in solutions of different composition. Smaller, thick-walled cells from which larger, thin-walled parenchyma cells appear to radiate are tracheary elements. All figures $\times 61$. (a) 2 M-NaCl; (b) 1 M-KCl; (c) 1 M-CaCl₂; (d) 1 M-ZnCl₂; (e) 1 M-AlCl₃; (f) 1 M-sucrose. For further explanation, see text.

TABLE 1. Firmness of cooked carrots: effect of pH

Nominal pH	Firmness (G.F. units)
3	50
4	43
5	39
6	43
7	30
8	27

that a variance ratio of 9.8 would be attained by chance is 0.0016 (Wallis & Roberts, 1956).

Plate 1 (a-f) gives a graphic portrayal of the histological relationships at the different hydrogen-ion concentrations. Note that at low pH values (Plate 1a and b) the cells are well-rounded and rather closely coherent, on the whole. Occasional regions of cellular separation, as at the upper left of Plate 1(a) will be found at pH 3 and 4. At pH 5 (Plate 1c), more cellular separation is evident, and from pH 6 (Plate 1d) to pH 8 (Plate 1f) the cells become quite isolated from one another. It is significant also that the higher the pH, the more the cells tend to collapse.

The firming relationship of different solutes in the cooking solution are presented in Table 2. Note that, except for the solution of AlCl_3 , the pH values generally range from 5 to 6 and hence are virtually equivalent among all the other solutes. If it be recalled that the average for firmness after cooking in water is 30, it may be seen that the solutions with monovalent cations tend to give somewhat lower values of firmness, whereas those with divalent cations give considerably higher values. With the trivalent cation, Al^{3+} , a lesser degree of firming is evident than with divalent cations. At the same molar concentration as most of the salts, sucrose produces a notable firming effect.

TABLE 2. Firmness of cooked carrots: effect of solutes

Solute	Concentration (M)	pH	Firmness (G.F. units)
NaCl	1	5.75	21
NaCl	2	5.47	28
KCl	1	5.20	21
CaCl_2	1	5.70	219
MgCl_2	1	6.15	204
ZnCl_2	1	5.20	184
AlCl_3	1	2.46	38
Sucrose	0.5	6.05	84
Sucrose	1	6.05	83

The histological picture following cooking in these different solutions is given in Plate 2 (a-f). Cellular separation is evident with NaCl (Plate 2a) and KCl (Plate 2b), whereas there is no indication of this effect with CaCl₂ (Plate 2c) or ZnCl₂ (Plate 2d). Some cellular separation occurs in AlCl₃ (Plate 2e), and a particular coagulation and shrinkage of the protoplasts of the cells is notable with this solute. With solutions of sucrose (Plate 2f), there is again a picture of relatively intact tissue.

Discussion

The results of the experiment with pH variations are by themselves consistent with two aspects of polysaccharide behaviour.

(1) With neutral polysaccharides, such as starch, the higher the pH, the greater the dispersibility and solubility of native grains and, under certain conditions, of starch paste (Fouard, 1907; Kalb & Sterling, 1962). This effect is indicated in the weakening of the whole cell wall, leading to its collapse.

(2) The higher the pH (above 3.0), the weaker is a hydrogen-bonded pectin gel (Kertesz, 1951). At pH values below 2.8 the pectin gel is again weakened, and a similar relationship may therefore be the case with the solution of AlCl₃. Possibly at the low pH value of this solution there is a hydrolysis of the polygalacturonide molecules.

If the firming of tissue in sucrose solution be regarded as due to an osmotic dehydration, the more than two-fold increase of firmness in the presence of divalent cations indicates that their role as salt-bridges between molecules of pectinic acid overrides their lyotropic role as dispersing (peptizing) agents. In water itself, then, the major links between the molecules of pectinic acid are hydrogen bonds, which can be more readily disrupted by heat.

The action of NaCl and KCl in further weakening the tissue, despite their expected osmotic role, can then be attributed to two effects: (1) the lyotropic effect of increasing the dispersion of the polysaccharide molecules by breaking hydrogen bonds (it being postulated that the pectic substances, as well as the other polysaccharides, are held together principally by hydrogen bonds); and (2) a small ion-exchange effect that might occur between monovalent ions from the salt solution and divalent ions already in the native pectic material (Mongar & Wassermann, 1952; Sterling, 1957; Thiele & Anderson, 1955). However, the methyl ester content of the pectic material of the raw carrot and the content of divalent cations are very low. These facts are indicated both by the pronounced firming due to the added divalent cations and by the low amount of water-soluble pectin in carrots (Buston & Kirkpatrick, 1931). It appears, therefore, that ion exchange is less important than the peptizing effect.

Acknowledgments

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References

- BUSTON, H.W. & KIRKPATRICK, H.F. (1931) *Ann. Bot.* **45**, 519.
- CONANT, G.H. (1950) *Triarch Catalog*, No. 8, p. 20. Triarch Botanical Products, Ripon, Wisconsin.
- DOESBURG, J.J. (1961) *Qual. Pl. Mat. Veg.* **8**, 115.
- ESAU, K. (1940) *Hilgardia*, **53**, 175.
- FOUARD, E. (1907) *C. r. hebd. Seanc. Acad. Sci., Paris*, **144**, 501, 1366.
- FRIEDMAN, H.H., WHITNEY, J.E. & SZCZESNIAK, A.S. (1963) *J. Fd Sci.* **28**, 390.
- HAVIS, L. (1939) *J. agric. Res.* **58**, 557.
- HODGMAN, C.D., WEAST, R.C. & SELBY, S.M. (1956) *Handbook of Chemistry and Physics*, p. 1615. Chemical Rubber Publishing Co., Cleveland, Ohio.
- JOHANSEN, D.A. (1940) *Plant Microtechnique*, p. 45. McGraw-Hill, New York.
- KALB, A.J. & STERLING, C. (1962) *J. appl. Polymer Sci.* **6**, 571.
- KERTESZ, Z.I. (1951) *The Pectic Substances*, pp. 194 and 266. Interscience, New York.
- MATZ, S.A. (1962) *Food Texture*, p. 109. Avi, Westport, Connecticut.
- MONGAR, J.L. & WASSERMANN, A. (1952) *J. chem. Soc.* 500.
- MORSI, M.K.S. & STERLING, C. (1963) *J. Polymer Sci.* **A1**, 3547.
- PERSONIUS, C.J. & SHARP, P.F. (1939) *Fd Res.* **4**, 299.
- STERLING, C. (1955) *Fd Res.* **20**, 474.
- STERLING, C. (1957) *Biochim. biophys. Acta*, **26**, 186.
- STERLING, C. (1963) *Recent Advanc. Fd Sci.* **3**, 259.
- STERLING, C. (1968) *J. Fd Sci.* (In press).
- THEILE, H. & ANDERSON, G. (1955) *Kolloid Zt.* **140**, 76.
- WALLIS, W.A. & ROBERTS, H.V. (1956) *Statistics: A New Approach*, p. 422. Free Press, Glencoe, Illinois.

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gram(s)	g	second(s)	sec
kilogram(s)	kg	cubic millimetre(s)	mm ³
milligram(s)		millimetre(s)	mm
(10 ⁻³ g)	mg	centimetre(s)	cm
microgram(s)		litre(s)	l
(10 ⁻⁶ g)	µg	millilitre(s)	ml
nanogram(s)		pound(s)	lb
(10 ⁻⁹ g)	ng	gallon(s)	gal
hour(s)	hr	milliequivalent	mEq
minute(s)	min	R _F values	R _F

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Symposium on Quality Control in the Food Industry

EDITED BY D. PEARSON

THIS special issue of the *Journal of Food Technology* contains the papers read at the *Symposium on Quality Control in the Food Industry* held on Wednesday and Thursday, 5th and 6th April 1967, at the Hoare Memorial Hall, Church House, Westminster, London, S.W.1. The symposium was arranged by the *Programmes Committee of the Institute of Food Science and Technology* and was organized by Dr S. M. Herschdoerfer. The contents of this special number were edited by D. Pearson (one of the Associate Editors) on the direction of the *Publications Committee*.

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Foreword

THIS special issue of the *Journal of Food Technology* is the first of what is hoped to be a series. From time to time, as topics of outstanding importance emerge in the food industry, we propose to organize similar symposia and issue the papers and discussions in one convenient publication. Such papers have a permanent value, and principles rarely change although methods are continually improving.

Our thanks are due chiefly to Dr S. M. Herschdoerfer for organizing this symposium, and he received considerable help from Mr T. McLachlan, Chairman of the Programme Committee and the members of this Committee. The Institute would like to record its gratitude to all who helped in this way. Finally we express our thanks to Mr D. Pearson for undertaking the very responsible task of editing this special issue.

J. G. DAVIS
Chairman, Publications Committee

Theme: What are the qualities we are concerned with?

1st Session. Chairman: LORD KINGS NORTON

Introduction

S. M. HERSCHDOERFER

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ON THE day I started to draft my contribution to this *Symposium* my desk diary showed the following motto: 'Far better to remain silent and be thought a fool than to speak and remove all doubts'. I now regret that I did not take this advice as I find myself in a rather difficult situation. All subsequent speakers are dealing with specific subjects on which I am sure they will speak with great authority. You will, however, have seen from the programme that my subject is quoted as 'Introduction' which is a rather vague term and can, of course, be interpreted in many ways. It is the privilege of the Chairmen to introduce the speakers and, therefore, all I wish to say on behalf of myself and all subsequent speakers is that while it is known which Companies or Organizations we serve any views expressed by us will be our own and will not necessarily coincide with the policies of our employers.

What I do consider to be the scope of my introductory talk is to say something about the reasons for holding this *Symposium*, to define some terms, such as quality and control, so as to avoid misunderstandings and, finally, I would like to devote a few minutes to the subject of standards and specifications. I trust that in doing this I shall not excessively encroach on the subjects of subsequent speakers.

Almost 10 years ago, in October 1957, our Vice President, Mr McLachlan, organized on behalf of the Food Group of the Society of Chemical Industry and the Association of Public Analysts a Symposium on *The Quality Control of Food* and in the course of the 2 days of the meeting a number of excellent and learned papers on quality control in various branches of the food industry were presented and discussed. One may, therefore, legitimately ask why it was considered desirable to organize again a Symposium to deal with apparently the same subject.

One might with some justification reply that 10 years is a long time, that in the meantime thousands of new people have entered the food industry and that anyhow a good thing may be worth repeating. There is, however, another answer to this question. The 1957 meeting addressed itself essentially to experts having a considerable background knowledge in the fields of food science and technology and the meeting discussed the application of such knowledge to quality control in the various branches of the food industry. This *Symposium* has been planned so as to take a broader view of the problems of quality control, not only from the angle of the manufacturer but also from the point of view of the distributor, the retailer and, of course, the consumer.

We shall, therefore, not deal with individual branches of the food industry but with problems which are common to most, if not all, types of the food business. In doing so we shall address ourselves not only to experts but also to the public in general and I trust we shall be excused if this requires the restatement of some facts which are common knowledge among many of those present.

I have already repeatedly used the term quality control and it is desirable that I should now define these words more closely. Everyone has some concept of what quality means but these concepts differ considerably. Professor John Hammond of Cambridge once stated that: 'the best definition of quality is that which the public like best'. I feel that this definition is too restrictive to be used in quality control. In fact I find it difficult to deal with quality in the singular and prefer to talk of qualities in the plural. Each food has a number of qualities or attributes—some positive or desirable, others negative, undesirable or even harmful—and it is the sum of all these different qualities which is implied by the general term of 'quality'.

Some qualities are obvious, being ascertainable to the consumer's senses such as taste, flavour, aroma and appearance, which includes shape, texture, colour and even packaging. These are the qualities by which the public judges our products and decides whether to purchase them or not. Such judgments are essentially subjective and different members of the public may express a favourable or unfavourable opinion on the same quality of the same product at the same time. The same cheese may be considered to have too strong a flavour or to be lacking in flavour depending on the actual or mental standard against which we compare it. A sausage may be considered to be too highly spiced or not spicy enough, again depending on personal preference. This implies that any statement on quality becomes meaningful only in relation to a standard and it is one of the main duties of a quality control organization to lay down, to formalize standards and to ensure that deviations from such standards do not exceed predetermined limits.

In addition to such obvious qualities as mentioned before we also have various 'hidden' qualities such as nutritional value, presence or absence of micro-organisms and compositional qualities, including the presence or absence of specified substances and also the keeping qualities. It is these hidden qualities which the public cannot easily assess, which are usually the subject of legislation or of other forms of regulation, but they form an essential part of a standard or to use a different term, of a specification for a food product.

I hope that I have now adequately explained what I mean by the words 'quality' or 'qualities' and I would like to turn now to the other term which we are concerned with, namely, *control*, especially quality control.

May I then draw your attention to the title of this *Symposium*. This is not the quality control of food but quality control in the food industry. The difference is obvious and important. The quality control of food goes back into prehistoric times. From the time man started using fire in the preparation of food he also started influencing, and that

means controlling, certain characteristics of this food. Similarly today's housewife controls the quality of the food she serves by a judicious selection of her raw materials, by using her skill in mixing them in a certain sequence under certain conditions, by subjecting them usually to some controlled heat treatment, by garnishing the food to improve its appearance and in some instances also by storing it or even packaging it. All these are operations roughly similar in kind to those carried out by the manufacturer of food products but the main difference lies in the aspects of quantity. The housewife is dealing with such small quantities that she can personally supervise and influence and thus control most, if not all, qualities of the food she is preparing. I deliberately said 'can' and not 'will' because the degree of her success in this respect will vary considerably according to her skill and the standard she will be setting herself.

The manufacturer of food is in a more difficult position. He may have an easier access to a wider market for his raw materials but he cannot apply the same methods of selection and full inspection as used by the housewife. The housewife can alter her processing conditions as she goes along for example by adjusting her mixing until the mixture has the desired consistency, thus allowing for the slightly different behaviour of different lots of raw materials. She can also watch the progress of her cooking or baking operation by bringing it to an end when the desired condition has been obtained. Such individually produced food will vary significantly from one lot to another but this does not appear to worry the housewife very much where her own cooking is concerned.

From the manufacturer, however, the housewife expects to be able to purchase a much more uniform product than the home produced one. She is also more cost conscious where purchased food is concerned. How many housewives could state the cost to themselves of, say, a Scotch Broth, including the cost of the gas or electricity used and even possibly the cost of their own time? They will, however, critically consider the relative values of similar soups manufactured by industry.

What I have been trying to convey is that the manufacturer in order to provide the housewife with a food of reliably uniform quality at a price she is willing to pay must organize his manufacture in such a way as to minimize all variable factors. He must attempt to standardize his raw materials, his processing methods, his storage and distribution system. Unfortunately, he then finds himself finally at the mercy of the retailer who through negligence or ignorance may undo much of the good work put in previously.

Every manufacturer of food products carries out certain quality control procedures but the organization and size of this operation varies enormously from industry to industry and even considerably within a given industry.

Industrial quality control has been gradually evolving in this century and has been more generally applied in the engineering and allied industries than in the food industry. This is mainly due to the difficulty of measuring objectively some

of the most important attributes of food, such as its organoleptic qualities and is also due to the variable nature of its raw materials. It may, however, be useful to have a brief look at the various stages in the general development of quality control.

The earliest stage, operator quality control, is of course the method used by the housewife and is probably still applied in some very small branches of the food industry, such as for instance, in the making of hand dipped chocolates, in some small bakeries, or in the manufacture of sausages by a butcher.

Larger units developed a system described as foreman quality control where a number of workers were responsible to a senior worker, who may also be the owner of the enterprise, both for output and quality. This system is still in operation in hotel or in restaurant catering where a number of cooks work under the supervision of a chef. It also applies to some small manufacturers of, for instance, flour or sugar confectionery and in the manufacture of highly specialized products with a comparatively small market, such as pâtés and other products for the 'delicatessen' trade.

As, however, the size of the operation grew it became gradually necessary to separate quality control from production and to introduce methods of control mainly based on inspection by individuals specifically charged with this function. This was helped by the development of food science and of relevant analytical methods. Thus the chemist and in some fields also the microbiologist started to assume an ever increasing role in the control of the quality of food products.

As the size of operations increased, the problems of representative sampling both of raw materials and of finished products became more urgent as even visual inspection of a large part of the goods produced became physically impossible. This led to the adoption of statistical methods both for sampling procedures and for the interpretation of analytical results. Charts were introduced for weight control procedures and for processing and product control and there is a vast literature available on the application of statistical methods and charts to quality control in industry in general and food in particular.

In 1951 A. V. Feigenbaum of the General Electric Company in New York published a book on quality control which in a revised version issued 10 years later bore the title of *Total Quality Control*.

Let me quote the author's definition of total quality control. Total quality control is 'an effective system for integrating the quality-development, quality-maintenance, and quality-improvement efforts of the various groups in an organization so as to enable production and service at the most economical levels which allow for full customer satisfaction'.

The author states that in the above phrase of 'quality control' the word *control* represents a management tool with four steps:

- (a) setting quality standards,
- (b) appraising conformance to these standards,
- (c) acting when the standards are exceeded, and

(d) planning for improvements in the standards.

You will note that all four steps quoted refer to standards and it is to the subject of standards that I wish to devote the rest of my introductory remarks.

What then are standards and how are they arrived at?

Standards can be classified in many ways and a full discussion of this subject would require a *Symposium* on its own. We can, however, attempt to look briefly at a few types of standards.

The most ambitious standard with the widest field of application is of course the *Codex Alimentarius*. Few people may be aware of the fact that a proposal for an international *Codex Alimentarius* was made as long ago as 1894 at an International Congress of Chemistry held in Brussels—but the idea, although resurrected from time to time, did not find any practical application until the late Dr Hans Frenzel in Vienna organized a Codex Committee. This was eventually absorbed into the Codex Alimentarius Commission set up under joint FAO/WHO auspices.

At present, when the possibility is being discussed that the U.K. may join the Common Market, many manufacturers of food are asking themselves and those who can advise them as to what effect such a step would have on British food legislation and, therefore, on standards with which they would have to comply. It is, however, equally important that they should be familiar with the work of the Codex Commission. The U.K. may or may not join the Common Market but there is little doubt that although acceptance of the Codex Standards is voluntary, the U.K. will accept the Commission's proposals. As this is such an important issue and as I would hazard the guess that the majority of those present here have never read the General Principles of the *Codex Alimentarius*, I do not apologize overmuch for quoting from them extensively.

The *Codex Alimentarius* is a collection of internationally adopted food standards presented in a uniform manner. These food standards aim at protecting the consumer's health and ensuring fair practices in the food trade. Their publication is intended to guide and promote the elaboration and establishment of definitions and requirements for foods, to assist in their harmonization and in so doing to facilitate international trade.

The *Codex Alimentarius* is to include standards for all principal foods, whether processed, semi-processed or raw, for distribution to the consumer. Materials for further processing into food should be included to the extent necessary to achieve the purposes of the *Codex Alimentarius* as defined. The *Codex Alimentarius* is to include provisions in respect of food hygiene, food additives, pesticide residues, contaminants, labelling and presentation, methods of analysis and sampling.

Codex Standards contain requirements for food aimed at ensuring for the consumer a sound, wholesome food product free from adulteration, correctly labelled and presented. A Codex Standard should, therefore, for any food specify: (a) the product designation, definition and composition, (b) its hygiene requirements, (c) the weight

and measure requirements, (d) labelling requirements, and (e) sampling, testing and analytical methods.

The Codex is an example of an international voluntary standard, which on adoption by a member country becomes a national, statutory or legal standard with all the necessary powers of enforcement behind it. As far as quality control is concerned all steps must be taken to conform to the standard. The preparation of a Codex Standard is a very lengthy procedure, taking several years of collaborative work between hundreds of people and one would like to expect from it a full identification of a product. Unfortunately this is not the case as at the present state of ignorance in food science it is still impossible to define in objective terms and to measure many of the organoleptic qualities which are of paramount importance to the consumer.

This aspect becomes more prominent when dealing with some apparently simpler standards such as, for instance, a buying specification for a raw material. Thus a manufacturer of jams might wish to draw up a contract with a grower for the supply of strawberries and has to draft a specification to ensure for himself a standard quality of fruit. The layman judges the quality of strawberries by their appearance, by the size and colour of the individual berries and by the absence of blemishes. These are the only criteria by which he decides whether to buy or not to buy an individual punnet of strawberries at the grocer's. To the manufacturer, however, these qualities are hardly relevant, the one all important quality to him is the flavour which the strawberries will impart to his jam. He must attempt to write this into his specification. He may try to reduce his difficulties by specifying the botanical variety of strawberries and the soil on which they will be grown, he may lay down certain analytical criteria such as sugar content, but by and large his acceptance or rejection of any particular batch of strawberries will be controlled by sampling and examination with little help from the documentary standard specification.

Let me now turn to another example of say, a vegetable oil or fat. The chemist who is usually concerned with the drawing up of specifications will have the inclination to draft it in such detail that it will specify all the measurable characteristics. He should instead ask himself which are the characteristics important for the product in question and limit himself to such relevant characteristics. There would be little point in specifying the colour of the oil or its clarity for a product where these factors do not influence its use. Unfortunately he may again come up against the difficulty of lack of knowledge. One of the most important and troublesome characteristics of some vegetable fats is their tendency to oxidize rapidly causing a deterioration in taste. In the very early stages of this process this is not accompanied by a significant rise in peroxide values and no reliable method is available to predict whether one batch of oil is more prone to oxidation than another. Thus in this case one of the most important factors in its quality assessment cannot be standardized.

Even apparently quite satisfactory standard specifications require constant revision. Most manufacturers will have had the experience that a raw material which over

a long period was used with full satisfaction is suddenly causing trouble, while still fulfilling the requirements of the standard specification. The search for the one unspecified and, therefore, uncontrolled attribute may sometimes be a very long and difficult one. If successful then obviously the relevant data should be incorporated in the standard. Sometimes the less satisfactory situation will arise that experience will show that 'As' raw material is satisfactory while 'Bs' apparently identical one behaves differently. It may thus become necessary to standardize not only the material but also its source of supply.

These few examples should have illustrated the difficulties facing the quality control manager especially in his all important task of laying down the standards on which all quality control must be based.

Finally, may I stress the fact that the setting up of a quality control organization within a factory does not by itself ensure the maintenance of quality. The Quality Control Manager is only the co-ordinator of all quality aspects in his Company and the success of his work will ultimately depend on the degree of quality consciousness which he will induce in all members of the Company from the Boardroom to the worker and on the degree of collaboration which he will succeed in obtaining from everyone concerned.

What does the consumer want?

BARONESS ELLIOT OF HARWOOD

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ALL CONSUMERS have to eat and all of us react in an entirely personal way to food. Very few of us I am afraid know that you, the food scientists, exist. And when we do, I am afraid that we may suspect you are up to no good. Adding additives there, changing flavours here, improving on nature's colours, defeating bacteria and enzymes, looking for qualities like 'improved shelf life' that the consumer never considers, creating new 'taste sensations', new foods.

It is quite understandable that weights and measures legislation relating to food is the earliest form of legislation controlling trade and that the earliest legislation that protected the consumer from the sophistications of the producer was the 1860 *Act for Preventing the Adulteration of Articles of Food and Drink*. The safety and the freshness of the food we eat is something we all feel strongly about. For most people food and drink is the largest item in the weekly budget. But we are a long way from the days when the family was primarily a food producing economic unit.

I am a farmer as well as a consumer and, therefore, I do produce some of my own food. I am well able to gauge the fitness and quality of my own produce from my own experience. But when it comes to manufactured food, I am in exactly the same position as all other consumers, i.e. a long way removed from being an informed producing, unit.

You, like all producers, have access to knowledge and information which does not filter through to the consumer. It is because this situation is general in all highly industrialized countries that consumers have begun to realize that they are not in as strong a bargaining position as producers and suppliers. The first manifestation of the growth of a 'consumer movement' in any country is almost invariably the production by one organization or another of a comparative test magazine. Reports on the comparative testing of branded products make it possible for consumers to see that they can have access to specialized knowledge hitherto kept within an industry or trade. Manufacturers and retailers no longer have a monopoly of material which can contribute to informed buying. In this country, this job of giving comparative information is done splendidly by the magazine *Which?* published by the Consumers' Association.

The Consumer Council has a different job. What we have to do is to inform ourselves about matters affecting consumer interest, promote action to deal with such problems

and to provide advice for consumers. The Council may not carry out comparative testing, take up complaints on behalf of individuals or engage in legal action. We concern ourselves with what consumers want from all types of goods and services.

I am bound to say that to find out what consumers want from food is one of the most difficult problems to tackle. We have to remember that you have persuaded us to buy not only the comparatively simple foods that are essential, but to want and to convince ourselves that we cannot do without a whole host of mouth-watering and delicious new products. You have helped to keep the United Kingdom not only fed, but over-fed. The products you have created and the way they have been marketed have made us happy to hand over the key of our kitchen to you. This means you have a great responsibility. After all, food science and food technology has revolutionized the food we eat, e.g. you are entirely responsible, not nature, or food producers, or farmers, for one of today's most sensational successes with young people—fish fingers and tomato ketchup. I hope you will not take it amiss if I suggest to you that it is more by good luck than good management that this food combination has good nutritional value. Many people are concerned that so many of your most attractive and palatable foods are merely empty calories that contribute nothing or very little to positive good nutrition and may indeed contribute quite a lot to the great nutritional problem of Western countries—overweight. It would have been easier if the title of this paper had been 'What doesn't the consumer want?'. We get letters from the public telling us of their dissatisfaction with food products. In the main they relate to lack of information about grades and compositional standards, about ingredients and additives and about the standards of hygiene in food shops. We are too, on the receiving end of the representations of the active lobbies that express concern about the use of food additives and about some of the methods used for rearing livestock.

The aspect of buying food which most consumers write to us about relate to the label and the package and to what I can only describe as quantitative assessments. For example, we are posed the question, how much water is there in canned fruit? Consumers with complaints about the eating *quality* of food, however, are more likely to return to the shop and complain or to get in touch with the local Public Health Department.

With modern retailing methods, the housewife has often few guide lines which will help her to judge the produce she is considering buying. If she is in an unfamiliar shop she has primarily two 'tests'—the label on the package and the general appearance of the shop. She may also get the advice of the shop-keeper, but this is becoming rarer. She may simply want to know the brand name as indication enough to her of the quality she can expect. If, however, she wants to shop comparatively she will need to know more. The appearance of the shop and its staff will give some idea of the standards of cleanliness and hygiene that prevail and she will use this as a yardstick to estimate how products that are perishable or not pre-packed may be handled. It is not necessarily a reliable yardstick, but it is the only one available.

The label

What information then should the label give? More than most manufacturers are prepared to concede and less than consumer theorists might like to see. This, at any rate, seems to be the first indication from a survey into consumer attitudes towards labelling that the Consumer Council is at present carrying out through Conrad Jameson Associates.

For the consumer a label should give clear information about the product and its use. For the manufacturer it has to be attractive enough to assist the sale of the product. There has to be more information than the average consumer can understand, or wants to be bothered to understand in order to provide a check on the manufacturer. The first phase of Mr Jameson's research consisted of four group discussions and ten depth interviews. This pilot research indicated that there are three areas where consumers want protection.

Water content

It seems that the amount of water is a sore point for consumers when they buy canned fruit and vegetables. Consumers would like to be able to compare brands according to the amount of fruit in, say, different brands of peaches.

No one is suggesting that every time a shopper buys a can of fruit she wants to compare labels but there are women who would like to check up on what they are buying. Why should not she have a statement of water content which would allow her to do this? The Consumer does not believe that the technical arguments put forward by manufacturers about the difficulty of defining water content are insuperable. The other argument that this would create a price war based on quantity and that quality would suffer is also one that does not impress the Consumer Council. If one thing is clear it is that consumers do not always equate quality and quantity.

Quantity is one of the basic checks available to consumers but information about quantity does not mean standardization of quality. In fact, I would go so far as to say that better information about quantity would mean that housewives would look for other features besides price when comparing products.

The consumer might ask where does the fruit come from and what is the strength of syrup used? We might then even get a consumer grades for canned food.

Anyway, in our view the Consumer Council has put forward a strongly felt consumer point when it asked the Ministry of Agriculture for regulations requiring a declaration of added water for goods such as canned fruit or stews.

Common or generic names

Consumers are confused by names like 'squash' or 'steaklet' or 'pork luncheon meat' or 'stewed steak with gravy'. It is not enough to have compositional standards for products. What the consumer wants to know is what the words mean in everyday language. The Consumer Council research indicates that housewives are making wrong assessments

about quality and price simply because they cannot find out the real meaning of words. For those of you who spend your working lives trying to improve quality standards it must be a cause of worry that to your ultimate customer the product you describe as 'Meat Loaf with Cereals' sounds much the same as 'Meat Loaf with Stuffing'. You know, and I know, that there is a difference of 30% meat. You know, and I know, that there is more meat in 'Meat with Cereal' than in 'Meat Loaf with Stuffing', but the consumer doesn't know.

I sympathize with one of the housewives in Mr Jameson's surveys. Asked to discuss various brands of tinned meat—stewed steak, braised meat, stewed steak with gravy and so on—without being told that the key to judging quality might be printed on the labels themselves, one woman settled on an Irish brand by telling the interviewer: 'This one must be the best. It says it's been passed by the Ministry of Agriculture'. Few of the housewives ever considered that there might be a difference between a brand labelled 'stewed steak' and another brand labelled 'stewed steak with gravy'.

Make no mistake about this. Consumers need more information to help them to judge quality. We all have some strange ideas about the meanings of words we see on everyday products, although I am bound to say that I have never gone as far as the housewife in our research interviews who said: 'I always thought that "cling peaches" meant that they come out all cuddly'.

Listing of ingredients

Another aspect of quality that interests consumers is the way ingredients are designated. Consumers would like an indication that the ingredients are listed in order of quantity. This would mean adding four words to the label after the word 'ingredients', 'in order of quantity'. Also a number of people are concerned about added chemical ingredients and would like to know that they have been added. Some say they want them listed in full, even if they are not sure what they mean. But as far as we can tell at this stage, most consumers would like to have chemical names simplified: 'with permitted colouring', 'with permitted sweeteners', etc. The word 'permitted' seems to act as a considerable reassurance. After all even 'sodium chloride' sounds a bit frightening!

All these desirable attributes required by consumers do lie in the sphere of influence of the food scientists and the food technologist. It is not a question of leaving the responsibility for what appears on the label or package to the marketing and sales department. I should have thought that it is just as much the concern of the food scientist to see that the results of his quality control are appreciated by the consumer as it is to know that he has control over the purity and safety of the products he makes. He must show concern from the time the ingredients are taken in to the factory until, after a long or short period in the shop, they reach the customer.

I have touched on some of the preliminary results of our research into consumer attitudes about labelling by indicating where consumers need information. You might, I think, like to know that they would probably resist legislation against the free pictorial

treatment that is now allowed on packages. They do not expect the picture on the pack to be an exact indication of what is inside. Our survey has shown that they use the picture only to evoke the food and taste sensations that go with eating it. Most people assume that what is inside the pack will not be anywhere nearly as nice as they picture. Consumers want attractive packs and they expect pictorial exaggerations. They do not, in most cases, mislead. Consumers agree that they are intrigued and attracted first by the picture on the pack and read the label second. In other words, they are beguiled into the possibility of considering the product by the attraction of the picture. By convention they do not expect to take the product literally. What would, and does, irritate them is if the pack makes a verbal claim which then proves to be false. If the label says it will serve six, consumers write to us to ask 'Six what?' 'Six semi-invalids with failing appetites?'

Consumers do not want packs that are drearily utilitarian. In the Consumer Council we are certainly against standardization of packaging to a dull uniformity of appearance. We do not believe that within a framework of rules that will protect the consumer from being misled, it is not possible to provide all the glamour consumers admittedly want with the information they need.

Most consumers have their own quality standards. I feel I know what I mean by quality and in all the papers that follow, different aspects of quality in the food industry will be discussed. You will say, and you will be right, that with food we judge the product in the end in relation to the senses of sight, smell and taste. These, I gather, are the 'organoleptic factors' which are difficult to measure accurately by scientific instruments. I am sure we take for granted—not too much for granted I hope—the quality control of the food industry in terms of safety and purity.

Food scientists and food technologists start with the food when it comes from the producers. At the other end, we consumers get it on our plates, or our foil television supper trays, or in a handy package. We are doing our best to make consumers more discriminating, more rational in their choice, more questioning. We are doing our best to educate consumers not simply to look for the cheapest product or to insist on the largest quantity for the smallest amount of money. We are trying to persuade consumers to look for guide lines and check points that will enable them to get better value for *their* money. We know that consumers have to learn how to choose correctly for their own needs. They have to learn to look more clearly at the value they are offered. We see it as one of the major tasks of the Council to persuade and educate consumers to be more discriminating and articulate but it is important that the lines of communication and all stages of production should be kept wide open and not, as happens too often to-day, become blocked and disconnected in the sales department. I feel that it is in your interest too that the consumer is well informed about the wide choice of goods available.

Technologists and retailers, believe it or not, are too reticent about explaining the capabilities of the products they sell. The real decision about which products to

buy, which products to make available to the consumer are made too often by the buyers in the supermarkets rather than by the customers at the counter. If it is true, and it seems it is, that it is the colour photographer who can first of all attract and persuade the consumer to consider a new food product, and that the second check is the information that is given, I am sure the real reason why the consumer comes back for more lies in *your* hands. At present it is the 'organoleptic qualities' that turn your customers into an addict. I look forward to the time when factors like nutritive value and value for money will be important too. This is surely a job for consumer education through consumer information; and I judge that the quality of information you give about your products is one of the factors that needs to be given high priority.

2nd Session. Chairman: LORD SAINSBURY

Chemical and physical qualities

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Summary. One of the main purposes of quality control is the economical production of standardized products which show the minimum variation from one day to the next. In formulating products various factors have to be considered such as relevant legal and specification requirements, organoleptic acceptability and changes which take place during processing and storage. Raw materials, intermediates and the product are controlled between pre-arranged limits by employing physical and chemical tests at appropriate stages.

Foods such as flour and margarine have to be artificially fortified. The Food Standards Committee have accepted the necessity of adding approved compounds to some foods to improve colour and texture or to inhibit putrefaction. Although raw materials are checked for purity, contaminants may be picked up during the process unless constant vigilance is practised.

Introduction

Although this paper mainly deals with chemical and physical qualities, the organizers of this *Symposium* suggested that it should commence with an outline of the quality control system as it is applied in the food industry. The following, therefore, represents a summary of the sequence of events in typical manufacturing schemes, where scientific control in some form should be considered in order to produce a standardized product:

(1) There is *Agricultural control*, where vegetables are grown from selected seed; where fruits are picked at the best stage of maturation for canning, freezing or jam making; also pigs are sent to the sausage factory when they are within a definite weight range as a control over the lean to fat ratio.

(2) The *raw materials* coming into the factory are checked. Although mostly of biological origin and, therefore, varying in properties, many of them have been standardized already by having been submitted to a certain degree of processing. Those from new suppliers in particular may be preceded by a small representative *buying sample* drawn from the bulk. Then the material subsequently delivered should correspond with that sample. Raw materials should be pure, able to withstand storage and be suitable for the purpose intended so that, apart from chemical and physical

tests, a small-scale *trial batch* of the product is prepared and examined for various properties and, particularly, by organoleptic examination.

(3) Having decided on our formulation, we then become involved with *process control*. The checking of level, temperature and pressure are important here, but it may also be necessary to control the composition and properties of intermediates rapidly, and in some instances, automatically. Such materials may include brines, syrups and pie fillings, or chlorinated cooling water for canning. Rapid or immediate feed back of information is important in process control and on-stream methods are applied in some instances.

(4) The *finished product* is also examined to ensure it complies with any relevant legal or recognized standards or limits, and, more generally, to ascertain that the necessary standardization has been achieved. Its composition and other measurable criteria should fall within any pre-arranged limits and no undesirable materials or organisms should have been picked up during the process. The product is also made up as the housewife would do in the home. Other checks on the product are for weight, appearance of the pack, and ability to withstand storage. Strictly speaking, *product control* is too late to be true control, but it is valuable – provided the results are regularly assessed on a statistical basis.

(5) In the final stage some *control of rotation of stock* in the retailers premises is also called for, especially with the more perishable goods. Here it is important to educate the shopkeeper and supermarket employee to carry out correct storage procedures; in fact, just as important as instructing factory personnel in hygienic handling.

Physical qualities

The first attribute of the title of this paper – physical qualities – are associated closely with factors that affect the senses and should preferably be measurable by instruments, such that the results correlate with organoleptic findings.

Firstly the general appearance of food and package is important – particularly when the customer is deciding on a first purchase. Appearance however is made up of several factors such as size, shape, colour and gloss. The grading of natural raw materials is often related to size, an operation which can be done by machinery. In canned, and more particularly bottled goods, for instance, it is important to have each individual fruit approximately the same size. Certainly many types of defects still have to be judged by eye, but equipment is available for removing discoloured units by deflection or flotation. With meat there is still nothing however which replaces the visual examination of the experienced inspector in the grading of carcasses for quality attributes.

Rheological properties

Flow

The ability to flow is of increasing importance in view of the use now made of bulk

delivery and handling. Viscosity or consistency, i.e. resistance to flow, is examined in creams, sauces, preserves, gelatine solns, syrups, chocolate couverture, oils and batters. Its measurement is used as a quality control tool on raw materials, and on intermediates, in order to standardize the physical nature of the product. Fluidity is the tendency of a liquid to flow, i.e. the reverse of viscosity. Most fluid food materials are non-Newtonian, however, so that the resistance to flow (viscosity) changes as the rate of shear alters. Therefore the apparent viscosity figure obtained with foods differs according to the manner in which the measurement is made. Thus although elaborate and expensive viscometers are available, empirical limits can be readily set up by measuring the time to flow down a funnel or for a metal ball to fall a standard distance through the material.

With gelatine and pectin-sugar-acid gels the jelly strength is affected by heat applied during the process. The strength is measured by finding the weight required to push a standard plunger into the set gel or to rotate a blade in it. The tenderness and, therefore, maturity of peas can be assessed from the laboratory determination of the alcohol-insoluble solids, but the method is time-consuming. We have available, however, standardized tenderometers, which rapidly measure the force required to shear through the peas. This instrument is often used as the basis for packer's bonus payments to farmers.

With frozen foods in general, texture of the thawed product is very closely related to the raw material and the rate of freezing. Thus rapid freezing reduces denaturation of protein, there is minimum drip on thawing, and the texture of the cooked material is satisfactory. Alternatively slow freezing from 32° to 23° F causes the production of large ice crystals outside the muscle fibres and the melted water is not re-absorbed on thawing. In consequence the cooked material tends to have a tough unattractive texture—perhaps best known with fish.

With meat, consumer studies have shown that tenderness is the most important palatability factor in acceptance, especially with beef. It depends on ante-mortem factors such as feeding and resting of the animal before slaughter, and post-mortem on the degree of conversion of tough collagen to soft gelatine during conditioning and cooking. The tenderness can be assessed using the Warner-Bratzler method by measuring the force required to shear through the meat, but unfortunately the values on the raw meat do not correlate well with the tenderness of the cooked material. There is also what is referred to as a recording strain gauge denture tenderometer, which consists of a pair of human dentures arranged to simulate the frequency and motions of chewing.

Other important physical factors to consider are bulk density of powders and particle size. With chocolate for instance the smoothness is directly related to the size of the sugar and cocoa particles. Also with many products we aim at achieving a reasonable degree of homogeneity. Additionally frequent use is made of measurements of refractive index for getting a rapid idea of the sugar concentration of syrups, preserves,

purées and fillings. Similarly solids in solution can be controlled from the density either automatically or by using specially calibrated hydrometers which give a rapid measure of brine and syrup strengths.

Chemical properties

The chemical composition of a food affects the physical properties.

In calculating out our recipe we have to consider any relevant legal and specification requirements, organoleptic acceptability, and changes which take place during processing and storage.

Also compositional qualities are in some way related to nutritional factors, the main components being protein (the term used loosely for the total nitrogenous matter), the fat, carbohydrate, water, mineral matter, acidity and fibre. These terms frequently appear in specifications and laboratory reports.

In addition there are often traces of many other compounds present in foods including the important vitamins.

The control of water content is of major interest in the food industry. Most natural foods contain 60–95% water and do not keep well. In the factory however we can store them after drying, e.g. as dried milk, dried egg and dried spices containing less than 10% moisture. Incidentally the reconstitutability of dehydrated foods is affected by the method of water removal; and the best type of packaging to use can be assessed from the equilibrium relative humidity.

Also apart from keeping qualities, the amount of moisture in powders such as starches and cereals, cocoa, sugar and salt has to be controlled below certain limits as its presence causes caking and lumpiness. On the other hand, if some meat products are made too dry they have a hard texture and there is a loss of juiciness and succulence. In a rather similar vein, ice cream with little air incorporated into it, is comparatively hard and lacks the creamy texture of products with a higher overrun.

The amount of oils and fats present also affects quality in different ways. With salad cream, statutory regulations require a minimum of 25% of oil to be present. Also minimum fat requirements apply in the case of liquid, condensed and dried milk, and with cheese, butter, ice cream and (in Codes of Practice) for butter confectionery and cream soups. The fat in milk is a good example of natural compositional variation, it being dependent on factors such as breed of animal, season, feeding, etc. The Sale of Milk Regulations recognize this by laying down minima of 3·0% fat and 8·5% solids-not-fat, such that, if a milk falls below these figures it is presumed to be adulterated unless proved to the contrary. Bulking nullifies the variation to some extent of course, particularly in the case of the effect of breed. For consumers who wish to avoid having animal fat, 'filled milks' are available in the liquid, condensed and dried forms.

With meat, the fat is less desirable than the protein-rich lean tissue, and maximum fat contents are often included in specifications. Fat does, however, contribute to the

texture and the varying proportions present in different cuts affects the binding properties with water and rusk in sausage meat.

Coffee and cocoa are examples of foods where the natural bean is unsuitable for use without a certain degree of modification. With cocoa for instance it is found necessary to remove the shell, reduce the fat by half and add alkaline emulsifier in order to produce a good cocoa that is not too oily and slakes well with milk. The shell in cocoa beans (and similarly the husk in peppercorns) have much higher cellulose or fibre contents than the corresponding interiors. The fibre figure is useful, therefore, for assessing the quality of cocoa and white pepper.

As, however, the outside of the wheat grain similarly contains more than the starchy endosperm, a *minimum* statutory requirement for fibre is applied to brown flour and brown bread in order to ensure that the whole wheat has been used. Milling produces a whiter flour, so the grade can be assessed from the lower fibre content; and allowing for fortification if necessary, from the mineral matter. Alternatively the whiteness of a paste made from the flour can be measured photoelectrically by means of the Kent-Jones and Martin Flour Grader.

Similarly in sugar refining, the mineral content falls as we remove colour, so that the whitest sugar produced has the lowest ash. At the same time it must be realized that the mineral content of many foods does include elements which are important nutritionally, and salt which affects the keeping qualities as well as the taste.

Turning to protein, a reasonable intake is important for body building and meat and fish represent valuable sources of it. However, the natural variation of the composition of flesh has represented one of the major problems in standardization of products for many years. The meat content of products such as sausages is assessed in relation to the average protein or nitrogen content in the raw flesh (Society of Public Analysts, 1952). After allowing for any nitrogen present in any filler the meat nitrogen is divided by an average factor for the nitrogen in the fat-free material to give an assessment of the lean meat content. The actual factors to use as the average nitrogen have been the subject of some controversy, but after extensive surveys, the Society for Analytical Chemistry (Table 1) have recommended figures for meat, poultry and fish, which should settle the position for some time to come. In deciding on the suitability of flour for breadmaking, the total nitrogen content is of less significance than the physical properties of the gluten, which in turn vary according to the country of origin and variety of the wheat.

With carbohydrates, the sugar content is important in many products as it affects the sweetness and the keeping qualities and in order to standardize the sweetness of canned fruit, an allowance has to be made for the dilution of the added syrup by the weaker juice in the fruit.

Further, yeasts and moulds are liable to grow in foods such as preserves unless there is about two-thirds of total sugars in the aqueous phase. Additionally, approximately two-sevenths to four-sevenths of the total sugars should be present as invert sugar, or

crystallization is liable to ensue on storage. Also with lemon curd there should be 3–4% effective gelatinized starch present to ensure homogeneity.

TABLE 1. Nitrogen in the fat-free factors of various flesh foods recommended by the Society for Analytical Chemistry (1961–67)

Type of meat, fish	Nitrogen in the fat-free (%)		References to reports of the Society for Analytical Chemistry, <i>Analyst, Lond.</i> (date, volume and page)
	Recommended mean	Range of values (approximate)	
Pork	3.45	2.8–4.2	1961, 86 , 557
Beef	3.55	2.96–4.53	1963a, 88 , 422
Chicken (breast)	3.9	—	1963b, 88 , 583
Chicken (dark meat)	3.6	—	1963b, 88 , 583
Chicken (whole carcass)	3.7	—	1963b, 88 , 583
Ox-liver	3.45	—	1964, 89 , 630
Pig livers	3.65	—	1964, 89 , 630
Veal	3.35	3.04–3.68	1965a, 90 , 256
Turkey (breast)	3.9	—	1965b, 90 , 581
Turkey (dark meat)	3.5	—	1965b, 90 , 581
Turkey (whole carcass)	3.65	—	1965b, 90 , 581
Kidney	2.7	—	1966a, 91 , 538
Cod	2.85	2.871 ± 0.137	1966b, 91 , 540
Tongue (ox and pig)	3.0	2.24–3.59	1967, 92 , 326

With fruit (after picking) it gets softer, the colour changes and there is an increase in the respiration rate and the soluble pectin content (Fig. 1). For manufacturing, the fruit is preferably used when nearly at the climacteric of the respiration curve, that is, just prior to maturity. Processing then completes the reaction to produce the desirable texture and the full effective pectin content. If over-soft fruit is used for canned fruit, mushiness is likely to appear in the product. In jam making not all fruits have naturally sufficient pectin setting power and pectin solutions may have to be added near the end of the boil to give a good setting product.

In apples, bananas and potatoes the starch tends to be converted partially to sugar. A high starch content in apples tends to cause an undesirable mealiness. For crisp manufacture potatoes are stored for a period at about 60–65° F in order to ensure they have a high starch content prior to slicing and frying. Alternatively storage at about 30° F encourages the reverse conversion of starch to sugar, thus producing the sweet tasting potatoes occasionally met with in bitter winters.

Overall the attractive taste of fruit products is due to a delicate balance between the sugar and acid contents, both of which vary during ripening and senescence. More

generally, acidity and, more particularly the pH, are controlled in various products as they affect taste, keeping qualities and the setting and viscosity of gels. With meat and fish, lactic acid is formed during rigor, thus producing its own preservative.

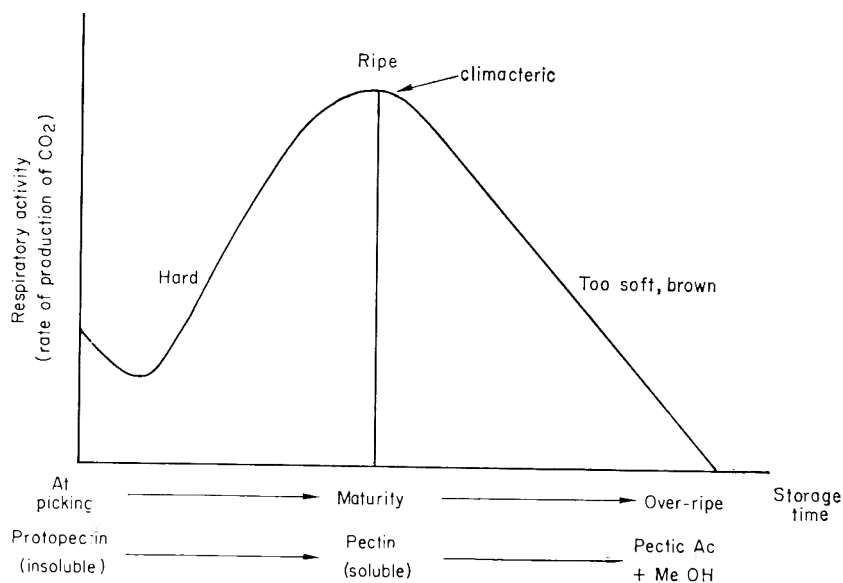


FIG. 1. Typical curve showing changes in respiratory activity of fruits during storage in air. The concomitant changes in the texture and pectin are also indicated. At the climacteric the fruit has the desirable colour, texture, taste and maximum effective soluble pectin content.

From the foregoing it is apparent that the main substances in foods—water, fat, mineral matter, fibre, protein, acid and carbohydrate—affect the quality of food products. Apart from the fact that many of these components consist of several substances, foods contain many other compounds which contribute to odour and taste. With spices, the odour is derived from the compounds in the volatile oil and in fish, ammonia and trimethylamine increase during storage, even in ice, and cause undesirable odours. With flavour, although there are simple examples such as that in vanilla being due to vanillin, it seems that overall taste is due to a subtle balance of numerous compounds, many of which are present in minute amounts.

Foods of course also supply us with important vitamins and minerals. Care is necessary, however, in manufacture to ensure particularly with vitamin C, that there are not undue losses due to heat processing. Some foods are of course designed for special diets and others represent more staple foods that are artificially fortified.

Fortification

Compulsory fortification was first seriously considered in World War II, when the (then) Ministry of Food looked into supplementing the diet to ensure it contained adequate amounts of certain minerals and vitamins. This has continued into the present day and statutory regulations require most types of wheat flour to be fortified with added calcium, and all types must comply with certain minimum requirements for iron, vitamin B₁ and nicotinic acid. As the outside of the grain is comparatively rich in these nutritional elements, fortification is more necessary in the case of the whiter flours. A special problem that arises with flour, however, is the difficulty of 'mixing in' the master mix of nutrients evenly.

Margarine has to be fortified by the addition of the fat-soluble vitamins A and D and although there are no minimum standards for these vitamins in butter, it is interesting to note that the recent Food Standards Committee Report on 'Claims and Misleading Descriptions', in mentioning the variable amounts found, recommends that butter should not be exempt from general labelling requirements and states that '... at present claims for the presence of vitamin A may legally be made for butter containing very little'. The Report then makes the following recommendation:

'There should be no exception to the rule that vitamin claims should not be made without a quantitative declaration. We recommend that no claim for the presence of vitamin A in butter should be permitted unless substantiated by a quantitative declaration in the same way as for other products.'

Additives and processing aids

Colours, emulsifying and stabilizing agents and (in the case of flour) bleachers and improvers must be drawn from the permitted lists. Legislation in the near future will also probably prohibit the use of certain flavours, and solvents may have to be drawn from a restricted list. Approved preservatives and antioxidants may only be added to certain named foods up to stated limits. Such compounds, although possibly undesirable from the aesthetic point of view, have become useful adjuncts in the manufacture of certain products.

The Food Standards Committee, in adopting several reports from Sub-Committees relating to the use of such additions, have in general accepted the necessity of adding compounds to some foods to improve the colour and texture and to arrest putrefaction, provided they do not adversely affect health. The first report on colouring matters for instance stated that: '... without the addition of colour many foods would have a drab unattractive appearance. If, therefore, there is adequate evidence that the colours used have no deleterious effects on health we see no objection to their use to replace natural colour during processing, to standardize appearance or simply to render a product more attractive.'

The Pharmacology Panel classified the various coal-tar dyes as to whether they were innocuous or had harmful effects, and the number of permitted colours has since been reduced in the light of later findings. Smoked fish is an example of a product which has changed considerably over the years—the softer dyed kipper now being perhaps the typical product to many consumers. More generally colours are an example where in view of differing Regulations in different countries the food manufacturer may have to vary the colours used according to the destination of the product.

Preservatives enable foods from other parts of the globe to be used, thus producing greater variety in the diet. Also the preserves manufacturer is able to cope with the glut seasons by putting the fruit under sulphur dioxide so that jam can be made from it over the year, thus evening out the use of plant and labour. Sulphur dioxide is also useful for retention of colour and vitamin C content, but it tends to inactivate vitamin B₁. By using benzoic acid in comminuted drinks made from the whole fruit the flavour is less affected than when sulphur dioxide is used, but the product tends to darken and lose vitamin C more rapidly. Fortunately the anti-fungal agents used on fruits such as diphenyl and orthophenylphenol tend to be absorbed almost completely on the outside, so that most of the preservative is removed if the peel is rejected.

With salted meat, nitrite acts quite differently by combining with the meat pigment to produce pink nitrosomyoglobin. If heated the pink denatured nitrosomyochromogen typical of hams is formed.

Emulsifying and stabilizing agents have various properties as the term includes substances that facilitate uniform dispersion of oils in water, some that enhance the palatable life of bread and cakes, others that produce froth (or reduce it) and others that reduce spattering of heated fats. Those on the permitted list are valuable aids in baking and in the manufacture of margarine, cheese, ice cream, sugar confectionery, soft drinks, flavours, cake mixes and caramel. There is a statutory permitted list of compounds for such purposes, without which according to the Food Standards Committee Report, it would be impossible to make many composite foods and soft drinks in the form to which the public have grown accustomed. Summarizing, there is little doubt that the problem of feeding large populations is markedly assisted by the use of various types of harmless additives in some of our foods.

Contaminants

Although raw materials are checked for purity, undesirable contaminants may be picked up during the process unless constant vigilance is practised. Typical examples are perhaps trace elements (especially metals from equipment), plasticizers, materials from conveyor belts, dirt in milk and mineral oil from the cutting heads of dough dividers.

The Food Standards Committee has published several reports on contaminants, including six of the trace elements, and mineral oil. Although limits for trace elements have either been imposed by regulation, or recommendation by the Food Standards

Committee, it must be borne in mind that, whereas large amounts of copper and zinc cause emesis, small amounts of both are necessary for normal metabolism. On the other hand arsenic and lead appear to have only detrimental effects when ingested, and statutory limits apply to almost all foods.

Similarly it has been established that the continued consumption of liquid paraffin may reduce the absorption of fat soluble vitamins A and D, and the ingestion of emulsified mineral oil may cause deposition in the liver and intestinal wall. In such cases statutory control is obviously desirable in the public interest.

A quite different form of contaminant is extraneous matter, which is given so much publicity when found in a very small proportion of manufactured goods. Although one must extend considerable sympathy to the customer when this occurs, in fairness to the manufacturer it must be said that sieving, flotation and settling, and the use of magnets and electrostatic deflection are often applied to raw materials and X-rays and electronic devices are used for detecting objects in the product. Also what may look like glass in canned products usually turns out to be sparingly soluble crystals which are natural to the fish (Purcell & Hickey, 1922; McFee & Swaine, 1953) or fruit (Manley & Alcock, 1950; Dickinson & Fowler, 1955).

Future trends

If we try to look into the future one cannot foresee that the variable biological raw materials can be made completely standard in composition and properties. Such advances as have already been made with spice extracts, which have a controlled composition and are cleaner than the crude materials, will probably be extended however to other ingredients.

Perhaps it will be in agriculture that the main changes will take place. Certainly for many years variety selection and crop treatment have helped to produce a steady supply of material, which in turn is reflected in our standardized fruit, vegetable and cereal products. We know rather less about genetic modification of animals and poultry. But this may well be the field in which the changes will come.

From the researchers we need more knowledge of what contributes to flavour, aroma, texture and colour, using modern sophisticated and sensitive tools. If corresponding rapid tests could then be devised this would be an extremely useful addition to the routine control system.

On a more mundane note we need more information distributed on modern compositional and physical data. With so many new materials and products it is impossible for any one organization to determine everything required. May I add, therefore, that at Weybridge we are prepared to act as a collection house for data—obviously we would acknowledge the source of any figures published. No one would expect formulations and recipes to be divulged, but typical analytical data obtained from raw materials and final products would be extremely useful. Also there is a need for International legislation such as that being formulated by ISO and the Codex Ali-

mentarius to standardize, for instance, lists of permitted additives and minimal composition standards for raw materials and products—then our formulations will be suitable for sale in any market—here or abroad.

References

- DICKINSON, D. & FOWLER, H.D. (1955) *Analyst, Lond.* **80**, 706.
MANLEY, C.H. & ALCOCK, A. (1950) *Analyst, Lond.* **75**, 280.
MCFEE, E.P. & SWAINE, R.L. (1953) *Fd Engng*, **25**, 67.
PURCELL, C.S. & FICKEY, C.H. (1922) *Analyst, Lond.* **47**, 16.
SOCIETY OF PUBLIC ANALYSTS (1952) *Analyst, Lond.* **77**, 543.

Microbiological qualities

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Principles

The purposes of microbiological quality control, whether carried out by the Government Inspection Services or industry, are mainly two-fold: prevention of food-borne disease and retardation of microbial spoilage. In addition quality control must also meet general microbiological quality requirements, having no direct relationship to health or spoilage, e.g. microscopical counts of moulds in canned tomato products or the enumeration of viable spores of the genus *Bacillus* in cocoa powder.

It is now generally accepted that microbiological quality control by the industry itself should be primarily of a preventative nature (Wilson, 1955). This is much more effective than the analytical approach, i.e. inspection of the microbiological condition of the food as it leaves the factory, and subsequently taking corrective measures if necessary. Authorities and buyers have also come to understand that, for statistical reasons (Juran, 1962; Steiner, 1967), factory inspection is more effective, for their purposes too, than examination of finished samples.

Preventative microbiological quality control entails, in essence, the drafting and supervision of the measures required to keep microbial counts low by reducing the numbers of micro-organisms contaminating the food and preventing the proliferation of those micro-organisms inevitably present. In this connection the following phases of contamination of, and proliferation in, foods have to be distinguished: (i) primary = microbial load of the raw materials; (ii) secondary = increase in counts during processing; (iii) tertiary = post-processing recontamination; and (iv) quaternary = contamination and/or growth during culinary preparation of the manufactured commodity.

Such control should be attained by the following measures:

- (i) procurement of raw materials of the best possible microbiological quality;
- (ii) prevention of contamination of raw materials prior to processing;
- (iii) proper processing, of whatever kind applied;
- (iv) prevention of contamination of materials during and particularly after processing;
- (v) proper packaging;
- (vi) adequate storage, transportation and handling of the finished product prior to consumption.

Obviously in this respect not all micro-organisms have the same significance. Specific enteropathogenic and enterotoxinogenic bacteria, viruses and particular helminths have to be controlled for the safety of the consumers. In addition the growth of moulds in foods has to be kept under control since many types of fungi have been found recently to be mycotoxinogenic (cf. Table 1). A great variety of bacteria, yeasts and moulds are typical spoilage agents for a given type of food (*vide infra*). Hence, in addition to the general precautions just outlined, special measures are often necessary to control the occurrence or growth of particular micro-organisms in foods (Bunker, 1967).

TABLE 1. Mycotoxinogenic moulds

Species	Clinical effect	Susceptible species
GROUP 1: PRODUCERS OF WELL-DEFINED MYCOTOXINS		
<i>Alternaria tenuis</i>	ATA	Man
<i>A. sp.</i>	Haemorrhages	Mouse
<i>Aspergillus amstelodami</i>	Emaciation	Poultry
<i>A. candidus</i>	cf. <i>Pen. citrinum</i>	
<i>A. clavatus</i>	Haemorrhages	Poultry
<i>A. flavus</i>	Hepatic carcinoma	Poultry, man
	Tremors	Mouse
	Haemorrhages	Swine
<i>A. fumigatus</i>	Perirenal oedema	Swine
<i>A. glaucus</i>	Haemorrhages and diarrhoea	Poultry
<i>A. niger</i>	cf. <i>Asp. flavus</i>	
<i>A. ochraceus</i>	Hepatic injury	Poultry, rat
<i>A. oryzae</i>	Hepatic necrosis	Various
<i>A. ostianus</i>	cf. <i>Asp. ochraceus</i>	
<i>A. parasiticus</i>	cf. <i>Asp. flavus</i>	
<i>A. ruber</i>	cf. <i>Asp. flavus</i>	
<i>A. terreus</i>	cf. <i>Pen. citrinum</i>	
<i>A. wentii</i>	Emaciation	Poultry
<i>Chaetomium globosum</i>	Haemorrhages and paralysis	Rat
<i>Cladosporium epiphyllum</i>	ATA	Man
<i>Fusarium culmorum</i>	Anorexia	Bovine
<i>Fusarium nivale</i>	Emaciation and gangrene	Bovine
<i>Fus. roseum</i> (Syn. <i>Gibberella saubinetti</i>)	Hepatic necrosis	Swine
<i>Fus. sporotrichioides</i>	ATA	Man
<i>Gibberella zeae</i>	Oestromimetic response	Swine
<i>Mucor hiemalis</i>	ATA	Man
<i>Penicillium brevicompactum</i>	ATA	Man
<i>Pen. citreoviride</i>	Ascending paralyses	Various
<i>Pen. citrinum</i>	Haemorrhages and renal damage	Poultry, mouse
<i>Pen. cyclopium</i>	Tremors	Mouse
<i>Pen. islandicum</i>	Hepatic atrophica and cirrhosis	Various
<i>Pen. puberulum</i>	cf. <i>Asp. flavus</i>	

TABLE 1 (continued)

Species	Clinical effect	Susceptible species
<i>Pen. rubrum</i>	Haemorrhages and hepatic injury	Swine
<i>Pen. rugulosum</i>	cf. <i>Pen. citrinum</i>	
<i>Pen. tardum</i>	cf. <i>Pen. citrinum</i>	
<i>Pen. variabile</i>	cf. <i>Asp. flavus</i>	
<i>Pen. viridicatum</i>	Renal damage	Swine, rat
<i>Pen. species</i>	cf. <i>Asp. ochraceus</i>	
<i>Pithomyces chartarum</i>	Angiocholecystitis	Sheep
	Facial oedema	Bovine
<i>Rhizopus</i> sp.	cf. <i>Asp. flavus</i>	
<i>Stachybotrys atra</i>	Haemorrhages	Horse

GROUP 2: PRODUCERS OF LESS WELL-DEFINED ORALLY ACTIVE TOXINS

Asp. avenaceus, *Asp. carneus*, *Asp. chevalieri*, *Asp. nidulans* and *Asp. niveus*

Cladosporium fragi

Fusarium moriliforme

Paecilomyces varioti (Syn. *Byssochlamys fulva*)

Pen. oxalicum, *Pen. piceum*, *Pen. purpurogenum* and *Pen. urticae*

Trichoderma lignorum

References from which data in Table 1 have been taken:

- CHRISTENSEN, C.M., NELSON, G.H. & MIROCHA, C.J. (1965) Effect on the white rat uterus of a toxic substance isolated from *Fusarium*. *Appl. Microbiol.* **13**, 653.
- DODD, D.C. (1965) Facial eczema in ruminants. *Mycotoxins in Foodstuffs* (Ed. by G.N. Wogan), p. 105. Cambridge, Massachusetts.
- FISHER, E.E., KELLOCK, A.W. & WELLINGTON, N.A.M. (1967) Toxic strain of *Fusarium culmorum* (W.G. Sm.) Sacc. from *Zea mays* L., associated with sickness in dairy cattle. *Nature, Lond.* **215**, 322.
- FORGACS, J., CARLL, W.T., HERRING, A.S. & MAHLANDT, B.G. (1954) A toxic *Aspergillus clavatus* isolated from feed pellets. *Am. J. Hygiene*, **60**, 15.
- FORGACS, J., CARLL, W.T., HERRING, A.S. & HINSHAW, W.R. (1958) Toxicity of *Stachybotrys atra* for animals. *Trans. N.Y. Acad. Sci.* **20**, 787.
- FORGACS, J. & CARLL, W.T. (1966) Mycotoxicoses: toxic fungi in tobaccos. *Science, N.Y.* **152**, 1634.
- GILGAN, M.W., SMALLEY, E.B. & STRONG, F.M. (1966) Isolation and partial characterization of a toxin from *Fusarium tricinctum* on moldy corn. *Archs Biochem. Biophys.* **114**, 1.
- HODGES, F.A., ZUST, J.R., SMITH, H.R., NELSON, A.A., ARMBRECHT, B.H. & CAMPBELL, A.D. (1964) Mycotoxins: aflatoxin isolated from *Penicillium puberulum*. *Science, N.Y.* **145**, 1439.
- IZUKA, H. & IDA, M. (1962) Maltoryzine, a new toxic metabolite produced by a strain of *Aspergillus oryzae*, var. *microsporus* isolated from the poisonous malt sprout. *Nature, Lond.* **196**, 681.
- JOFFE, A.Z. (1962) Biological properties of some toxic fungi isolated from overwintered cereals. *Mycopathol. mycol. applic.* **16**, 201.
- KINOSITA, R. & SHIKATA, T. (1965) On toxic moldy rice. *Mycotoxins in Foodstuffs* (Ed. by G.N. Wogan), p. 111. Cambridge, Massachusetts.
- KROGH, P. & HASSELAGER, E. (1968) Studies on fungal nephrotoxicity. *Royal Veterinary and Agricultural College, Copenhagen. Yearbook 1968*, p. 198.
- LARSEN, S. (1928) Om kronisk degeneration af nyrerne (nefroser) hos svin forårsaget af skimlet rug. *Maanedsskr. Dyrk.* **40**, 259.

- MCERLEAN, B.A. (1952) Vulvovaginitis of swine. *Vet. Rec.* **64**, 539.
- MERWE, K.J. VAN DER, STEYN, P.S., FOURIE, L., SCOTT, D.B. & THERON, J.J. (1965) Ochratoxin A, a toxic metabolite produced by *Aspergillus ochraceus* Wilk. *Nature, Lond.* **205**, 1112.
- MIROCHA, C.J., CHRISTENSEN, C.M. & NELSON, G.H. (1967) Estrogenic metabolite produced by *Fusarium graminearum* in stored corn. *Appl. Microbiol.* **15**, 497.
- MIYAKE, M. & SAITO, M. (1965) Liver injury and liver tumors induced by toxins of *Penicillium islandicum* Sopp growing on yellowed rice. *Mycotoxins in Foodstuffs* (Ed. by G.N. Wogan), p. 133. Cambridge, Massachusetts.
- MORTIMER, P.H. (1963) The experimental intoxication of sheep with sporidesmin, a metabolic product of *Pithomyces chartarum*. IV. Histological and histochemical examinations of orally dosed sheep. *Res. vet. Sci.* **4**, 166.
- RABIE, C.J., TERLANCHE, M., SMIT, J.D. & DE KLERK, W.A. (1965) Toxicity of *Aspergillus wentii* Wehmer. *Sth Afr. J. agric. Sci.* **8**, 875.
- REHM, H.J. (1967) Mykotoxine und ihre Bedeutung für die Lebensmittelchemie. *Deut. Lebensm. Rundschau.* **63**, 269.
- RUTQVIST, L. & PERSSON, P.A. (1966) Studies on *Aspergillus fumigatus* experimental mycotoxicosis in mice, chicks, and pigs with the appearance in pigs, of perirenal edema. *Acta vet. scand.* **7**, 21.
- SCOTT, D. (1965) Toxicogenic fungi isolated from cereal and legume products. *Mycopathol. Mycol. appl.* **25**, 213.
- STOB, M., BALDWIN, R.S., TUIE, J., ANDREWS, F.N. & GILLETTE, K.G. (1962) Isolation of an anabolic, uterotrophic compound from corn infected with *Gibberella zeae*. *Nature, Lond.* **196**, 1318.
- TATSUNO, T. (1964) Metabolites of *Penicillium islandicum* sopp. *Fd cosm. Toxicol.* **2**, 678.
- THERON, J.J., MERWE, K.J. VAN DER, LIEBENBERG, N., JOUBERT, H.J.B. & NEL, W. (1966) Acute liver injury in ducklings and rats as a result of ochratoxin poisoning. *J. Path. Bact.* **91**, 521.
- THORNTON, R.H. & PERCIVAL, J.C. (1959) A hepatotoxin from *Sporidesmium bakeri* capable of producing facial eczema diseases in sheep. *Nature, Lond.* **183**, 63.
- TOWNSEND, R.J., MOSS, M.O. & PEEK, H.M. (1966) Isolation and characterization of hepatotoxins from *Penicillium rubrum*. *J. Pharm. Pharmac.* **18**, 471.
- WALBEEK, W. VAN, SCOTT, P.M. & THATCHER, F.S. (1968) Mycotoxins from food-borne fungi. *Can. J. Microbiol.* **14**, 131.
- WILSON, B.J. & WILSON, C.H. (1962) Extraction and preliminary characterization of a hepatotoxic substance from cultures of *Penicillium rubrum*. *J. Bact.* **84**, 283.
- WILSON, B.J. & WILSON, C.H. (1964) Toxin from *Aspergillus flavus*: production on food materials of a substance causing tremors in mice. *Science, N.Y.* **144**, 177.
- WILSON, B.J. (1966) Toxins other than aflatoxins produced by *Aspergillus flavus*. *Bact. Rev.* **30**, 478.
- WILSON, B.J., HARRIS, T.M. & HAYES, A.W. (1967) Mycotoxin from *Penicillium puberulum*. *J. Bact.* **93**, 1737.
- YATES, S.G., TOOKEY, H.L., ELLIS, J.J. & BURKHARDT, H.J. (1968) Mycotoxins produced by *Fusarium nivale* isolated from tall fescue (*Festuca arundinacea* Schreb.). *Phytochemistry*, **7**, 139.

Supervision and laboratory examination of the factory

Microbiological quality control, in this context, involves, first of all, frequent checks on proper processing and handling of goods throughout the plant. An important example of factory quality control is found in the heat processing of foods, the most frequently used mode of decontamination as well as preservation. The net microbicidal effect of a heat treatment depends strongly on: (i) the time-temperature combination applied; (ii) the pH and water activity (a_w) of the food treated, and (iii) the efficacy

TABLE 2. Verification methods used in microbiological plant control

Parameter to be verified	Recommended instrument or method	Reference
Time-temperature integrals	Thermograph; indicator strips; tests for residual enzyme activity	Stumbo (1965), Ingram & Roberts (1966)
Relative humidity or a_w	Polyelectrolyte resistance humidity elements	Musa & Schnable (1965)
Decontamination efficiency	Spec. count of nat. or artific. contam. sample	Mossel, van Schothorst & Kampelmacher (1967)
	(i) $\frac{\text{Same count of treated sample}}{\text{Count of spores of } Bacillus}$	Mossel (1967)
	(ii) $\frac{\text{Total bacterial count}}{\text{Total bacterial count}}$	
Surface decontamination efficiency	(i) Swabs	Higgins (1950), Thomas (1961), ten Cate (1965), Holt (1966), Mossel, Kampelmacher & van Noorle Jansen (1966)
	(ii) Impression plates, agar slices, tapes or pads	
Microbiological condition of water supply	(i) Total count of mesophilic and psychrotrophic bacteria	
	(ii) MPN enumeration of <i>Enterobacteriaceae</i> and <i>E. coli</i>	Mossel (1967)
Microbiological condition of air supply	(i) Slit sampler technique	Bourdillon, Lidwell & Thomas (1941)
	(ii) Absorption in an impinger, or moistened plugs of calcium alginate	May (1966), Richards (1955), Mossel (1964)
Sanitary condition of hands	Five-finger impression counts on ETGPA* and VRBG†	de Vries & Knape (1964)
Body cleanliness	VRBG, ETGPA and blood agar impression plates of predilection sites	Sarkany & Gaylarde (1967), Mossel <i>et al.</i> (1968)
Effectiveness of disinfectants and detergent sanitizers	(a) Strains: <i>Staph. aureus</i> , <i>Salin. typhimurium</i> , <i>Pseud. aeruginosa</i> , <i>Str. faecalis</i> , spores of <i>B. cereus</i> ; <i>Sacch. cerevisiae</i>	
	(b) Exposure: 5 min at <i>c.</i> 15° C	Mossel (1963)
	(c) Neutralization: Tween-blood-thiosulphate broth	
	(d) Requirements: 5 D, except sometimes for spores	

*Egg yolk tellurite glycine pyruvate agar.

†Violet red bile glucose agar.

of measures taken to avoid postprocess recontamination. Each of these factors has to be controlled within narrow limits if heat processing is to be effective (Lennington, 1967).

Also regular examinations have to be carried out of the food processing machinery. This has to be dismantled and decontaminated when required by the bacteriological data obtained. Otherwise considerable build-up of various types of organisms may occur due to continuous culture (Perry, 1928; Collins *et al.*, 1968; Bacic, Cousins & Clegg, 1968; Hyndman, 1968). In addition, periodical checks are to be made of premises, cleanliness of the staff, packaging materials and the water supply. Also, the pressing need for adequate surveillance of the air supply has been demonstrated repeatedly (Mossel, 1964; Lennington, 1967; Collins *et al.*, 1968; Schulz, Voss & Leder, 1968). Finally the evaluation of any disinfectants and detergent sanitizers used forms an important part of the task of the laboratory (Mossel, 1963; Litsky & Litsky, 1968).

Table 2 reviews the methods which have been found useful in the author's Institute during the last 15 years. The types of organisms for which one seeks, obviously, depend greatly on whether one is interested in those that may spoil a given food, or in organisms that might render the food unsafe for consumption, that is, lead to food-borne disease eventually if not immediately. However, whatever types may be of interest, one is always concerned with their numbers per unit of surface area or weight. Qualitative results, often formulated simply on the basis of the absence or presence of a certain organism on 'the' machinery or in 'the' air supply, etc., are usually of lesser value, unless formulated in a semi-quantitative way, i.e. absence of the organism in a well specified quantity or surface area.

Microbiological quality control of raw materials

It should be stressed that it may not always be easy to procure raw materials of satisfactory bacteriological quality. This is particularly so in the case of red meats and poultry. To obtain carcasses with a tolerable degree of *Salmonella* contamination, the meat and poultry industry has had to begin to extend control measures to the farms. This includes provision of pathogen-free young animals, bacteriological control of feed, and considerable improvement of the sanitary conditions of the animals on the farm, during transportation, and while awaiting slaughter (Saulmon, 1966; Newell 1967; Gerriets, 1967; Edel *et al.*, 1967; Heard, Jennett & Linton, 1968). Pending these developments, initially rather tolerant specifications will have to be used, but these will gradually have to be made stricter, applying the 'sliding scale' principle (Goldenberg, 1968).

Even where such vast problems do not exist, the drafting of microbiological specifications for raw materials should be done very carefully and particularly with proper reference to ecological principles. This involves taking the following aspects into account: (i) the significant organisms amongst the microflora of the raw materials under study; (ii) the numbers of each of these that can be allowed in the final products;

(iii) the desired safety margins as determined by the processing to be applied in the course of manufacture; and (iv) attainability of the specifications. With respect to the latter point, considerable collaboration is often required before suppliers can consistently meet purchasers' specifications, although this is, very fortunately, not always as difficult as in the case of carcase meats or poultry.

The microflora of minor components, e.g. spices, may also impair the microbiological quality of manufactured foods. As to requirements for such materials, their microflora should not essentially influence the load of organisms, which determine wholesomeness and/or keeping quality that are encountered in the major component of the food. Hence, requirements for minor ingredients can be more lenient, the less their concentration is in the final product (Mossel, 1955).

Microbiological examination of the final product

Careful implementation of microbiological quality control at the production level, on the above basis, will usually assure adequate keeping quality and wholesomeness. In addition, one might wish to assess the microbiological condition of the food itself. Such final product analysis is of interest to the processor. However, to the public health official of the country importing the food products, and to the bacteriologist working on behalf of the buyers, it is often the only information available. In other words, microbiological examination of the final product takes an important place in quality control of the food industry, but only as a mode of *assessing* proper production, not as a means of *attaining* the required microbiological quality.

Keeping quality

Direct determination of stability of shelf-stable items. Shelf life tests carried out for this purpose should be designed carefully.

Firstly, the number of samples and the degree of randomization with which these are to be taken should be determined accurately. Too small a number of samples, or inadequate randomization may lead to a false feeling of security, with regard to the keeping quality of the final product, whereas too high a degree of sampling may lead to exceeding the capacity of the laboratory. A proper mathematically justified approach to optimal sampling is therefore always required (Steiner, 1967).

With regard to the choice of storage temperature it is necessary to use in shelf life tests a cyclic system of incubation. The reasons for this are that: (i) changing temperatures will also occur in practice; and (ii) water vapour migration effects often limit the shelf life of packaged products (Mossel & Sand, 1968), and such effects are potentiated by the use of temperature changes during incubation. It is, therefore, our practice to incubate samples of shelf-stable foods at $30 \pm 1^\circ \text{C}$ during the day time and at $15 \pm 1^\circ \text{C}$ overnight.

A final point is the choice of criteria of spoilage. In order to obtain fast preliminary results, it can often be recommended that counts of significant types of organisms (*vide infra*) are carried out in fresh as well as in incubated samples after the storage test has been in operation for say about a week. By comparing the counts of such organisms in incubated samples with the counts of the same organisms in fresh samples, or samples stored under refrigeration, a tentative impression of the growth of potentially dangerous organisms in the food under examination can be obtained. Quite often such an increase in counts indicates inadequate shelf life very much earlier than organoleptic tests may do. Clearly, the greatest caution has to be exerted in interpreting the course of such counts. On no account should significance be attributed to minimal increases in counts, until proper statistical treatment of the data has confirmed these differences to be significant. It is our experience that a significant increase requires at least that counts have doubled and often, i.e. in more heterogeneous materials, even tripled.

Prediction of the keeping quality of perishable foods. In general no direct assessment of keeping quality is attempted but instead quantitative determinations are made of the organisms capable of spoiling the food under study – the so-called spoilage association. From such data the keeping quality can then be estimated (Mossel & Ingram, 1955).

This association depends on certain properties of the food, viz. those which determine whether or not a given organism will be able to proliferate in the food. These properties are primarily related to water activity and pH, and the presence of antimicrobial compounds, either naturally occurring ones or those added during processing such as salt, vinegar, smoke components, and synthetic preservatives. In Table 3 a review of the organisms which play a rôle in the microbial spoilage of particular foods is presented. In Table 4 the methods which have been found useful for their enumeration have been compiled.

Wholesomeness

Pathogens. Various groups are relevant to the determination of the wholesomeness of foods (Hobbs, 1967). A review of these organisms and recommended methods for their enumeration are presented in Table 5. Those currently of major interest from the standpoint of outbreaks of food-borne diseases are *Salmonella*, *Staph. aureus* and *Cl. perfringens* (Vernon, 1966), and to a lesser extent *B. cereus* (Nikodemuss, 1966) and *Vibro parahaemolyticus* (Zen Yoji *et al.*, 1965). Also, as indicated before, quantitative examinations for moulds should be made regularly, since so many types of fungi, according to Table 1, form orally active mycotoxins and can, therefore, not be tolerated in foods. Either viable mould spores or microscopically visible mycelia and spores have to be determined, dependent on whether the majority of the moulds is still viable or already dead. Suitable methods for this purpose will be recommended in the following sections.

TABLE 3. Spoilage association of the most important classes of staple foods

Class	pH	a_w	Processing with microbicidal effect	Examples of commodities	Association						
					Gram-negative rods	Catalase pos. Cocci	Catalase neg. Cocci	<i>Lacto-bacillaceae</i>	<i>Bacillaceae</i>	Moulds	Yeasts
1	> 4.5	> 0.95	None	Fresh meats, fish, shell fish, poultry, eggs and egg products	+++	+	±	0	0	+	0
2	> 4.5	> 0.95	None	Vegetables	+++	0	±	+	+	+	0
3	> 4.5	< 0.90	None	Cereal grains, pulses	+	+	0	+	+	+++	+
4A	< 4.5	> 0.95	None	Fruits	0	0	0	+++	0	+++	+++
B				Juices	++	0	++	++	0	±	++
5	> 4.5	> 0.95	Pasteurization	Liquid milk	±	±	+	+	+	0	0
6	> 4.5	c. 0.95	Cooking	Cooked sausage, canned large size hams	0	0	±	+	+	0	0
7	> 4.5	c. 0.95	Baking	Bread, rolls, cakes	0	0	0	0	+	+	±
8A	> 4.5	< 0.90	None	Dried vegetables, cereals and cocoa	0	0	0	0	0	+	0
B	> 4.5			Marzipan, chocolate fillings	0	0	0	±	0	+	++
C	< 4.5			Dried fruits	0	0	0	±	0	++	++
9	c. 4.5	c. 0.96	None	Butter and margarine	0	0	+	0	0	+	+
10A	> 4.5	> 0.95	Appertization	Meats, vegetables and milk packed in hermetically sealed containers	0	0	0	0	0	0	0
B	< 4.5	> 0.95	Appertization	Fruits and juices packed in hermetically sealed containers	0	0	0	0	0	+	0

Frequency scale used: +++ = virtually exclusive, ++ = dominant, + = significant, ± = minor or occasional, 0 = virtually of no importance in spoilage.

*The *Pseudomonas/Acinetobacter/Achromobacter*-group, unless otherwise indicated.
 †*Acetobacter*.

TABLE 4. Review of recommended methods for the microbiological examination of foods with particular reference to spoilage aspects

Group of organisms sought	Presumptive counting procedure	Confirmation	Completion or identification
Gram-negative, rod shaped bacteria	Plating on Olson's (1961) 1 ppm Crystal Violet plate count agar and incubation at 13° and 32° C	OxGNIKlMo	The necessary biochemical tests, including oxidation of 10% lactose and test for pigment formation on MP
<i>Lactobacillaceae</i>	Counts in Rogosa, Mitchell & Wiseman's (1951) acetate agar at 32° C	Microscopy, Ca	TAesLacMaltSaMaSoCell
Non- <i>Lactobacillaceae</i>	Counts in 0.5% gelysate 0.3% malt extract agar at 32° C	Microscopy, Ca	If required: biochemical confirmation tests
Aerobic count	Counts in Mossel & Krugers Dagneaux's (1959) TDYM agar* at 32° C, or 13° C when psychrophilic or psychrotrophic organisms have to be enumerated (Mossel & v.d. Moosdijk, 1964)	—	—
Anaerobic count	Counts in SAn vs TAn (Mossel <i>et al.</i> , 1965) at 32° C	—	—
Spore counts	Counts in TDYM, or SAn/TAn, after heating the suspensions for 1 min at 80° C (Mossel, 1967)	Microscopy	—
Moulds and yeasts	Counts in yeast extract glucose oxytetracyclin agar, † incubated at 22° C (Beech & Carr, 1955; Mossel, Visser & Mengerink, 1962; Sainclivier & Roblot, 1966; Koburger, 1968)	Microscopy — but hardly necessary	—

Ovolytic organisms	Plating on blood agar base with 0.5% NaCl and 2% egg yolk, and incubation at 32° C	If required: microscopy and further examination of egg yolk clearing and/or precipitating colonies	—
Lipolytic organisms	Counts in thin layers of a suitable agar † layered over an appropriate lipid phase (Eijkman, 1901; Tuynenburg Muys & Willemse, 1965)	If required: microscopy and further examination of colonies surrounded by a precipitation zone	—
Osmophilic organisms	Counts in 60% (w/w) fructose-0.5% yeast extract agar ($a_w = 0.83$) at 30° C (Mossel, 1951; Mossel & Bax, 1967)	If required: microscopy and further examination of colonies obtained	—

* If food sample high in moulds add 1000 µg/ml primaricin (Mossel & Sand, 1968).

† If food sample is high in mould spores which form copious aerial mycelia, add 1 : 15 000 rose bengal (Smith & Dawson, 1944).

‡ For bacteria, see aerobic count; for moulds and yeasts, see moulds and yeasts above. If the food sample may contain lipases of non-microbial origin, it is recommended to correct for non-microbial lipase activity by spreading 0.1 ml of the 1 : 10 dilution on to plates containing a thin layer of oxytetracylin pimarinin agar plus the standard fat layer (Mossel & Sand, 1968).

TABLE 5. Review of recommended methods for the microbiological examination of foods with particular reference to Public Health aspects

Group of organisms sought	Presumptive counting procedure	Confirmation	Completion or identification
<i>Salmonella</i> and <i>Arizona</i>	MPN-enrichment procedure with at least 10-g aliquots using: (i) for general purposes: Muller (1923)-Kauffmann's (1935) tetrathionate broth at 43° C (Harvey & Thomson, 1953; Read & Reyes, 1967; Harvey & Price, 1967); (ii) if resuscitation is required: use of Hobbs' (1962) nutrient broth or North's (1961) lactose broth incubated at 30° C, followed by (i) above (Mossel & Vincentie, 1968) <i>When counts > 10/g might occur:</i> enumeration in sulphite iron agar (Mossel, 1959) contained in plastic pouches (Bladel & Greenberg, 1965; de Waart & Smit, 1967)	Plating of 0.1 ml of 10 ⁻⁵ dilutions, simultaneously on 1.5-cm dia-plates of (i) special Brilliant Green phenol red agar (Kampelmacher, 1967); (ii) deoxycholate citrate agar; (iii) bismuth sulphite agar, aged according to Cook (1952)	Subsequently: (i) KI → βGal. → P → UDKAMal (ii) agglutinations (iii) phage test (Buttiaux, Moriametz & Papavassiliou, 1956; Ewing, Davis & Reavis, 1957; Le Minor & Ben Hamida, 1962; Ewing & Fife, 1966)
<i>Shigella</i>	Plating of at least 100 mg on SS-agar and Taylor's (Taylor & Harris, 1965; Taylor & Schelhart, 1968) XLD agar in 15 cm dia. plates and incubation at 37° C	Subculturing black colonies onto BPA	βGal. → P → as above
<i>Enterobacteriaceae</i>	(i) <i>When counts > 10/g are expected:</i> Plating in MacConkey ('Violet Red bile') glucose agar (Mossel, Mengerink & Scholts, 1962) incubated 20 hr at 37° C, unless resuscitation is required — <i>vide supra</i> (ii) <i>When counts < 10/g are expected:</i> MPN-enrichment procedure with 0.1, 1 and 10 g using buffered Brilliant green bile glucose (EE) broth, at 30° C, unless resuscitation is required — <i>vide</i>	OxGNi <i>Either:</i> plating on MacConkey ('Violet Red bile') glucose agar (VRBG) <i>Or:</i> DEG ≡ plating onto VRBG, Chapman's (1951) tergitol/TTC/agar at 44° C and	Completion tests as for <i>Salmonella</i> + Mo Lac. + → C → E Lac. θ → βGal. → P → UDKAMal. OxGNi → McC → McC → <i>vide supra</i>

<i>Staph. aureus</i>	0-1, 1 and 10 g, using Brilliant Green bile (BGB) broth at 44° C, unless resuscitation is required — <i>vide supra</i>	Adequate numbers of typical egg yolk positive as well as typical but egg yolk negative (de Waart <i>et al.</i> , 1968; Hall, 1968) colonies: Co	Phage typing
	(i) <i>When counts > 10²/g are expected:</i> Plating on Baird Parker's (1962) ETGP agar incubated for 30 hr at 37° C	Plating on ETGP agar, etc., <i>vide supra</i>	Co, phage typing
	(ii) <i>When counts < 10²/g are expected:</i> MPN-enrichment procedure with 0-1, 1 and 10 g, using Giolitti & Cantoni's (1966) anaerobic tellurite glycine mannitol pyruvate broth, incubated at 37° C		
Lancefield group D streptococci	Plating in Packer's (1943) crystal violet sodium azide blood agar, incubated at 37° C*	TAzLCa	Sherman characters, especially BiT; So
<i>Str. mitis</i> and <i>Str. salivarius</i>	Plating on Chapman's (1944) TCTS-agar, incubated at 37° C	TAzBiT → AesArGLevRaSo (Cowan & Steel, 1965)	
<i>Clostridium</i> sp.	Counts, in glucose free 0-05% sulphite iron polymyxin agar (Mossel, 1959; Narayan, 1967) contained in Miller-Prickett tubes or plastic pouches, at 32° C	AnCaSu	MiMoNiIGSaLacGelLec (anaer.)
<i>Cl. botulinum</i>	MPN-enrichment procedure in sulphite iron polymyxin broth (Mossel & de Waart, 1968)		MoNiIGiSal (anaer.) → mouse test, FA (Midura <i>et al.</i> , 1967)
<i>Cl. perfringens</i>	Counts in Marshall, Steenbergen & McClung's (1965) TSN-agar at 46° C	Alcohol treatment (Johnston, Harman & Kautter, 1964) → plating on BYCA	Mo
<i>Bac. cereus</i>	Plating on Mossel, Koopman & Jongerius (1967) MYP-agar incubated at 32° C	Plating on LENA at 46° C	
<i>Vibrio parahaemolyticus</i>	Enrichment in brain heart infusion broth with 20% NaCl, 10 µg/ml polymyxin and 5 µg/ml tylosin	MiGXyAnNiGelVLec	—
		Plating on 5% NaCl soluble-starch, penicillin, bromothymol blue agar incubated anaerobically at 32° C (Baross & Liston, 1968)	OxGMoINi St

*Where blood is not available, the recommended medium is methylene blue azide agar of pH 8-0 (*Enterococcus* confirmatory agar of Winter & Sandholzer, 1946) with 10 µg/ml kanamycin added (Mossel *et al.*, 1968).

For nomenclature see Table 4.

<i>Nomenclature used in Tables 4 and 5</i>			
A	= Agglutination with <i>Salmonella</i> polyvalent anti O-serum	G	= Dissimilation of glucose, studied in stabbed, freshly steamed agar tubes (Mossel & Martin, 1961)
Acs	= Dissimilation of aesculin	Gel	= Dissimilation of gelatin
An	= Growth under anaerobic conditions	Gl	= Dissimilation of glycerol (cf. G)
Ar	= Dissimilation of arabinose (cf. G)	I	= Formation of indole from tryptophan
Az	= Azide tolerance	K	= KCN-tolerance (Buttiaux <i>et al.</i> 's 1956 method)
β Gal.	= β -Galactosidase reaction	KECA	= Kanamycin methylene blue azide agar of pH 8.0
BGB	= Brilliant green bile broth	KI	= Behaviour in stabbed, freshly steamed tubes of Kligler (1918) iron agar
BiT	= Growth in 40% bile broth	L	= Tolerance of azide + ethyl violet, according to Litsky, Mallmann & Fifield (1953)
BPA	= Brilliant green phenol red lactose sucrose agar	Lac	= Dissimilation of lactose (cf. G)
BYCA	= Brain heart infusion egg yolk cystein agar	Lac 10	= Oxidation of lactose on 10% lactose slants
C	= Assimilation of citrate (Simmons', 1926 method)	Lec	= Lecithinase activity
Ca	= Catalase activity	LENA	= Lactose egg yolk neomycin agar (Willis & Hobbs, 1959)
Cell	= Dissimilation of cellobiose (cf. G)	Lev	= Formation of levan from sucrose
Co	= Coagulase activity	M	= Methyl red test
D	= Lysine decarboxylase activity (Taylor's, 1961 method)	Ma	= Dissimilation of mannitol (cf. G)
E	= Eijkman's (1904) thermotolerance test, in the modification of MacKenzie, Taylor & Gilbert (1948); cf. Guinée & Mossel (1963)	Mal	= Assimilation of malonate
EE	= <i>Enterobacteriaceae</i> enrichment broth (Mossel, Visser & Cornelissen, 1963)	McG	= Crystal violet neutral red bile lactose agar (McConkey, 1905)
ETGP	= Egg yolk tellurite glycine pyruvate agar (Baird-Parker, 1962)	Mi	= Microscopic examination
FA	= Fluorescent antibody stain technique	Mo	= Motility test in U-tubes
		MP	= Mannitol peptone agar (Sellers, 1964)
		MPN	= Most probable number
		MYP	= Mannitol egg yolk polymyxin agar (Mossel, Koopman &
		G	= Dissimilation of glucose, studied in stabbed, freshly steamed agar tubes (Mossel & Martin, 1961)
		Gel	= Dissimilation of gelatin
		Gl	= Dissimilation of glycerol (cf. G)
		I	= Formation of indole from tryptophan
		K	= KCN-tolerance (Buttiaux <i>et al.</i> 's 1956 method)
		KECA	= Kanamycin methylene blue azide agar of pH 8.0
		KI	= Behaviour in stabbed, freshly steamed tubes of Kligler (1918) iron agar
		L	= Tolerance of azide + ethyl violet, according to Litsky, Mallmann & Fifield (1953)
		Lac	= Dissimilation of lactose (cf. G)
		Lac 10	= Oxidation of lactose on 10% lactose slants
		Lec	= Lecithinase activity
		LENA	= Lactose egg yolk neomycin agar (Willis & Hobbs, 1959)
		Lev	= Formation of levan from sucrose
		M	= Methyl red test
		Ma	= Dissimilation of mannitol (cf. G)
		Mal	= Assimilation of malonate
		McG	= Crystal violet neutral red bile lactose agar (McConkey, 1905)
		Mi	= Microscopic examination
		Mo	= Motility test in U-tubes
		MP	= Mannitol peptone agar (Sellers, 1964)
		MPN	= Most probable number
		MYP	= Mannitol egg yolk polymyxin agar (Mossel, Koopman &
		Ni	= Nitrate reduction
		Ox	= Oxidase activity (Kovacs' 1956 method)
		P	= Phenyl alanin desaminase (Buttiaux <i>et al.</i> 's 1956 method)
		Ra	= Dissimilation of raffinose (cf. G)
		Sa	= Dissimilation of saccharose (cf. G)
		Sal	= Dissimilation of salicin (cf. G)
		SAN	= Soytone yeast extract cystein agar (Mossel <i>et al.</i> , 1965)
		So	= Dissimilation of sorbitol (cf. G)
		SS	= <i>Salmonella</i> - <i>Shigella</i> -agar
		St	= Dissimilation of starch
		Su	= Sulphite reduction in 0.05% Na ₂ SO ₃ /FeSO ₄ -agar
		T	= Growth at 45 \pm 0.1° C
		TAN	= Tryptone yeast extract cystein agar (Mossel <i>et al.</i> , 1965)
		TCTS	= Trypan blue crystal violet tellurite sucrose agar (Chapman, 1944)
		TDYM	= Tryptone dextrose yeast extract peptonized milk agar (Mossel & Krugers Dagneaux, 1959)
		TSN	= Tryptone sulphite neomycin agar (Marshall <i>et al.</i> , 1965)
		U	= Hydrolysis of urea (Christensen's (1946) method)
		V	= Voges-Proskauer test for acetyl-methylcarbinol
		VRBG	= Violet red bile glucose agar (Mossel <i>et al.</i> , 1962)
		XLD	= Xylose lysine deoxycholate agar (Taylor & Harris, 1965)
		Xy	= Dissimilation of xylose (cf. G)

A short remark may be in order here with regard to the use of so-called selective media in general and in microbiological quality control in the food industry in particular. The formulation, preparation, sterilization and inoculation of such media should be most carefully standardized. The selectivity and productivity of selective media depend entirely on the active concentration of one or more added inhibitory compounds. The latter is determined by: (i) the concentration of the inhibitor added to the medium; (ii) the degree of inactivation by the sterilization treatment of the medium used; and (iii) the extent of neutralization by one or more components of the food sample examined. If such details are not properly taken into account, quite unreliable and irreproducible results—leading amongst others to a complete lack of agreement between the laboratories of buyers and sellers, or food processors and Public Health Authorities—must be anticipated (Read & Reyes, 1968).

Indicator organisms. It is, in addition, often useful to enumerate so-called indicator organisms—bacteria indicating objectionable bacteriological condition of foods. The conventionally sought proof of the absence of pathogens in a representative sample of food has significance only for the consignment under investigation. However, the repeated failure to find suitably chosen types of indicator organisms in successive samples makes it unlikely that foods manufactured in the same way will ever be dangerously contaminated. This is valuable information, not only for the manufacturer but also for the Food Inspection Services and the consumer (Mossel, 1967).

The use of indicator organisms has often been criticized (McCoy, 1967) and not always without reason (Mossel, 1967). However, provided that proper ecological consideration is given to the significance of given numbers of particular organisms in a specific food, no criticism is justified against this most valuable analytical parameter. The use of *Enterobacteriaceae* as indicator organisms may be given as an example.

In the case of foods processed with heat to such an extent that the lethal effect on non-spore bearing bacteria is somewhere between 4 and 7 decimal reductions, the presence of *Enterobacteriaceae* in numbers over 1–10/g cannot be accepted. The reason is that this would indicate that processing has not been completed or that an adequate decontamination treatment has been carried out, but it has been followed by post-process recontamination. Either of these conditions is highly undesirable, because it may have resulted in, or will result in the future in, the presence of enteric or other pathogens in such foods. However, for fresh foods or commodities that have been processed at temperatures well below 50° C, where virtually no bactericidal effect can be expected (Shelton, 1961; Stephenson, 1967), the situation is entirely different. Because the natural association of such foods is known to include *Enterobacteriaceae*, the mere presence of these bacteria in such products is no reason for their rejection. Rather, one is interested, in this instance, in knowing more exactly what types of *Enterobacteriaceae* occur, since it makes a noteworthy difference whether e.g. *Serratia* or *Enterobacter* of vegetable origin are present, or *E. coli* which might indicate a more

dangerous contamination. For this purpose we have developed the differential enterobacteriogram, in which the types of *Enterobacteriaceae* are determined in addition to their numbers (Mossel & Vincentie, 1968). There is a valid reason to reject a sample only if one or more of the following deficiencies are encountered: (i) occurrence of pathogenic species of *Enterobacteriaceae* in aliquots of 10–20 g; (ii) the presence of *E. coli* in an approximately 1–5-g sample; or (iii) the presence of viable *Enterobacteriaceae* at a level that exceeds the numbers encountered in commodities produced under sanitary manufacturing conditions (Mossel, 1967).

Enzyme tests. Reliance is often placed on the absence of the enzymatic activity of, for example, phosphatase or amylase in heat-processed products as an index of proper manufacturing (Ingram & Roberts, 1966; McCoy, 1967; Gillespie, 1967; Gantner & Körmeny, 1968). No doubt a negative result of such enzyme tests indicates that the commodities under examination have been sufficiently heat-treated to eliminate the pathogenic organisms originally present. However, enzyme tests are not sensitive enough to indicate the absence of recontamination; and this is the most frequent source of failure of pasteurization treatments (Colenso, Court & Henderson, 1966; Jones, Gibson & Cheng, 1967; Zehren & Zehren, 1968). Hence the sole use of these enzyme tests seems to lead to a false sense of security. (Bulling, 1968.) However, the use of a combination of an enzyme test with an examination for example *Enterobacteriaceae* is very profitable, since it will provide differentiation between inadequate pasteurization and post-pasteurization contamination. The food microbiologist, expected to advise on improvements in processing, can then decide, guided by the two tests, whether the heat-treatment or rather the sanitation of the post-pasteurization stages needs closer attention (Mossel, 1967).

Because of their very high radiation resistance, none of these enzymes has been found to be inactivated by doses of radiation used in radiation pasteurization ('radicidation'). This makes enzyme tests unsuitable for the control of radicidation treatments, whereas tests for indicator organisms maintain their validity here (Ingram & Roberts, 1966).

Total counts. It may sometimes be of interest to determine the total numbers of viable aerobic and facultatively anaerobic micro-organisms per 1 g of food. The latter parameters are often called total counts. They will show to what extent microbial contamination, but more particularly proliferation in general, has occurred. However, in studying food wholesomeness, the enumeration of the specific pathogenic or toxigenic bacteria or suitably chosen indicator organisms has a higher priority.

Choice of tests

An important general aspect of final product examination is that the number of tests should be limited as much as possible. This will allow large numbers of samples

to be examined, thereby satisfying statistical requirements (Steiner, 1967). This also necessitates the use of methods of maximal simplicity and rapidity which have been carefully standardized so that reliability will not suffer.

In this connection, again, the use of well chosen indicator organisms can be particularly valuable.

Necessity of standards for end products

It is a fruitless procedure to make these end product examinations without having standards available against which the results obtained can be evaluated. Hence an essential part of microbiological quality control is the drafting of specifications.

The general principles of drafting standards have been presented in the third section of this paper (p. 406). The numerical details of such an endeavour are the following. A representative number of samples of the commodity under study is taken from production lines previously checked for the absence of any technological and microbiological deficiencies. These samples are then examined for the criteria for which standards have to be set up, in view of the health or spoilage record of the food under study. A tentative standard is then the figure met by over 90 or 95% of the samples subjected to this microbiological evaluation (Surkiewicz, Groomes & Shelton, 1968).

In end product specifications, the time-temperature combination to which samples are to be exposed prior to testing also merits careful consideration. Preserved foods must obviously tolerate extensive periods of storage at temperatures up to 30–45° C, depending on their destination. Also in the case of semi-preserved or even highly perishable foods, it is useful to store them for some time at a suitable temperature before microbiological examination is carried out with the purpose of being able to anticipate the bacteriological condition of the commodity as it reaches the consumer, in addition to checking the microbiological quality at the end of the manufacturing line.

Retrospective examination of complaints

There is a third and last aspect of microbiological quality control that should be mentioned. The author suggests that it be called retrospective microbiological quality control. It consists of microbiological examination of samples of consignments that have been returned to the manufacturer because of complaints, whether because of lack of microbiological stability, or because pathogenic or indicator organisms have been detected at a level not acceptable to importing countries or buyers. The examination of such products should not be considered a nuisance, but should rather be looked upon as providing a valuable source of additional information on what may happen to the food under practical conditions.

When examining sub-standard samples, the package should firstly be examined very carefully for leakage (Maunder, Folinazzo & Killoran, 1968). In addition microbiological data on the contents can often be of considerable help in establishing the

causes of the trouble. This was, for instance, our experience when charged with the bacteriological examination of canned meats of the solid pack type following the Aberdeen typhoid outbreak, allegedly caused by contaminated corned beef (Milne, 1964; Howie, 1968). MPN counts were made of *Enterobacteriaceae* and catalase positive cocci in the material taken from the centre and the peripheral areas of the packs. A comparison of such sets of counts enabled us to estimate whether post-process recontamination of the commodity had occurred or not.

Particularly in the examination of foods preserved by other means than by heat, or not by thermal treatment exclusively, the anti-microbial parameters of the product (pH, a_w and concentration of natural or added inhibitory substances) should be checked, in order to assess whether the product might have been insufficiently preserved.

When the cause of the complaint is not detected by any of these types of examination, the microflora has to be studied in more detail. For this purpose, the methods summarized in Tables 4 and 5 can be used. When this is done in extensively spoiled samples, however, the counts found may not correspond to the amount of deterioration detected by organoleptic methods. This is due to what is often called auto-sterilization, i.e. spontaneous dying off of part of the microflora, due to anti-microbial conditions created in the food by the initial microbial proliferation. In this event a quantitative microscopic examination can first be attempted (Mossel & Zwart, 1959; Duitschaever & Ashton, 1968).

In some instances, a large fraction of the microbial cells has lysed subsequent to death and microscopic examination does not then reveal significant abnormalities. It is our experience that a chemical search for specific microbial metabolites may then often yield the information required (Mossel & Tollenaar, 1955; Salwin, 1968). When foods packed in hermetically sealed containers are examined, analysis of the gases formed (CO_2 , H_2 and sometimes CH_4) may provide the answer (O'Brien, 1967). Generally, quantitative determination of other metabolites such as volatile aliphatic acids, lactic acid, succinic acid, acetyl methyl carbinol and nitrogenous substances like histamine or aliphatic amines (Salwin, 1968) may give useful indications of the type of spoilage that has occurred.

References

- BACIC, B., COUSINS, C.M. & CLEGG, L.F.L. (1968) *J. Dairy Res.* **35**, 247.
BAIRD-PARKER, A.C. (1962) *J. appl. Bact.* **25**, 12.
BAROSS, J. & LISTON, J. (1968) *Nature, Lond.* **217**, 1263.
BEECH, F.W. & CARR, J.C. (1955) *J. gen. Microbiol.* **12**, 85.
BLADEL, B.O. & GREENBERG, R.A. (1965) *Appl. Microbiol.* **13**, 281.
BOURDILLON, R.E., LIDWELL, O.M. & THOMAS, J.C. (1941) *J. Hyg., Camb.* **41**, 197.
BULLING, E. (1968) *Arch. Lebensmittelhygiene*, **19**, 78.
BUNKER, H.J. (1967). Dealing with microbiological trouble-makers. *Biology and the Manufacturing Industries* (Ed. by M. Brook), p. 63. Academic Press, London.

- BUTTIAUX, R., MORIAMEZ, J. & PAPAVALASSIOU, J. (1956) *Annls Inst. Pasteur, Paris*, **90**, 133.
- CATE, L. TEN (1965) *J. appl. Bact.* **28**, 221.
- CHAPMAN, G.H. (1944) *J. Bact.* **48**, 113.
- CHAPMAN, G.H. (1951) *Am. J. Publ. Hlth*, **41**, 1381.
- CHRISTENSEN, W.B. (1946) *J. Bact.* **52**, 461.
- COLENZO, R., COURT, G. & HENDERSON, R.J. (1966) *Mon. Bull. Minist. Hlth*, **25**, 153.
- COLLINS, R.N., TREGER, M. D., GOLDSBY, J. B., BORING, M. R., COOHON, D. B. & BARR, R. N. (1968) *J. Am. med. Ass.* **203**, 838.
- COOK, G.T. (1952) *J. Path. Bact.* **64**, 559.
- COWAN, S.T. & STEEL, K.J. (1965) *Manual for the Identification of Medical Bacteria*. Cambridge University Press.
- DUISSCHAEVER, C.L. & ASHTON, G.C. (1968) *J. Dairy Sci.* **51**, 665.
- EDEL, W., GUINÉE, P.A.M., SCHOTHORST, M. VAN & KAMPelmACHER, E.H. (1967) *Zentbl. Veterinaer.Med.*, **14**, 393.
- EWING, W.H., DAVIS, B.R. & REAVIS, R.W. (1957) *Publ. Hlth Lab.* **15**, 153.
- EWING, W.H. & FIFE, M.A. (1966) *Int. J. syst. Bact.* **16**, 427.
- EIJKMAN, C. (1901) *Z. Bakt. ParasitKde Abt. I*, **29**, 841.
- EIJKMAN, C. (1904) *Z. Bakt. ParasitKde Abt. I*, **37**, 742.
- GANTNER, G. & KÖRMENDY, L. (1968) *Fleischwirtschaft*, **48**, 188.
- GERRIETS, E. (1967) *Fleischwirtschaft*, **47**, 949.
- GILLEPSIE, G.H. (1967) *Chem. Ind.* **14**, 588.
- GIOLITTI, G. & CANTONI, C. (1966) *J. appl. Bact.* **29**, 395.
- GOLDENBERG, N. (1968) *Fd Mf.* **43**, 29.
- GUINÉE, P.A.M. & MOSSEL, D.A.A. (1963) *Antonie van Leeuwenhoek*, **29**, 163.
- HALL, H.E. (1968) *Bact. Proc.* **77**.
- HARVEY, R.W.S. & PRICE, T.H. (1967) *J. Hyg., Camb.* **65**, 423.
- HARVEY, R.W.S. & THOMSON, S. (1953) *Mon. Bull. Minist. Hlth*, **12**, 149.
- HEARD, T.W., JENNETT, N.E. & LINTON, A.H. (1968) *Vet. Rec.* **82**, 92.
- HIGGINS, M. (1950) *Mon. Bull. Minist. Hlth*, **9**, 50.
- HOBBS, B.C. (1962). Salmonellae. *Chemical and Biological Hazards in Food* (Ed. by J.C. Ayres *et al.*), p. 227. Iowa State University Press, Iowa.
- HOBBS, B.C. (1967) Health problems in quality control: microbiological aspects. *Quality Control in the Food Industry* (Ed. by S.M. Herschdoerfer), Vol. 1, p. 67. Academic Press, London.
- HOLT, R.J. (1966) *J. appl. Bact.* **29**, 625.
- HOWIE, J.W. (1968) *J. appl. Bact.* **31**, 171.
- HYNDMAN, J.B. (1968) *FDA Papers*, **2**, 25.
- INGRAM, M. & ROBERTS, T.A. (1966) Microbiological principles. *Food Irradiation, Proc. Symp. Karlsruhe*, p. 267. IAEA, Wien.
- JOHNSTON, R., HARMON, S. & KAUTTER, D. (1964) *J. Bact.* **88**, 1521.
- JONES, G.A., GIBSON, D.L. & CHENG, K.J. (1967) *Can. J. Publ. Hlth*, **58**, 257.
- JURAN, J.M. (Editor) (1962) *Quality Control Handbook*, 2nd edn. McGraw Hill, New York.
- KAMPelmACHER, E.H. (1967) *Z. Bakt. ParasitKde Abt. I, Orig.* **204**, 100.
- KAUFFMANN, F. (1935) *Z. Hyg. InfektKrankh.* **117**, 26.
- KLIGLER, I.J. (1918) *J. exp. Med.* **28**, 318.
- KOBURGER, J.A. (1968) *Bact. Proc.* **13**.
- KOVACS, N. (1956) *Nature, Lond.* **178**, 703.
- LE MINOR, L. & BEN HAMIDA, F. (1962) *Annls Inst. Pasteur, Paris*, **102**, 267.
- LENNINGTON, K.R. (1967) *J. Milk Fd Technol.* **30**, 372.

- LITSKY, B.Y. & LITSKY, W. (1968) *Am. J. Publ. Hlth*, **58**, 534.
- LITSKY, W., MALLMANN, W.L. & FIFIELD, C.W. (1953) *Am. J. Publ. Hlth*, **43**, 873.
- MAC CONKEY, A. (1905) *J. Hyg., Camb.* **5**, 333.
- MAC KENZIE, E.F.W., TAYLOR, E.W. & GILBERT, W.E. (1948) *J. gen. Microbiol.* **2**, 197.
- MARSHALL, R.S., STEENBERGEN, J.F. & MCCLUNG, L.S. (1965) *Appl. Microbiol.* **13**, 559.
- MAUNDER, D.T., FOLINAZZO, J.F. & KILLORAN, J.J. (1968) *Fd Technol, Champaign*, **22**, 615.
- MAY, K.R. (1966) *Bact. Rev.* **30**, 559.
- MCCOY, J.H. (1967) *Chem. Ind.* **14**, 587.
- MIDURA, T.F., INOUE, Y. & BODILY, H.L. (1967) *Publ. Hlth Rep. Wash.* **82**, 275.
- MILNE, D. (1964) *The Aberdeen Typhoid Outbreak 1964*. H.M.S.O., Edinburgh.
- MOSSEL, D.A.A. (1951) *Antonie van Leeuwenhoek*, **17**, 146.
- MOSSEL, D.A.A. (1955) *Annl. Inst. Pasteur, Lille*, **7**, 171.
- MOSSEL, D.A.A. (1959) *J. Sci. Fd Agric.* **10**, 622.
- MOSSEL, D.A.A. (1963) *Lab. Pract.* **12**, 898.
- MOSSEL, D.A.A. (1964) *J. Sci. Fd Agric.* **15**, 349.
- MOSSEL, D.A.A. (1967) *J. Ass. Off. anal. Chem.* **50**, 91.
- MOSSEL, D.A.A. & BAX, A.W. (1967) *Mitt. Geb. Lebensmitteluntersuch. Hyg.* **58**, 154.
- MOSSEL, D.A.A., BEERENS, H., TAHON-CASTEL, M.M., BARON, G. & POLSPOIL, B. (1965) *Annl. Inst. Pasteur, Lille*, **16**, 147.
- MOSSEL, D.A.A., HERTOOG, C. DEN, VELDE, J., HAMMINK, J., KLAASSEN, A. & VINCENTIE, H.M. (1968) *Voeding*, **29**, 105.
- MOSSEL, D.A.A. & INGRAM, M. (1955) *J. appl. Bact.* **18**, 232.
- MOSSEL, D.A.A., KAMPFMACHER, E.H. & NOORLE JANSEN, L.M. VAN (1966) *Z. Bakt. ParasitKde Abt. I, Orig.* **201**, 91.
- MOSSEL, D.A.A., KOOPMAN, M.J. & JONGERICUS, E. (1967) *Appl. Microbiol.* **15**, 650.
- MOSSEL, D.A.A. & MARTIN, G. (1961) *Annl. Inst. Pasteur, Lille*, **12**, 225.
- MOSSEL, D.A.A. & KRUGERS DAGNEAUX, E.L. (1959) *Antonie van Leeuwenhoek*, **25**, 230.
- MOSSEL, D.A.A., MENDERINK, W.H.J. & SCHOLTS, H.H. (1962b) *J. Bact.* **84**, 381.
- MOSSEL, D.A.A. & MOOSDIJK, A. VAN DER (1964) *J. appl. Bact.* **27**, 221.
- MOSSEL, D.A.A. & SAND, F. (1968) *Conserva*, **17**, 23.
- MOSSEL, D.A.A., SCHOTHORST, M. VAN & KAMPFMACHER, E.H. (1967) *J. Sci. Fd Agric.* **18**, 362.
- MOSSEL, D.A.A. & TOLLENAAR, F.D. (1955) *Antonie van Leeuwenhoek*, **21**, 247.
- MOSSEL, D.A.A. & VINCENTIE, H. (1968) *Lab. Pract.* **17**, 1344.
- MOSSEL, D.A.A., VISSER, M. & CORNELISSEN, A.M.R. (1963) *J. appl. Bact.* **26**, 444.
- MOSSEL, D.A.A., VISSER, M. & MENDERINK, W.H.J. (1962) *Lab. Pract.* **11**, 109.
- MOSSEL, D.A.A. & WAART, J. DE (1968) *Annl. Inst. Pasteur, Lille*, **19**, 13.
- MOSSEL, D.A.A. & ZWART, H. (1959) *Arch. Lebensmittelhygiene*, **10**, 229.
- MULLER, L. (1923) *C.r. heb. Séanc. Soc. Biol., Paris*, **89**, 434.
- MUSA, R.C. & SCHNABLE, G.L. (1965) *Humidity and Moisture* (Ed. by A. Wexler), Vol. I, p. 346. Reinhold, New York.
- NARAYAN, K.G. (1967) *Z. Bakt. ParasitKde Abt. I, Orig.* **202**, 212.
- NEWELL, K.W. (1967) *Am. J. publ. Hlth*, **57**, 472.
- NIKODEMUSS, I. (1966) *Annl. Inst. Pasteur, Lille*, **17**, 229.
- NORTH, W.R. (1961) *Appl. Microbiol.* **9**, 188.
- O'BRIEN, R.T. (1967) *Fd Technol, Champaign*, **21**, 1130.
- OLSON, H.C. (1961) *J. Dairy Sci.* **44**, 970.
- PACKER, R.A. (1943) *J. Bact.* **46**, 343.
- PERRY, C.A. (1928) *Am. J. Hyg.* **8**, 694.

- READ, R.B. & REYES, A.L. (1967) *J. Dairy Sci.* **50**, 946.
- READ, R.B. & REYES, A.L. (1968) *Appl. Microbiol.* **16**, 746.
- RICHARDS, M. (1955) *Nature, Lond.* **176**, 559.
- ROGOSA, M., MITCHELL, J.A. & WISEMAN, R.F. (1951) *J. Bact.* **62**, 132.
- SAINCLIVIER, M. & ROBLLOT, A.M. (1966) *Annls Inst. Pasteur, Lille*, **17**, 181
- SALWIN, H. (1968) *J. Ass. off. anal. Chem.* **51**, 281.
- SARKANY, I. & GAYLARDE, C.C. (1967) *Lancet*, **i**, 589.
- SAULMON, E.E. (1966) *J. Am. vet. med. Ass.* **149**, 1691.
- SCHULZ, M.E., VOSS, E. & LEDER, K.H. (1968) *Milchwissenschaft*, **23**, 137.
- SELLERS, W. (1964) *J. Bact.* **87**, 46.
- SHELTON, L.R. (1961) *J. Am. diet. Ass.* **38**, 132.
- SIMMONS, J.S. (1926) *J. infect. Dis.* **39**, 209.
- SMITH, N.R. & DAWSON, V.T. (1944) *Soil Sci.* **58**, 467.
- STEINER, E.H. (1967) Statistical methods in quality control. *Quality Control in the Food Industry* (Ed. by S.M. Herschdoerfer), p. 121. Academic Press, London.
- STEPHENSON, R.M. (1967) *Chem. Engng News*, **45**, No. 43, 7.
- STUMBO, C.R. (1965) *Thermobacteriology in Food Processing*. Academic Press, New York.
- SURKIEWICZ, B.F., GRCOMES, R.J. & SHELTON, L.R. (1968) *Appl. Microbiol.* **16**, 147.
- TAYLOR, W.I. (1961) *Appl. Microbiol.* **9**, 487.
- TAYLOR, W.I. & HARRIS, B. (1965) *Am. J. clin. Path.* **44**, 471; 476.
- TAYLOR, W.I. & SCHELHART, D. (1968) *Bact. Proc.* 107.
- THOMAS, M. (1961) *Mon. Bull. Minist. Hlth*, **20**, 37.
- TUYNENBURG MUYS, G. & WILLEMSE, R. (1965) *Antonie van Leeuwenhoek*, **31**, 103.
- VERNON, E. (1966) *Mon. Bull. Minist. Hlth*, **25**, 194.
- VRIES, P.J. DE & KNAPE, M. (1964) *Ned. Tijdschr. Geneesk.* **108**, 1302.
- WAART, J. DE & SMIT, F. (1967) *Lab. Pract.* **16**, 1098.
- WAART, J. DE, MOSSEL, D.A.A., BROEKE, R. TEN & MOOSDYK, A. VAN DE (1968) *J. appl. Bact.* **31**, 276.
- WILLIS, A.T. & HOBBS, G. (1959) *J. Path. Bact.* **77**, 511.
- WILSON, G.S. (1955) *J. appl. Bact.* **18**, 639.
- WINTER, C.E. & SANDHOLZER, L.A. (1946) *J. Bact.* **51**, 588.
- ZEHREN, V.L. & ZEHREN, V.F. (1968) *J. Dairy Sci.* **51**, 645.
- ZEN-YOJI, H., SAKAI, S., TARAYAMA, T., KUDO, Y., ITÔ, T., BENOKI, M. & NAGASAKI, M. (1965) *J. infect. Dis.* **115**, 436.

Organoleptic qualities

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Introduction

The five primary senses are, based on Aristotle's theory, sight, hearing, touch, smell and taste, of which smell and taste are the most primitive. In man, taste and smell respond to chemical stimuli and while man is primarily sight guided in his search for food, dogs, pigs and other animals are guided by scent. When considering a food product the sensory characteristics which may be involved in assessment of quality are appearance, odour, taste, feeling factors and sound.

Appearance

Appearance has, as a rule, the greatest initial impact and there are at least two appearances to be considered. These are:

- (1) The actual appearance of the product itself.
- (2) Its packaging and presentation.

It must be borne in mind that a food product must be attractive enough to stimulate the appetite after its removal from the packet. Appearance encompasses of course, colour, consistency and homogeneity, gloss and sheen, transparency and turbidity. Any one, or a number of these factors can be associated with product quality depending upon the type of product and its presentation. For instance:

- (1) In canned meats the outer surface of the product is important. The questions are asked is it slimy, dark in colour, are there deposits of fat, etc.?
- (2) In polythene packed products, i.e. bacon and cheese, are there droplets of moisture inside the pack?
- (3) In beverages is there a sediment, e.g. who likes cloudy beer?
- (4) In the case of canned fruits and vegetables, is the colour of the product a good colour?

Obviously certain criteria are more relevant than others depending on the type of product under question.

Some of these factors of course cannot be controlled at factory level and often undesirable characteristics develop after the food product has left the place of manufacture.

Colour

Colour is a topic which merits a more detailed discussion. Discolouration and fading of colour can often be accompanied by changes in odour, taste and texture which in turn can be associated with reduction of food quality. Even though a change in colour may not mean an inferior product, consumer acceptance can be affected. A number of workers obtained results which indicate the importance of colour even if there is no change in flavour. In 1956 Foster stated that food selection and judgement of food quality would be very difficult if there were no differences in colour, even though the size, texture, shape and other characteristics remained the same.

It is difficult to find a method of quantifying food colour. We have all had the experience of trying to colour match by memory only to find that we have not carried the colour well in our minds at all. The human eye is able to make qualitative discriminations to a great extent but it cannot act as a quantitative instrument.

In spite of this however, a great deal of information concerning a product can be obtained from its appearance as this is also the case with the odour.

Odour

Like the appearance, the odour of food products can attract or repel consumers and odours are, therefore, of prime importance to the food industry. There could possibly be more discussion concerning olfaction and the sense of smell than with any of the other senses and yet perhaps least is known about this sense. Smell is more complex than taste and more highly developed. It has been shown that the olfactory organ can detect dilutions of alcohol 24,000 times greater than those required to stimulate the organs of taste. The human subject is a remarkable instrument when dealing with odorous materials and extremely dilute concentrations can often be identified by the human nose and yet remain undetected by conventional chemical and physical methods. With training, odours can be recognized at very low concentrations and the nose can distinguish several thousand different odours. Because the method of odour perception is not completely understood, descriptive terms of odours are subjective in that there is no one definition of an odour.

Sagarin defined an odour as 'The property of a substance or substances perceived in the human and higher vertebrates by inhalation in the nasal or oral cavity, that makes an impression upon the olfactory area of the body and that during and as a result of such inhalation is distinct from seeing, hearing, taste and feeling and does not cause drying, wetting or other functions foreign to the olfactory area.'

Apart from indicating whether odours are weak or moderate or strong, just as colours they cannot be measured quantitatively to any extent. Problems encountered in food manufacture have included:

- (1) Odours associated with the use of preservatives in food, e.g. sulphur dioxide, benzoic acid.
- (2) Production of an 'off' odour due to vitamin fortification.
- (3) Development of an odour due to pesticide and fungicide residues.

As change in colour can be a warning that the quality of food products is changing so can the change of odour, e.g. the rancidity of fat, the denaturation of protein.

Taste

The sense of taste is comprised of four basic tastes: sweet, sour, salty and bitter, and these are perceived by specific areas of the tongue, sweet at the front, sour and salty at the sides, and bitter at the back. When we taste, we taste in solution and therefore the salivary glands play an important part in the mechanism. Chewing stimulates the secretion of saliva as do the stimuli of thought, sight and odour—hence the well-known expression 'it makes my mouth water'.

The sense of taste which is being considered here is actually the taste as perceived by the tongue and not the general taste which includes the senses of smell and feeling. As early as 1592 tastes were classified into nine categories viz. sweet, sour, sharp, pungent, harsh, fatty, bitter, insipid and salty. In 1754 Linnaeus added astringent, viscous, aqueous and nauseous. Wundt, however, the founder of the first experimental psychology laboratory, described the four which we now take to be the basic tastes and his work has been corroborated by more recent workers.

Dependent on their concentrations the four basic tastes have different degrees of acceptability. The pleasantness tends to increase with increasing concentration until a limit is reached and then it decreases.

Workers have shown that:

9% sugar (0.263 M)	= pleasant
0.28% tartaric acid (0.0186 M)	= pleasant
2% sodium chloride (0.324 M)	= pleasant
0.0007% quinine sulphate (0.000009377 M)	= pleasant

When considering mixtures of tastes, the reaction is not predictable, whereas with colours neutralization occurs on mixing two colours. This does not apply with taste, although one taste can help counteract the harsh effects of another, e.g. sugar in tea and coffee counteracts bitterness, and sugar in lemon juice counteracts sourness and astringency. In a mixture of two tastes, if one is strong and the other just above threshold level (and by threshold level is meant that level below which the taste would not be detected) then the one just above threshold will not be detected at all even by the most sensitive person.

Measurement of taste thresholds has been performed on numerous occasions and there are two threshold values. These are:

- (1) The detection or sensitivity threshold which is the level at which a difference

is noted, for instance from water, and

- (2) The recognition threshold which is that level of concentration at which identification is made.

Hunger studies have shown that the greatest sensitivity to the basic tastes exists at about 11.30 hours with a decrease in sensitivity 1 hr after a meal. Effects of age, smoking, lack of sleep, temperature and the taste medium on threshold levels have all been studied and should be investigated when work is being prepared on threshold levels.

Feeling factors encompass the texture of the product and the actual sensations produced in the mouth. Astringency produced by alum, the cooling of menthol, the burning of black pepper due to its piperine content, the tooth coating of rhubarb due to the sequestering action of oxalic acid and the calcium of the teeth, are all examples of feeling factors. These combine with the taste and aromatic or volatile components to produce the overall flavour of a product.

Sound

The last sensation to be mentioned is hearing and it is as well to remember that sounds can be associated with both the preparation of a food and its being consumed. Perhaps what is best said is that the sound should be appropriate. We are all familiar with the snap, crackle and pop of certain cereals and associate this with a healthy crunching sound as we chew the product.

Having mentioned the sensory properties which may be associated with food, the question is how are the appropriate requirements maintained in food manufacture? This leads directly to the question of taste panels, whether it be an organized taste panel group or a single taster checking the product of each batch against the previous batch to ascertain whether there is any change in the product. The choice of taste test employed depends on the situation and the type of information required but it is true to say I think, that the purpose of all testing is to establish differences and similarities. Laboratory panels can provide answers to the two general questions relating to the sensory properties of food, viz.:

- (1) Is there a difference?—and if a difference does exist
- (2) What is the nature and intensity of that difference?

Considering the first question, Is there a difference?; we find that there are a number of established tests designed to answer this question and the results of such tests are straightforward enough. Either a panel were able to discriminate or not. However, by using one or two people, it may be just by chance that a difference was noted and a statistical approach is necessary in order that correction for a chance result be made.

Test methods used in difference testing range from the well-known triangle test where a test consists of three samples, but only two products so that the panel is given three coded samples, say two of sample X and one of sample Y and is asked to identify the different sample having been told that two are the same. Since there are three

unknowns the chance probability is one in three. Another method is the paired test method where the subject is given two samples and is asked to select one of these two according to a predetermined criterion, such as sweetness or saltiness. In this case there are two unknowns and the chance possibility of a correct answer is one in two. Other types of difference tests include the duo-trio test which is a variation of the paired test method as is the duel standard method. These methods are designed to establish whether or not differences exist between products and standards and deal with small differences. Apart from indicating whether or not small differences exist, an expert panel is often used to evaluate the overall quality of a food. Quality has been defined as a composite response derived from all the sensory properties of a specific food, that causes it to be judged superior by users who have been exposed to a random selection of the product over a period of time. Often the absence of undesirable characteristics is taken as an indication of whether the quality is good or not. In order to obtain reliable results, expert panels must be used. These consist of personnel who have been selected on the basis of their sensitivity and who are familiar with the procedure being used.

Ranking, scoring, descriptive analysis, dilution and hedonic scaling procedures are all used in the definition of quality. Ranking and scoring test are generally employed:

- (1) When there are more than minor differences in a specific quality factor of a given food product.
- (2) When a large number of samples is to be examined.
- (3) When a combination of quality factors is to be evaluated.

As previously stated these are not the same as the difference tests which have been mentioned, as these are concerned with relatively small differences between samples and in establishing statistically the extent to which the samples may be considered to be alike or dissimilar. Ranking in its simplest form requires the panel to arrange a series of samples in an ascending or descending order of intensity based on a specific taste, odour or flavour characteristic. It is in fact a comparison technique wherein a series of samples are rated against each other. For effective use of this method it is essential that the panel are especially familiar with all aspects of the sample characteristics under consideration at the time of testing. Advantages of ranking include the fact that it is one of the fastest methods of sensory testing and it allows the testing of multiple samples. However, there is confusion sometimes as to how best to treat the data resulting from ranking tests.

Scoring is the most frequently used of all sensory testing systems and consists of evaluating, on a pre-established numerical basis, single or combinations of quality factors of a food product. For the purpose of scoring, the quality of many foods can be broken down into three categories.

- (1) Appearance factors including size, shape, colour, gloss and consistency.
- (2) Flavour factors which are a combination of taste, smell and feeling factors.

(3) Kinaesthetic factors including sensation-giving effects.

Examples cited in the literature include a ten-point scale for evaluating acidity and the flavour of edible oils, a seven-point scale for the crumbling of cheese and other variations include score values: 1 to 3, 1 to 5, —5 through 0 to +5, and —4 through 0 to +4. The first and probably the oldest application of scoring is in the field of dairy products. Before any effective scoring programme can be established for the evaluation of one or more quality factors of a given food product there are certain pre-requisites to be met. These are:

- (1) A realistic scoring card must be developed with the factors properly weighted in respect of their importance so that a product receiving a high score will necessarily represent one of high quality.
- (2) The panel should be familiar with all aspects of the product under consideration.
- (3) The scoring scale should have significance so that a difference in the score will reflect a detectable variation in the factors being scored.
- (4) Agreement must be reached between judges as to standards of perfection so that the entire scoring range can be utilized. This precept is not observed in the scoring of dairy products where flavour is never given a perfect score.

Descriptive sensory analysis is becoming used more and more in studies relating to the quality of food. The best known method of descriptive analysis is the Arthur D. Little Flavour Profile technique. The procedure of the profile method is to make use of a panel of four to six persons working in standardized fashion in surroundings which are conducive to concentration. All glassware and utensils are odour free, each panel member approaches his sample aliquot in the same way and in the case of a hot product, aroma and taste measurements are made over the same predetermined temperative ranges. Each panel member independently examines his sample and his findings are tabulated and later summarized by the panel leader. Then the session is open for discussion and ideas are exchanged and language and vocabulary differences are resolved. Those which are not resolved are sorted out for re-scrutiny at future sessions and in order to aid description, standards are brought in by the panel leader so that panel members can refer to these and so aid their descriptive terms.

The flavour profile of any product is given therefore in terms of:

- (1) The individually recognizable character notes.
- (2) Their intensity—whether weak (1), moderate (2), or strong (3).
- (3) The order of their perception.
- (4) The after-taste which is important in any food product, as even a very pleasant product which leaves an unpleasant after-taste in the mouth can influence a consumer against buying the product.
- (5) The amplitude or initial overall impression.

The definition of flavour profile is that of a semi-quantitative descriptive analysis of odour and flavour. Semi-quantitative because in any product there is a combination of a basic blended flavour complex and separate character notes which confer interest

on the product. Flavour profiles may be used to study processing changes. We have recently studied flavour changes of soups and confectionery products made by different processes and have completed work on beer.

Utilizing the principle of the flavour profile descriptive method and a dilution procedure, Professor Tilgner characterizes the sensory properties of food qualitatively, quantitatively and sequentially. The dilution technique is said to establish the smallest amount of unknown that can be detected when it is mixed with a standard material and resembles the dilution procedures which are used for determining taste thresholds.

Dilutions of dried egg in whole fresh egg, roast beef in water, and dried milk in whole fresh milk have been studied as have spices, margarines and syrups. Although the dilution of food samples can reveal components which are masked in the composite, it must be remembered that the dilution approach may not give a true positive picture of food as it is normally consumed. Also the texture of the food, which in many cases plays an important part in the conception of flavour, is not taken into consideration using this method.

Hedonic scaling relates to the psychology of pleasurable and unpleasant states of consciousness. States of like and dislike are measured on a rating scale in which the choice of words or phrases used is of great importance. It has been shown that a nine interval scale between the limits, 'like extremely' and 'dislike extremely' is more sensitive to differences among foods.

The hedonic scale rating reflects the reactions of a group of people towards certain food products and has been used: (1) to detect small differences in the degree of liking for similar foods, and (2) to reveal differences in group preference attitudes in questionnaire studies. This is a useful method for measuring consumer acceptance, but it is not designed for following quantitative changes in food.

Theme: How are the desired qualities achieved

3rd Session. Chairman: LORD TRENCHARD

Control of raw materials

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THE BASIS for the control and maintenance of the quality of any manufactured food product is the specification for the ingredient. A specification is defined here as a document giving a detailed description of the material, its origin, how it shall be packed, its organoleptic properties, the limits for certain characteristics (desirable and undesirable) and their methods of measurement, the sampling scheme for deliveries and the method and maximum period of storage. These details will give information in respect to identity, genuineness, purity and conformity to the official or recognized standards. A specification provides a common ground for commercial negotiations between the manufacturing company and suppliers of the commodity and as such must be written, as far as possible, in unambiguous terms which leave no doubt as to what standards are expected by the purchaser. Before it is possible to stipulate a number of the characteristics in a specification, it is necessary to carry out development work. Most manufacturers in the food industry will consider themselves to be experts in the field of their particular products but they would not claim to have that detailed knowledge of each ingredient which the specialized supplier will possess. Hence, in this development work there must be an exchange of views and ideas between the two 'partners' in the operation, the supplier and the user. Frankness in the discussion should extend even to the proposed storage on the user's premises about which the supplier will be able to advise.

First and foremost, the manufacturer must know what he expects from an ingredient. If he is a bread manufacturer, he will want a flour with a low α -amylase content. He may also make biscuits and the flour for this purpose has to be one with a low protein content, the gluten having the physical characteristics of distensibility. There are many types of egg on the market and it is important to distinguish between those suitable for the manufacture of salad dressings and those for other purposes. The making of marshmallows and meringues will require an egg white with good whipping properties. For salad dressings, the function of the egg is to stabilize the oil-in-water emulsion and the solubility of the protein is an indication of this characteristic. Dried whole egg can contain glucose which, reacting with the protein in the Maillard reaction, may give rise to off-flavours in a finished product. The manufacturer will, therefore, need to specify that glucose has been removed by the use of glucose oxidase. The food industry uses starch for many different reasons depending on the product being made. A manufacturer will know that the common starches contain amylose and amylopectin, both

fractions having distinct properties which will influence the gel in the product. Besides being of high microbiological quality and free from foreign odours, the canner will want a starch which will provide the viscosity required at the time of filling, but which will not change physically during the shelf life of the product. A specification for a fruit or vegetable will detail the acceptable limits for size, colour, texture and freedom from certain defects and blemishes.

Certain raw materials will need to be tested microbiologically for any Public Health hazard as, for example, *Salmonella* in dried egg. Total counts, presence of organisms of faecal origin, insect fragments and rodent hairs will give information on the hygienic nature of the processing. In the canning industry, the kinds of organisms present and their number are important. The final sterilizing conditions for the manufactured product have to be sufficient to render it microbiologically safe and, at the same time, organoleptically acceptable. Any large increase in the bacterial population of a raw material will require additional heat treatment but the resulting product may be unacceptable by being over-cooked and unpalatable.

Most food manufacturers have to purchase a number of ingredients from abroad. To obtain the quality required, it is frequently necessary to visit the source of supply. Import agents can arrange these meetings, the results of which are of the utmost benefit to the exporter, the agent and the user. If the ingredient is one which itself has to be processed in the country of origin as, for example, a fruit concentrate, a manufacturer should specify that the containers shall be marked to give information on the batch number, the date of manufacture and name of the processor. However, it is not always possible for reasons of expense to visit the foreign producer and then the manufacturing company must rely on an examination of buying samples. An undertaking must be sought that all materials offered conform to the United Kingdom Food and Drugs Act and our regulations relating to the sale of foods. Those manufacturers not having their own laboratories make use of consultants in order to obtain confirmation that the quality is to an acceptable standard. The buying sample together with the information given thus acts as a specification.

Many of the larger companies use a system of inspection of supplier's premises where the ingredient is being prepared and this applies to both foreign and U.K. processing. A knowledge of the management of a factory, the efficiency and the 'climate' of quality existing in the organization can provide valuable support to the evidence from the local laboratory's spot checks in assessing the quality of the processed raw material. Another way of ensuring that specification quality is met and maintained is to use the system of forward contracting for crops of vegetables and fruits. The contracts are made between growers and manufacturers on the understanding that a particular strain of seed is grown and that certain principles in agronomy are followed in relation to crop rotation, manuring, spraying and harvesting.

When the final specification for a raw material is written, it ought to be a realistic document resulting from the discussions between the suppliers, the agents and the

purchaser. It may be a tentative one to commence with, which can be modified in the light of experience. Those who have entered into commercial agreements with a manufacturer will have agreed on a method of packaging and they will understand the technical examinations involving chemical, microbiological and physical considerations. An agent, too, should be fully conversant with these details because, in the event of a dispute arising from a complaint by the buyer, he may be called upon to represent the interests of the ingredient processor. The specification will describe the method of sampling on receipt into the purchaser's premises and it will be appreciated that the objective is an acceptance of every delivery. It is not in the interests of the food manufacturer or the ingredient processor if deliveries have to be rejected because a requirement agreed upon has not been met. From time to time difficulties can arise with a supplier which result in the delivery of a substandard ingredient. To recognize the deviation from the specification and to take appropriate action before the material is incorporated into the manufacturer's own processes is the application of true quality control. The faults which are so often found are connected with contamination with a non-toxic foreign material. Sieving, grading, metal detection and gravity separators, however, prove their usefulness in raising the standard of quality of the ingredients. In other cases, the microbiological condition may be such that it is necessary to re-grade the ingredient so that its use is confined to a restricted number of products but if this is not possible, rejection is the only course.

To manufacture a food product to a constant standard of quality requires rigid control of factory processes. All the operations will have been detailed on the basis that the ingredients being incorporated into the recipe have qualities within acceptable ranges. If a manufacturing process has to take into account a variability outside the agreed specification, then it becomes necessary to include in the chain of operations an additional process, the purpose of which is to rectify the substandard material. This operation will need equipment and labour, the cost of which must be included in the product costings.

As more companies manufacture their products by continuous processes, there will be increasing emphasis placed on rigidity of specifications which demands supplies within agreed quality limits. By working closely with suppliers, the food industry is beginning to enjoy the benefits of accredited supplies. In this scheme, the supplier will declare his analysis and grading on each delivery which, when compared with the specification, will allow immediate acceptance and usage. However, even in the best circumstances of good relationships between supplier and user, the manufacturer will want to have at some point a 'barrier' before the ingredient is blended into the Company's recipe. The 'barrier' could be in the form of a screen, a gravity separator, a magnet or a visual appraisal of colour. These act as true barriers in that an operator could stop a process continuing if there was evidence that the material was substandard.

There is another aspect in the control of raw material quality which has assumed importance in the last several years. Many food manufacturers find themselves short

of that labour which is so vital in the pre-processing of the ingredients. The peeling and trimming of vegetables is a good example to quote. Once again it is necessary to develop with a supplier situated in a favourable locality where labour is at hand a specification giving the exact details of the quality desired. The specification needs the minimum of tolerances and has to be of the same standard as that in operation in the manufacturer's own factory. The exchange of supervisory staff is sometimes useful in achieving this objective. Similar operations are being used by the industry for the preparation of cuts of meat, the sorting of fresh and dried fruits and the cleaning of cereal grains.

The practical operations of receiving the raw materials into the user's premises involve the taking of samples for laboratory checking. The purpose of any inspection scheme is to prevent substandard raw material being accepted. Since 100% inspection is rarely feasible, methods have to be worked out which will ensure that the quality can be evaluated with maximum reliability but at a cost which is commercially reasonable. Not all ingredients can be purchased in bulk containers or under the accredited scheme and the examination of materials in casks, in sacks, in tin containers, is now being approached statistically by many companies. The terms 'reduced level', 'normal level' and 'tightened level' are freely used and these refer to the extent of sampling which the manufacturer operates. With suppliers with a good history, a manufacturer may feel justified in examining each new consignment less extensively than deliveries from other suppliers.

Statistical sampling schemes can ensure that those tests which are made are sufficient to give a good assessment of quality of the ingredient. However, the greater the protection needed to prevent substandard consignments being accepted, the greater the amount of sampling required. Good facilities must be available where the initial inspection is made. Many companies have arranged that each consignment arriving by road passes immediately to a checking building. Simple physical tests can be carried out before off-loading is authorized. Such tests are particularly useful for fresh vegetables. Garden peas can be checked for texture using a tenderometer and carrots and potatoes can be graded for size, washed and inspected for blemishes and excess soil. Cereals can be sieved to look for insect contamination and oils tested for free fatty acids and peroxide values. Conductivity moisture meters give quick measurements of moisture, hydrometers measure salt concentrations and the amount of soluble solids can be obtained from refractive index readings. The examination of the trade vehicle carrying the raw material is equally important. Any evidence of visible contamination can be dealt with immediately. If laboratory tests are required, authorization can be given after visual examination of the consignment for unloading when it will then await further checks.

It must be recognized that where specifications detail subjective characteristics, difficulties can arise between supplier and user. This is particularly so with deliveries of farm vegetables where the degree of blemish is not always easy to measure. Similarly,

the assessment of fresh meat for the amount of 'visual lean' requires a great deal of experience. Efforts are being made to correlate these and similar characteristics with laboratory measurements as, for example, the amount of fat present in meat as obtained by solvent extraction compared with the 'visual lean' assessment. For commercial reasons it is desirable that the quality testing of ingredients is carried out speedily. Much of the checking is carried out using the simple physical equipment just described and the results can be related to the more detailed tests performed in a laboratory. The quick maturometer test for peas bears a simple relation to the more lengthy alcohol-insoluble solids content, the conductivity moisture meter can be equated to a four hour oven drying operation and the Lovibond Comparator measurement of colour can be compared with the Macbeth–Munsell Disc Colorimeter assessment. There is no rapid factory quality control method for metallic or similar contamination so these need to be checked from time to time by a laboratory not governed so much by time.

The operation of storing the ingredients will have been borne in mind when the specification was developed. Multi-walled paper bags have replaced most of the hessian sacks used in the past with the result that the contamination hazards of bag fibres have been removed. In addition, the use of fibre board for the packing of many items of food raw materials is now extensive. Cartons for meat and dried fruits and 'chips' for mushrooms are just a few examples.

All cool and cold storage areas should be equipped with automatic temperature recorders. To inspect these records for abnormal running and then to pay particular attention to the store contents when abnormality shows, is just part of the eternal vigilance possible from the well-trained member of the quality staff. All areas of storage will have frequent inspections by trained personnel to ensure freedom from pests. In addition to the traditional methods of spraying, baiting and trapping, there are devices on the market which many companies are using or experimenting with ranging from electrocution of flying insects to ultrasonic bird scarers. The maintenance of a high standard of visual cleanliness and tidyness not only helps in keeping down rodents but encourages employees to use good working habits. Structural repairs and redecoration are necessary from time to time and it is generally known that the quality of stored ingredients can be harmed if strong smelling paints are applied in their storage areas. On this matter of structural maintenance, it is good policy to ensure that any building contractor working on premises where foodstuffs are stored or processed is made aware of his responsibilities. The provision of areas for smoking and meal breaks is essential for his staff and he, in turn, must always ensure that the working area is tidy and the site well cleaned when the work has been completed. There can be one more stage before the ingredients are taken into the processing kitchens for inclusion in the Company's recipes. This is the transfer to an area where they are unpacked. Here wooden boxes can be opened, cartons and paper wrappings removed, casks emptied, sack contents sieved or sorted into bulkier metal containers. This pre-treatment in the control of raw materials is most important in reducing the risk of contaminants being found in

the finished product.

The need for realistic specifications has been emphasized, but it must be understood that rigidity must not hamper considerations of these same ingredients with different characteristics or in some other form.

In all companies there is a continual pressure to work more efficiently, to modify a process or to use a more common form of an ingredient in order to reduce the overall costs. The resulting savings when passed on to the consumers must benefit all concerned. The work of the chefs in the experimental kitchens of the food industry has been interpreted during the last decade into chemical engineering processes. These processes demand an unvarying standard of quality in each of the ingredients being used. If in the course of experimental work it is thought that an ingredient in some other form would give advantages, it becomes necessary to redevelop the specification. In the more progressive companies in the industry, it is acknowledged that the life of a specification can be short in the face of the buyers, the food technologists and the chemical engineers all asking why this material in this form at this price is being used. As we in the industry know, all the scientific staff is not to be found on the premises of the manufacturing company—the suppliers have them also. It is with their help, their specialized knowledge, that the manufacturers can control the quality of the incoming materials.

To conclude this brief but broad outline of the steps which are taken to ensure that the ingredients are of the highest quality, it must be stated that a detailed raw material specification will not give on its own 100% safeguard. There has to be a wish by all concerned, from the highest management down to those operating on the factory floors, to produce a quality item. This calls for a programme of training so that the principles of quality are fully understood by all ranks. Training by example cannot be overstressed. If management genuinely wants quality work, it will ensure that the factory buildings are well maintained and well cleaned, that all operatives have the opportunity of wearing a neat uniform which is frequently laundered and that special areas are available for smoking and tea or coffee drinking. Quality then becomes an attitude of mind to production staff in addition to the technicians in Quality Control. With medical advice and supervision, assurance is being given that the health and habits of the food handlers are to the highest possible standards. The meaning of quality may differ from supplier to supplier, manufacturer to manufacturer, country to country but personal contact, free and frank discussions between suppliers, agents and users add to the printed specifications the desire of the food industry to control the quality of its raw materials with the objective of satisfying the ultimate customer—the housewife.

Plant and process control

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PLANT and process control is a very wide subject and this paper attempts neither to give a manual of do's and don'ts nor to treat the subject entirely mathematically, but rather to look at the question of how far is process control possible in the Food Industry how it is tackled at present and how this may change in the future.

Excluding the preparation and cooking of foods for immediate consumption as a meal, either at home or in a catering establishment, the main aim of food processing is preservation. The processes used are those of freezing, dehydration, sterilization, pasteurization or the use of chemical preservatives. Preservation may be short term, as with fresh meat and dairy products, or long term, up to a year or more, but the clear aim of processing is to achieve some measure of preservation. The success of the food technologist, particularly in the development of new products, is the extent to which, in achieving preservation, he makes a product wholesome, palatable and attractive to the consumer.

Having defined what is meant by 'Process' it is necessary to consider 'Control'. In the domestic kitchen, the housewife seldom produces exactly the same meal from standard ingredients and a recipe—life would be dull if she did. The end result depends on how she feels, how busy she is, and a number of factors of which she is probably unaware. The food processor, however, has to be always consistent in the way he carries out his processing. He also has to be right first time, as food processing is unfortunately not a reversible reaction. Hence he needs a good measure of Control of the process. How much control can he reasonably expect? and how can he achieve it?

An early and simple example of process control outside the food industry was the use of the steam governor to control the supply of steam to an engine that must run at constant speed. Control systems have since become much more sophisticated, particularly in the chemical industry, and the solution of control problems often requires computer analysis of a model set up to show the various inputs and outputs to the system. A topical example of very good process control must surely be that of putting a spacecraft onto the moon. The inputs into a control model of such a system would presumably include forces of gravitation, forces of propulsion derived from chemical fuels, measurement of distances and mass, stress and heat resistance of materials, etc. All of these are complex, but ultimately definable; the fact that space shots are successful shows that they must be.

Consider now a typical model for a food process (Table 1). It is not suggested that this is a comprehensive list of inputs and outputs, but it serves to illustrate the problem. If all the inputs were capable of definition and quantification, a series of control loops could be constructed, each with a transfer function and the model optimized for a required quality of product. Equally it could be optimized for any level of profit, capital or operating cost, labour or yield, so that the result would be, and indeed is, a compromise. However this model cannot, in most cases, be subjected to a complete analysis because of lack of understanding of the inputs or because they are biological, and therefore to some extent undefinable. Thus, inputs 1 and 3, the biological raw material and the biological human factors are the main difference between food processing and, say, the chemical industry. However, with this limitation, considerable control is possible and the various inputs will be discussed in turn. It is in the first place clear that management has considerable choice in selecting the level of inputs. It can for example use a low quality raw material and attempt to upgrade it, or can pay more for a better raw material. Similarly it can choose between a labour intensive process or a capital intensive one, probably a continuous one and making maximum use of automation. If the latter is properly understood it will give a tighter potential control of quality.

TABLE 1. Model of a food process

Inputs	Outputs
1. Raw material food sources containing: <ul style="list-style-type: none"> (a) Desirable protein, carbohydrate, fats, etc. (b) Impurities, including bacteria 	1. Product defined by: <ul style="list-style-type: none"> (a) Organoleptic properties (b) Physical and chemical measurement (c) Microbiological tests
2. Chemical ingredients, e.g. salt, water, sugar, acid-alkaline buffers, preservatives	2. By-products, e.g. <ul style="list-style-type: none"> (a) Solid wastes (b) liquid effluent (c) Dirty plant
3. Human effort <ul style="list-style-type: none"> (a) Management (b) Process operatives 	
4. Physico-chemical process (The Unit Operations of Chemical Engineering), e.g. heating, cooling, size-reduction, mixing, mass, transfer, etc.	
5. Process plant, e.g. materials of construction	
6. The package gives: <ul style="list-style-type: none"> (a) Protection (b) Portion control (c) Information 	

Raw material food sources

Many processes use a relatively perishable fresh raw material and in most cases little modification or control of uniformity is possible once these are received at a factory. Process control must, therefore, extend back to the producer, so that quality on receipt at the factory can at worst be predicted, and at best be controlled, at a uniformly high level. Ultimately this is factory farming and to some extent it is with us already. Thus in fresh vegetable processing many crops are grown on contract and the processor controls the seed, cultivation methods, harvest date and frequently the method of harvesting. He cannot control the weather, but frequently insists on irrigation and determines when it should be applied. Meat processors are increasingly applying research in animal breeding and animal husbandry to procurement of the most suitable animals. In particular much more attention is being given to the ante- and post-mortem conditions. Fish farming is another interesting possibility for the future.

Some fresh raw materials can be modified by storage to a more uniform feed-stock. A well-known example is that of potatoes used in potato crisp manufacture. It is essential to avoid a high reducing sugar content in the raw potatoes at the time of processing, or unpleasantly dark brown crisps result. If the processor has control of the growing of the crop he can usually ensure low reducing sugar levels at harvest. He then has the choice of relatively sophisticated storage which will maintain a low sugar content until the time for processing, or he can use normal (low temperature) storage, accept a high resulting sugar content and bring this back to a low level by short time high temperature conditioning. There are of course subsequent steps in the process which can correct for a high sugar content, e.g. leaching of sugar from slices, or use of microwave final drying, but there is surely a better chance of good quality control of the product by ensuring a uniform starting material, than by applying subsequent additional process steps.

Water

Water is a universal input of many food processes, and not merely those of brewing or soft drinks manufacture where it is the major ingredient. It is wrong to regard water used in a process merely as a service like electric power, as water can directly influence product quality. Thus vegetables may be toughened when cooled in hard water following a heat treatment. There is sufficient calcium to form cross linkages in cell walls, particularly if heat treatment has resulted in demethylation of some of the pectin. Similarly the colour of blanched green vegetables is slightly greener when cooled in a hard water than in a very soft water. In the latter case there is insufficient reserve of alkalinity to offset the natural buffering of vegetables at about pH 6.5 and some of the chlorophyll reverts to olive-brown phaeophytin. Traces of copper in a process water can adversely affect the colour of cured meat products and a very soft water is corrosive to some materials of construction, and this can obviously influence product quality.

Human effort

This is one of the variable biological inputs. The administration of quality systems is worthy of being the subject of a separate paper, but there is no doubt that every operator in a food plant has a vital responsibility for quality control and most of all the line manager. He is the real Quality Controller, though he is not usually so named. Other people are checking quality, but he is controlling it. One of the more neglected areas is training in hygiene and sanitation. Not so long ago the dismantling and sanitation of plant was something that took place in the middle of the night and was often left to chance. The importance of clean-up schedules and training in their use is fortunately now increasingly accepted.

Physico-chemical processes

The problem in chemical engineering terms of many food processes is that they involve simultaneous heat and mass transfer, e.g. frying, cooking, drying and concentration. Which property of many should be monitored? The answer requires a detailed understanding of the process before a suitable property, capable of measurement, and truly representing the process, can be selected for control purposes. Thus vegetable pieces, air-dried in a tunnel system, are susceptible to scorching. Experience may show that control of the inlet air temperature below a given value is effective. More detailed study of the process, however, shows that scorching takes place near the exit of such a drier, when the air is nearly saturated and the temperature of the part-dried pieces, which is the wet-bulb temperature of the air, approaches a critical scorching temperature. With this knowledge a good quality control can be achieved using the wet bulb exhaust air temperature to control inlet air temperature and at the same time take care of fluctuations in material input.

When the heat transfer medium remains in the system (unlike air drying when it is vented) there is the problem of progressive change in composition of the medium due to mass transfer from the product. An example here is in water blanching of vegetables. The composition of the product is different at the start-up of such a process when the rate of leaching-out of solids is high, than when equilibrium has been reached, after the process has run for some time. Fortunately the organoleptic difference is usually barely detectable.

When the heat transfer medium is itself subject to chemical change, mass transfer into the product is possible, and the system is even more complex. Such a process is that of dehydration by frying in oils. Frying oil in an atmospheric system oxidizes progressively with time and oxidation products in the fried material are precursors of rancidity during storage. Hence in addition to the obvious control of temperature and residence time of the process for required product properties there must be regular monitoring of oil quality, each level of which has to be correlated with storage life of the product.

Continuous processes

In theory a continuous process should be capable of better quality control than a batch process. One area where good results have been achieved is in the sugar boiling of sweets. This is partly because sugar is a relatively pure, and hence definable, raw material and also the process is well understood.

The full exploitation of continuous processing requires a much greater knowledge both of physical constants and process monitoring than is necessary with batch processes. Mr Pearson has already made a plea for determination and publication of physical constants such as specific heats, rates of heat transfer and rates of diffusion. BFMIRA have recently commenced a study of methods for process monitoring. Already we can make in-line measurements of temperature, pH, viscosity and flow rates and can divert aliquots from process streams through continuous chemical analysers. The rapid in-line determination of moisture content is more difficult, but none the less important.

The results of such process monitoring can be fed back to effect quality control of the process, again assuming that sufficient is known about the system. There may, however, be a hold up in the system. This may represent a considerable amount of different quality, possibly substandard material before the system responds to a signal. Surely it is better to feed forward, i.e. to recognize a change in the input to a system at the earliest possible point rather than monitor near the end of a process. Areas where feed-forward is urgently needed are those where changing bulk density of product is incompatible with the fixed dimensions of a package. A typical illustration is in biscuit manufacture. Much has been said already about the peculiar weights, e.g. $6\frac{1}{2}$ or $7\frac{1}{2}$ oz of biscuit packages. Having determined a pack size and being constrained by law to declare a packet weight, the manufacturer in fact has little tolerance. He will package a fixed stack height of biscuits, i.e. a constant volume or otherwise the packaging plant will jam. If a process stream of biscuits has risen too much, the entire hold up of a travelling oven may have to be diverted until a correction is applied possibly to oven temperature or residence time. If, however, some meaningful change in property of the original dough could be measured, possibly a rheological or moisture measurement, the necessary plant corrections could be made in advance and production wastage avoided. This is surely an area of plant and process control offering a real challenge to food science and technology.

Packaging control

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Introduction

In the paper which I am presenting, I shall try to distinguish very clearly between the measurement of quality and quality control. I believe that the best results are only possible with the full realization that the control of quality for any type of product is a team operation involving everyone concerned from the raw material to the ultimate consumer. So called quality controllers can only operate by providing a quality measurement service on which managers and operators can make decisions. Secondly, someone previously raised the question of cost, stating that quality control costs money and many smaller firms cannot afford this expenditure. I think there is again a misconception of terms here. What was really meant was that formal quality measurement carried out on a statistical basis with a special inspection department is often not possible at an economic cost in a small company. Nevertheless, there must be a conscious measurement of quality even if it is done by the production operatives. The sooner it is recognized that the control must be exercised, and indeed can only be exercised, by the men operating machines, the sooner we can get good results. I would add that, no firm can afford not to control its quality—whether it does it on a statistical sampling basis by a specific department is neither here nor there—it should evolve its own method within the limits of the organization. Any progressive company must always have production control decisions based on a predetermined quality standard which is known by everyone and this control will be initiated by information from some form of quality measurement. Many smaller companies are not even aware, at present, that they control their quality quite informally. The provision of a quality measurement service should be as economical as we can make it depending upon the operation and the scope involved.

Successful food manufacturing involves two necessary, inseparable and almost equally important requirements:

- (a) Producing a good product which is both nutritious and tasty.
- (b) Delivering that product to the consumer in as near perfect a condition as possible.

The first requirement is in the realm of food science. The second is almost entirely packaging. It is perhaps, therefore, superfluous to say that since the first is almost

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entirely dependent on the second, that food manufacturers will only be successful if their packaging is right. It is, however, equally clear that the best packaging in the world will only sell a product to each consumer once and although there are a considerable number of consumers, they have a habit of informing each other about bad products very rapidly.

By way of introduction, I would like to tell you of what I believe is the first recorded instance of quality measurement having been carried out. I refer to the records in the *First Book of Genesis* where verse 4 records that the work specified in verse 3 was inspected and passed inspection for good quality. Moreover, there was a sorting operation involved as there is frequently today. The verses are:

Verse 3: 'And God said, let there be light and there was light'.

Verse 4: 'And God saw the light that it was good, and God divided the light from the darkness.'

This particular piece of quality measurement and control was undoubtedly well worth while. Since that time complaints about light have been few. In marked contrast to this, the work specified in verse 1, has no record of inspection for quality. It states:

'In the beginning, God created the Heaven and the Earth.' There is no record in verse 2, or anywhere in the Chapter, that any inspection was carried out and although no authenticated complaints about Heaven are known to me, people have been complaining about the state of things on Earth ever since.

Preservation and packaging

Almost all foods are perishable. That is to say, after a certain period of time, it is possible to distinguish between 'fresh' goods and goods which have been kept. Thus the changes which have taken place must be regarded as a deterioration. Foods which ripen or mature after being packed can have a period of improvement, but this will then be followed by a deterioration. Once the food has passed beyond a certain stage it can only be sold with difficulty or at a lower price. The deterioration which occurs may be spontaneous, but it is more often a response to external circumstances, and much of the packaging which is used intervenes between the foods and their external surroundings to delay the process of deterioration for the required time. Hence, in all problems concerning food packaging, the first considerations must be about the product itself. The ways in which it can deteriorate must be considered and the influence of transport, storage, distribution and sales conditions on the rate at which this takes place determined or estimated.

The factors causing deterioration of food can be divided into two principal categories:

- (a) Those which are inherent in the nature of the food product and cannot be prevented by packaging alone, and
- (b) Those which are dependent on the environment and may be controlled by the

type of packaging employed.

The first class includes physical changes due entirely to temperature, such as the softening of chocolate at tropical temperatures and the breaking of emulsified aqueous fluids at freezing temperatures. Biochemical and chemical changes due to micro-organisms, or interaction between various components of the food product, such as the 'browning' reaction in meat, are also examples of factors that cannot be controlled completely by packaging. Simple physical changes due to temperature can be partially controlled for short periods with insulated containers as in the packaging and transport of ice cream or by the use of ice as in the packaging of wet fish, but when anything over 48 hr is involved, a temperature controlled transport vehicle is best.

The second group of factors leading to spoilage are four in number:

- (1) Spoilage due to mechanical damage.
- (2) Spoilage due to changes in moisture content.
- (3) Spoilage due to the absorption of oxygen.
- (4) Spoilage due to flavour gain or loss, which includes both the take-up of odours or taints from the packaging materials as well as absorption of foreign odours from the atmosphere outside the package.

It is very seldom that the packaging is required to prevent spoilage caused by only one of these factors, but it is usually necessary to consider them separately before attempting to assess the packaging problem as a whole. It is obvious, for example, that mechanical damage, in addition to physically damaging the product itself, may lower the resistance of the package to moisture, oxygen or foreign odours.

This preservation of a state of freshness is not the only aspect of the food with which packaging is concerned, but it obviously plays a vital part in ensuring that the food remains clean and in a hygienic condition. Indeed, food manufacturers were the first to appreciate the benefits of preventing the adulteration of food by themselves carrying out the operations of weighing out retail quantities, putting them into units and identifying them instead of leaving it to the shop assistant. Moreover, since we do not merely eat food for the goodness and benefit we derive from it, but also for its taste, smell and those tactile sensations which are normally lumped together under the heading of flavour, the packaging must also play its part in retaining the aromatic and volatile constituents of flavour which are normally associated with the food. Also the packaging material must not contribute anything to the food and cause a different flavour from that required. It is undoubtedly true today that without packaging, much of the nation's food materials would either be wasted during distribution or would be less nutritious and hygienic than they are at present.

Packaging concerns materials and/or containers and operations

Packaging is defined in the British Standard Glossary of Packaging Terms (B.S. 3130: 1959) as 'The art of, and the operations involved in the preparation of articles or commodities for carriage, storage and delivery to the consumer.' A more technical

definition is: 'Packaging is a means of ensuring the safe delivery of a product to the ultimate consumer in sound condition at the minimum cost.' These two definitions together with the concise statement that 'A retail package must sell what it protects as well as protect what it sells' give a very adequate concept of the wide functions packages must perform. Practically every operation from the development of the original idea of the product to its use by the final purchaser will have an influence on and in turn be influenced by its packaging.

Early consideration in the marketing of any food product must, therefore, be given to the form of packaging required, or an unbalanced economic situation coupled with possible protective inefficiencies may result. During this consideration the functions the packaging will have to perform must be defined. A package for use in the food trade has to perform a number of primary functions. First of all, it must keep the product clean and provide barriers against dirt and other contamination. It must also provide protection to the food against physical damage, moisture, oxygen and light. Thirdly, it must function smoothly, efficiently and economically during the packaging operation. This means it must either be designed to run on already existing machinery or new machinery must either be bought or hired for the purpose. A further consideration is that it should have a degree of convenience built into its design. This includes not only the convenience supplied to the ultimate consumer, in terms of easy opening, reclosure and so on, but also convenience at the intermediate stages in handling through warehouses and transports during distribution. In particular, the size, shape and weight of the units are essentials. A fifth requirement is the provision of identification, information and sales appeal. The retail units must 'sell what they protect as well as protect what they sell'. Space and copy will also be necessary for such items as recipes or instructions for use as well as the description, etc., to comply with statutory regulations. Finally, in addition to the descriptive matter already mentioned, the package must not contravene the food regulations in any respect. For example, it must not contain undesirable materials within its own construction which might diffuse into or transfer through the vapour phase into the food.

Our first task then, in setting a quality standard for the packaging of any food, is to write down as concisely as possible a description of the package or packaging material required. A specification has been described as 'a statement of the requirements to be met in attaining the objective'. It has two purposes: Firstly, to communicate the requirements to a supplier and, secondly to determine whether or not those requirements have been met. By definition, a specification should be specific. It must also be complete, clear and as unequivocal as possible.

However, a specification can be written with several different motives other than that of communicating to the supplier what is required and providing a basis for the user to check what is supplied. At the conference on *Quality and Reliability in Packaging* last October, one paper listed no less than six different motives for writing a packaging specification. It made the point that a specification is not a document to be brought

out of a drawer when an inquest is being held on what went wrong. It should, in simple terms, state what is required. It should then proceed to provide as much information as possible about how those requirements can be met. Finally, it should give a means of judging, without using all the material supplied, whether the batch is satisfactory for the purpose. That is to say, the document is really a statement of intent drawn up jointly by the user and the manufacturer to cover the points which are essential to the operation. It is extremely important that the specification is right at the beginning, as otherwise this can lead to endless trouble later on. While one does not prevent difficulties by writing a specification, such a statement drawn up by the two parties will mean that they must both have made the effort to understand the other's difficulties. The liaison between technical personnel on both sides will mean that an understanding will have been reached before the document is written and therefore it can be used as a basis for improvement.

The relation between the user and the supplier

The second main point is that packaging will never be effective unless there are good contacts and understanding at all levels between the suppliers of the package or packaging material and the users. This is not necessarily a single relationship between the two sides, but it may be one of a number of relationships from the raw material suppliers to the final consumer. For example, a board maker supplies board to a converter to produce folding box board cartons, which are then supplied to a food manufacturer to pack cakes or biscuits and he in turn supplies a wholesaler who distributes them to a retailer, who sells them to a housewife who opens the package and consumes the product. There are a number of contacts here and each one of them will have a bearing on the packaging requirements. The best marketing operation is always achieved when every link in the chain has been considered beforehand and the requirements of the product from the point of view of packaging have been specified adequately and taken care of at all stages.

While we must not forget that there are a chain of users and suppliers it is necessary for each to repeat every test? Furthermore, it must not be thought that the operation is purely one of buying and selling. In particular, the relation between the packaging supplier and the food manufacturer is rarely just an operation of buying and selling. They can argue, disagree or play poker with one another during bargaining as much as they like, but once it has been decided that a particular food manufacturer is going to buy his packaging material from a particular supplier and agreement to work together has been reached, the supplier-user relationship must become a partnership working to maintain production and distribution of the packaged food at the correct level of quality. To achieve this, one not only has to reach understanding on all the things that happen when the flow of packaging is proceeding correctly, but one must also have worked out in advance what to do if things go wrong. Obviously the particular procedures for delivering cartons or bags, or bottles or wrappers in the

correct quantities at the right time and the right quality level, must be worked out. The tolerances that are allowable on, for example, various dimensions must be agreed upon and the methods by which the package quality will be measured must be fully understood. The procedure for taking repeat samples must be considered in instances where agreement is not reached. Also the actions to be taken if no agreement can be achieved must be arranged before the operation starts. This same 'partnership' arrangement is also necessary between the converter of the packaging material into containers and his supplier of raw material and, at the other end of the scale, the retailers' and consumers' viewpoints must also be taken into account. In such a complex operation, it is easy to see how problems can arise, particularly if specifications are used not as statements of intent, but as weapons for an inquest on what went wrong and if the relationship between various parts of the chain are less than true partnerships.

Inspection, quality measurement and control

First of all, we must ask ourselves, what do we mean by quality and what are the particular aspects of quality with which we are concerned in packaging? Quality can be defined in many ways, but probably one of the best is its definition in relation to 'perfection'. Perfection is something which we can very rarely, if ever, attain and the quality is the distance away from perfection that any particular specimen happens to be. This is the level of quality and must be determined by the particular product involved. It is obviously not necessary to produce a level of quality higher than that required to do the job. The second important aspect of quality (and this is particularly important in terms of packaging standards) is the consistency of quality throughout the job. For example, if we assume that perfection is rated as a figure of 100 and decide that for our particular purposes we need a package at a level of 85, then we want to have all the specimens that are submitted between, say, 80 and 90 and not varying to such an extent that we may have some at 95 or 96 and others as low as 75. In other words, quality in the sense of uniformity must be present irrespective of the level that is required. In setting packaging standards for food, it is necessary therefore to answer these two questions. Firstly, what is the level of quality we need to do the job and, secondly how much variation about that level can we tolerate?

It is, of course, very important to realize that quality is something that is built in at the time of manufacture. Consequently, it is impossible for what is often called Quality Control in a food manufacturing unit actually to control the quality of the packaging material which they receive from their suppliers. They are only able to determine whether the supplier has reached the required standard. In other words they are a quality inspection department. True control of quality can only be done on the machines which are actually producing the packages or packaging material.

There is one possible exception to this where the quality inspection also involves removal of defectives, particularly where re-usable containers are concerned. For

example, let us consider the use of milk bottles. These make a considerable number of journeys before being taken out of service and they must, therefore, be thoroughly cleansed every time they return to the beginning of the cycle, and chipped, broken and other defectives removed. In this instance the inspection which is being done not only checks the cleansing but also sorts out any defective or dirty containers. This type of sorting is best done by machine automatically at the exit to the cleaning cycle. In any event, in such operations where containers are re-used, the presence of special quality inspection problems must be countered by using special techniques.

A similar set of circumstances arises in the re-use of outer containers in fibre board where the containers on return must be inspected to see that they are fit to go out again and if not, removed from the system.

So far, we have used the term inspection on a number of occasions and it is obvious that there are at least two different kinds of inspection. There is the sort of inspection which involves looking at everything and throwing out the rejects and the type which looks at a small quantity and decides on the basis of that small quantity whether in fact the batch is acceptable. Looking at these two possible methods without any other consideration, it would appear, quite conclusively, that the system of looking at everything and throwing out the wrong ones is the best from the customer's point of view, but this is in fact true only where the numbers concerned are relatively small. Immediately the number involved becomes large and, as a general rule when we are dealing with packages they are large, we come up against the problem of not having sufficient time or sufficient pairs of eyes to look at everything and throw out the doubtful ones. Additionally, if one is looking at very large numbers it is quite easy not to be looking at the time when the wrong one comes along and to miss it. A level is reached (which varies according to the numbers involved and the type of inspection required) where it is far better to take a small sample and look at it carefully and in detail than it is to try and look at everything more briefly. The next question which arises if we are going to take a sample is how big should that sample be? The answer to this is related to the cost of inspection. How many inspectors can we afford to use in the operation and what is the minimum number required to give sufficient information to enable us to make a realistic judgement?

Once again, I would stress the two aspects of quality for which we must cater. First of all the level of quality required and, secondly, the variation about that level throughout the batch. Setting a level which is higher than necessary normally means that costs will be increased for two reasons. Firstly the maintenance of the higher quality level on the production machines will mean that more rejects will be produced and, secondly the requirements for a higher quality level will almost inevitably mean that the variation will become more obvious over a smaller range than if the level were lower. This constant stress on uniformity as one of the major factors in maintaining quality is quite deliberate and applies not only to the packaging but also to the food product. Variations in the quality of the food product can have an apparently

adverse effect on the package. Consider biscuits which are to have a shelf-life of 2 months when packed at a moisture content of $1\frac{1}{2}\%$. We are bound to get a variation in the protective quality of the packaging material but the calculations have taken care of this by making certain assumptions about the initial moisture content of the biscuits. Providing the moisture content of the biscuits does not rise above $1\frac{1}{2}\%$ we can arrange that whatever the packaging variation, all the packages in a batch are going to give $7\frac{1}{2}$ weeks shelf-life. If we get a variation on this initial packing moisture content, say a rise in moisture content in some batches to 2% and a fall in moisture content on other batches to 1% , then our shelf-life will start varying from between $4\frac{1}{2}$ weeks to 12 or 13 weeks according to whether the biscuits were moister or drier initially. This sort of variation in shelf-life is often interpreted as a variation in package quality, whereas in fact it is varying product quality which is causing the trouble.

Setting packaging standards

Like most products, packages and containers have aspects which can be measured and other factors which are non-measurable and these must be taken into account in setting standards. We use a system of defect levels (critical, major and minor) and divide the variables and attributes into those concerned with package functions and those concerned with package appearance. Any defect which causes a package to fail in any part of its function is regarded as critical and the aim should be not to deliver such material to the user. As far as appearance is concerned the standard here may vary from product to product and market to market.

It is essential in any package-making operation for all departments in the manufacturing unit and for those sections of the food manufacturing plant who use packages, to have a clear unequivocal target for both the package performance and appearance. Realistic tolerances about the standard and appropriate inspection methods must be set up in advance.

How to make complaints useful

From time to time in any operation, examples will be found of material which causes trouble for one reason or another. Such material is usually the subject of a complaint.

Making a complaint to a supplier always has the possibility of relieving the feelings of the complainant, but in order to make the best use of complaints, it is essential that the information required by the packaging material supplier should be given to him. For example, it is much more helpful to say: 'Here is an example of a box we do not like and we are finding something like 3% of these in a batch of 200 samples that we took at random', than it is to merely send two specimens back saying: 'Please do not send us any more of these'.

With regard to functional complaints, very frequently the particular piece of packaging material or container which caused the trouble has been destroyed in the

machine and the samples which are sent back on either side of it may or may not have been ones which caused trouble and hence the information they give is extremely scanty. One thing, however, can be taken for certain. That is that if sub-standard material which does not function properly is delivered to a food manufacturer, then he will complain and send examples of the complaint back to the supplier. When he does so the inference is 'Please do not send me any more like this' and while such information can be useful in a negative sense, I think we could be far more positive if instead of complaining only, the food manufacturer were also to send back samples when his packaging lines are running perfectly with the statement 'Please send them all like this'. It is much better to know what is required than it is to know what is not. So my final plea is—please send back the good ones sometimes.

Conclusion

In conclusion I would like to stress five things about setting packaging quality standards in the food industry:

- (1) Know the requirements and define them.
- (2) Decide how to 'measure' quality.
- (3) See that both supplier and user have all the knowledge and understanding necessary to work in partnership to the same end.
- (4) Agree standards and tolerances and decide what happens when things go wrong.
- (5) Feed back positive information rather than negative complaints.

4th Session. Chairman: SIR SAMUEL SALMON

Administration of quality systems

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AFTER hearing the previous speakers at this symposium, I feel that the most significant point that was highlighted by all speakers is that quality does not arise by chance. One cannot dismiss the quality problem by saying, 'It will be alright on the day', or, 'Don't worry, everything will sort itself out'. With this attitude one is forever fighting rearguard actions for quality. Today, with the rapid advance of communications and advertising, public taste can change quickly and if the housewife gets a second rate product she will buy something else. The opportunity for choice for the housewife has never been so great. Gone are the days when the local supplier was the only supplier. Distribution systems have advanced to the point where foodstuffs can and are, brought hundreds of miles to the shop in a fresh attractive condition. The housewife has no longer to 'take it or leave it'. She can 'take it, or choose something else—or someone else's'. Products now become obsolescent much more rapidly. By product, I mean the product and the package containing it—the two are almost inseparable these days (and that's not intended as a comment on the difficulty of getting into some of the present-day packed goods!).

All this means that one must get it 'right first time'—to use a cliché. We must plan to get the right quality prior to production and follow it right through to the customer. *Who* follows it through? If the responsibility for quality is fragmented at the stages of raw materials, process, product, packaging and distribution, then a lot depends on each member of the chain first catching the ball and then passing it—and its no use having someone running up and down the chain picking up dropped catches and misdirected passes. This will only add to the confusion when, as there nearly always are, several balls being passed along several chains. I believe that the biggest single bar to good quality is bad communications. Too often individuals are not sure what they are trying to produce (in precise terms), and how, (in precise terms) they are going to make it. A quality system must have as its backbone a good communication system that covers all activities that affect quality. Then it is possible that data collected at the packaging stage can be fed back to the raw materials or process stage, and conversely, data from the processing stage can be fed forward to packaging or distribution stages. Thus, if a quality system allows information to feed backwards and forwards between various stages in the manufacture and distribution of the product, then the benefits of that system are much greater than the sum of the benefits

from several independent quality systems at the various stages. This is the real justification for the total quality control concept, and incidentally, a pretty good case for the existence of a quality manager. There is a danger when one talks of quality being everyone's job. It is, but without the proper co-ordination of effort, it becomes no-one's job.

Before one can control anything properly, the raw materials, process conditions and product specifications must be prepared, and this is what I call quality planning. When these product specifications have been produced they must be implemented by some inspection scheme and this I call quality measurement. When these product specifications have been produced they must be continually evaluated to see whether the specification is being satisfied and to see in which way they should be changed to improve the final quality of the product. This is quality audit.

Thus the basic needs for any quality system are three functions.

- (1) Quality planning.
- (2) Quality measurement.
- (3) Quality audit.

These functions may be combined depending on the size of the operation or the particular needs or resources of the company, but essentially work must be performed in each area.

Quality planning

This is the most critical area when a quality system is being introduced. The efficiency of the quality planning initially determines the acceptability of the proposed quality measurement plan by production, sales, marketing and development and also determines the effectiveness of the plan in reduction of waste and improvement in the consistency of the product. Broadly speaking, quality planning consists of gathering all the relevant ideas and data, evaluating them by measurement when possible, collating the data into a specification for a process, product or raw material and then getting the acceptance and hence some commitment of the various functions to this specification. This could include outside suppliers and customers. From these specifications a plan is devised for sampling the product, process or raw material which will give statistical assurance that the specifications are being met.

This raises a most important point on specifications. These must be related to the resources of the company. A specification which says every piece of diced apple must be a specific size is unrealistic if it means that every piece of apple must be measured and also unnecessary if the finished product does not require it. A suitable specification for diced apple might include the most desirable size (with tolerances) and a sampling plan which states the limiting number of oversize or undersize pieces of apple in a given sample size taken from a given lot size. This will then ensure, with a level of statistical probability, that the accepted lots will only have a certain percentage of undersize pieces of apple. Obviously care has to be taken in sampling, but

there is a far higher chance that this kind of specification can be used by both supplier and customer to initially standardize the supplies and which will lead to their improvement. It also gives a reasonable basis for negotiation which will not be unjust to either party. Similarly, specifications relating to process and product should be based on process capability studies which again give the assurance of quality in terms of statistical probability. Quality planning is involved every time that a change is made to a specified process or product, since there must be an evaluation of the changes that this will cause in other areas.

Specifications are not inviolate, but the reasons for changing them must be demonstrable. If people are to be committed to working to specified levels and tolerances they must be demonstrably feasible and not just someone's opinion. This means that any proposed change must be carefully checked out by experiment, or sequence of experiments, before a permanent change of specification is made. This is a most useful discipline since it means that the reasons for change are properly documented and the benefits of the new specifications, over the old, clearly shown. If a change cannot be shown to be demonstrably better, then the changes should not take place. I make no apologies for this statement. This may mean the delay of a possibly beneficial change until some means of measurement of the benefit can be devised, but it also eliminates the high proportion of changes which are made on someone's opinion, or under pressure for production or sales, which could affect quality and profit adversely in the long run. A lot of thought and work will have gone into the original specifications and the causes for their inadequacy must be carefully determined. Otherwise a series of panic changes lead one into the unknown position where one is even more insecure in that if anything goes wrong after the changes are made, (and it nearly always does), there is no 'sense of direction' for further change.

This raises an important point on all specifications—that there should be a constant awareness of the need to know what happens on either side of the tolerances. I shall deal with this point later, but it is important to establish this 'sense of direction'.

Quality measurement

This is the area which most people think of when they think of quality control. This is the function that carries out the quality plan—the sampling and inspection of the raw materials, process and product.

In some cases the quality measurements are carried out by the machine operator, or sorters, but more and more separate inspection bodies are being set up in factories. Why should this be, when having the operator doing the inspection eliminates communication problems and interpersonal frictions between inspectors and operators, and shortens the feedback loop? To answer this question one must look at the basic requirements of operator inspection and why they are difficult and, at times impossible, meet.

- (i) *Requirement:* He must have enough time to take samples at the appropriate times.
Objection: He may be required to be somewhere else in emergency, when the samples need to be taken.
- (ii) *Requirement:* He must be trained in, and capable of understanding, the interpretation of the data.
Objection: A very big training programme would be needed to train all the operators, and there would be no flexibility if one left.
- (iii) *Requirement:* He must be honest and responsible enough to report that the product or process goes out of control when he is in charge.
Objection: The climate of production supervision could militate against a man reporting an 'out-of-control' situation. It is not easy for a man to admit that the process is out of control when he may lay himself open to disciplinary action.
- (iv) *Requirement:* He must be willing to tighten the tolerances if events show that the process is capable of working to tighter tolerances.
Objection: The man would have to work harder if he tightened tolerances. Most bonuses are production oriented, so there would be opposition to the suggested tightened tolerances.

An objective evaluation of the two alternatives normally results, in some part at least, of the inspection and evaluation being done by full time inspectors. I feel very strongly that the quality control inspectors should be independent of other departments in accountability. There are too many pressures to get a given production, whatever the difficulties, to allow full consideration to be given to the quality of the product at times. This also applies to Sales Departments and in some degree to Research and Development departments. It is *not* being unrealistic to say that the specified quality of the product should not be influenced unduly by short term considerations. At the same time a climate must be engendered that encourages the inspector and the operator to work as a team, although each has different duties and is in a different chain of command. This is helped by being completely open from the start about what the inspector is doing and why he is doing it. The Quality Department is in principle advisory although its aims are to ensure that the laid down standards and specifications are met. Any deviation from these specifications should be notified and agreed with the quality department prior to implementing the deviation. This allows the effect of all changes to be followed and documented. Here the efficiency of the quality planning is important. If the planning work has been done well then the number of deviations necessary should be small. The specifications should be able to cope with all batches of raw materials coming in within specification and all ambient conditions. If they do not then the process has not been properly evaluated and quality planned. Quality measurement must have the power to prevent any raw material being used in the factory if it does not meet the specification and to stop any products going out if they do not meet the requirements laid down. These mandatory powers make it obvious that the

Quality Department must be independent of production and sales. These powers can be overruled, but not by the Production Executive or the Sales Executive alone.

Quality devices. The most effective devices are very often the simplest—the average and range chart and number defective chart. The more sophisticated devices give more precision and more information but are harder to communicate to those not skilled in this field. Thus the confusion or the hesitation of production staff can quite quickly lose the extra benefits of the more sophisticated techniques. The really effective devices are those that have been introduced with proper concern for the operator whose work is controlled by them and where time has been taken to explain *all* the implications of the system. If this is done with the more sophisticated techniques then the benefits of greater precision and greater information can be obtained, but the time taken to explain and teach is that much longer and that much more difficult.

Quality audit

This is an often neglected area of quality control and any quality manager should look carefully at this aspect of his activities to make sure that:

- (1) He is giving value for money in his quality systems.
- (2) He is using the data coming back from the inspection to extract the maximum use from it.

The savings claimed by the use of quality control schemes can often be inflammatory documents incensing production, development and works study who unbeknown to the quality manager are already claiming to have saved the same sum of money. I have a feeling that if all cost savings claimed by individual departments in some companies were added up they would represent several times the actual cost saving achieved! Nevertheless, a figure can be put on the savings achieved by quality systems. During the planning stage of any quality system there should be an evaluation of the current situation in terms of waste, excess people employed reworking material and efficiency of the plant in terms of good production rate. This becomes the standard against which the waste, labour costs and efficiency after the installation of the quality system can be compared. It is true that during this period of intensive investigation, problems are highlighted in areas that Engineering, Work Study, Production, and Development departments work on and solve, but the question is whether this would have come to light so soon, had an investigation not taken place. A careful look at the figures for waste, labour and efficiency over the previous year would confirm or deny this. Another (but more difficult to assess) aspect of a properly quality planned product and process is that one finds that it is possible to get a higher quality level than was thought to be the case. This is an added and not easily calculable benefit.

Inevitably the level of defectives drops after the installation of a tight quality control system and eventually the level of defectives flatten out at some economically acceptable figure. This is the time to look at the cost of inspection. By analysing the data coming back from the inspectors one can determine the nature of the trends and drifts in a

process accurately, probably for the first time, and it should be possible to decrease the frequency of inspections, or, if retaining the same frequency, reduce the sample size. It may even be desirable to have alternative sampling plans with the intensity of inspection dependent on the state of the process, i.e. if a high level of defectives is current then a highly intensive sampling is started, but when the process is running well a much less intensive sampling plan can be used.

The use of inspection data a second time is a very fruitful occupation in another sense. A statistical analysis of the inspection data by multiple correlation and other techniques can give clues to which variables are relevant in controlling the product quality and so direct attention to those variables by engineering, development, production, etc. This can also be done between raw materials and the process and the packaging. This provides the feed-back loops and the feed-forward loops of information and is possible because the inspection is co-ordinated by the quality department and so the standards of inspection are known and can be maintained at the same level of efficiency and objectivity. This will also give a 'sense of direction' which can indicate the direction that change is needed.

Quality audit can, and in the case of Lyons Bakery does, include the collection, investigation and tabulation of complaints which can give very useful clues to the problems of the product in distribution and sales. In addition to this, some monitoring of the distribution system is done to investigate the problems of various products as they go through the distribution system. These are problems such as deterioration and damage of the product as it goes to the shops.

An important aspect of quality audit is the production of summaries of quality department activities, plant reject rates and the outgoing quality level and the costs of inspection against the cost of rejects. These reports are the longer term equivalent of the reports from the product inspector each time he takes a sample and reports on it. This is a management control tool which shows where attention is needed, and if possible, what steps should be taken. Thus a quality concept is established in which specifications, standards and procedures are prepared and implemented, data is collected for immediate control purposes and immediate feed-back or feed-forward and the data is then analysed again to detect longer term drifts for strategic planning of inspection and to establish a sense of direction for future change.

Relationships between quality department and other departments

The immediate interface problem when a quality system is installed is between Quality Department and production since production (in particular operatives) see their authority questioned and an independent check being introduced using a system they know little about. This fear of the unknown is the main obstacle in the way of the successful installation of a quality plan and this can be overcome to a large extent by

taking the time and the trouble to explain to each individual what the quality system consists of, and what he has to do and when and where. The quality system should cover, as much as is humanly possible, all of the aspects of each individual's work which affect quality, so that he does not work to laid down standards in one area of his activities and without standards in a large area which is just as important. This type of inadequate quality system leads people to ignore the standards in those areas which are covered by the quality plan. It also helps if the individuals are encouraged to contribute to the quality system in the planning stage.

Another interface problem not always anticipated is between the Research and Development Department and the Quality Department. Running a plant to laid down standards, procedures and specifications brings with it the philosophy that since the process has been carefully evaluated, there should be no change to these specifications unless the benefits can be demonstrated. In other words, if the effect of a change cannot be assessed and shown to be beneficial it should not be carried through. This necessarily means that all changes must be tried out under conditions which are as closely controlled as possible to examine all the possible effects prior to making the change permanent.

One of the means of overcoming this interface problem is for the Quality Department to join in and help with the experiments by providing background information on the raw materials, process and product, etc., and providing help with measuring the effects. This can be done by the Research and Development man submitting a request for a trial with all the reasons for doing this trial and the possible risks involved and this to be countersigned by the Production and Quality departments who will work out the best way of organizing the trial and measuring its effects together with the Research and Development man. Thus when a change is carried out, it is done with the knowledge that the three parties involved (also Marketing if they are vitally interested in the change) have participated in a thorough evaluation of the change before it is made permanent.

Another area in which Quality Department are able to contribute is in the very early stages of product or process development where they can ensure that standards and specifications are available at an early stage and that the changes are documented and the 'grey areas' which need to be evaluated are clearly understood before a new product or process becomes a major line.

A last word about specifications. No specifications should be issued dictatorially by the Quality Department. When all the data has been gathered and sifted in conjunction with all functions of a company a draft specification must be issued. This specification cannot become official until all the interested functions have read, and agreed it and signed a master copy. Any objections to the draft specifications have to be examined and met, and subsequent modifications agreed so that the official specification starts out in life with the heads of the interested departments behind it. It has then a real chance of succeeding.

Horizons of quality control

The manner in which the philosophy of running a process has evolved can be described in three stages.

1. *Random chance*

This is the way processes used to be run, which was on day to day (almost minute to minute) decisions on standards. This produced a lot of varying products coming from a lot of varying process conditions and the chances were that if you went on long enough you would come across one process which was better than the others and produced a better product. This was understandable if you could afford to wait long enough for this to happen (and afford the waste and substandard material produced), and pre-war (even post-war) a product, in more or less the same form, was sold for an indefinite period. Nowadays, products have built in obsolescence and time is too short to wait for random chance.

2. *Selective experimentation*

This is the current pattern of running a process. One carefully evaluates a process and lays down specifications and then any change in these specifications must produce a demonstrable improvement. One can speed this up by developing the 'sense of direction' that quality services can provide by deep analysis of the data and by increasing the Research effort to provide a scientific basis for the technology. But in some areas, the very speed of obsolescence and change is making great demands on the technological resources, which can escalate in a frightening manner.

3. *Optimization*

This is a much over-used and ill-defined term and I would define it as a continuous logical search for the method of running a plant to obtain maximum profit. I see a plant being run in a dynamic state of change, where changes are small, planned and evaluated at a very rapid rate so that one is moving to a more profitable area from the very start-up of a plant. Many variables will be involved and probably a computer would be needed to carry out this work. Major changes could be superimposed on this system without interruption of the programme, so that they can be evaluated and confirmed as successful, or rejected. In other words, to use a genetic analogy; neither natural selection (which corresponds to random chance) nor mutation (which corresponds to selective experimentation) is enough. A combination of the two logically planned and carried out, will produce a product that fulfils customer requirements consistently and which travels up a rising spiral of profitability. This third method is only just beginning to be thought about and it is this concept that Quality Departments will be vitally involved in making a reality. The changes in thinking and expertise within Quality Departments and industry will have to be great at all levels. But this is no bad thing, since today's concepts of quality already have obsolescence written into them whether we know it or not.

Distribution control

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THE SCOPE of distribution is taken as applying to the interval between the producing or packing factory and the point of sale, which in most cases is to the housewife. Control must, therefore, take into account all the varying conditions and different outlets from small shops to self-service stores and supermarkets. Where the life of the product is limited it is essential to hold only the amount which can be sold while it is in good condition. The interval includes depot storage and, therefore, co-operation with the manufacturers will ensure that storage is under the best conditions for the product. To some extent an increased shelf life for some products has been made necessary owing to changes in buying habits. Transport is also involved, especially in the case of perishable products, and the condition of vehicles requires attention.

Should queries or complaints arise subsequent to a sale these must of course be investigated and any necessary action taken. In the case of complaints we want to be able to trace back the product as far as possible. It may be necessary to isolate or even reject a particular batch. Adequate coding is, therefore, necessary, which should give all the required information and if this can be made available to the distributor, it would enable him to ensure good stock rotation and to keep a check on the shelf life of the product and prevent excessive storage. Complaints can be valuable in that they indicate a breakdown somewhere along the line and investigation of a complaint may lead to a necessary tightening up of a process and so prevent future trouble.

General types of products

For control purposes, we can divide products roughly into three general types.

Long Life products refer mainly to canned or preserved foods such as meats, vegetables and fruit, dry goods, and cereals although these are subject to certain limitations of storage life. These products are affected by vacuum, the oxygen content of the head-space, the material of the container, the composition of the pack and the method of preparation.

Medium Life products include butter, self raising flour, soft drinks, dried fruits, nut products, cheese, etc., which although they have normal good keeping qualities, are subject to such changes as evaporation, oxidation of flavours, loss of active ingredients, etc. These are affected by the length and temperature of storage and the relative humidity under which they are kept. Other factors are the type of wrap or container.

Limited Life products include sausages, bacon, cooked meats, milk, bread and pies and products which have to be sold within a few days of receipt at the shop. These products are affected by temperature, storage time and humidity and handling is a vital factor. Where cooked meat products are concerned the distributor must constantly bear in mind possible contamination from hands, equipment and exposure. It must be remembered that cooked meats are not cooked again before consumption and any organisms present will be eaten with the product.

Legal aspects

Many foods are subject to compositional requirements, limits of impurities and of permitted additives.

For home produced foods the packers are fully aware of all requirements and these goods are normally purchased under warranty, but in the case of imported foods packers abroad must be made aware of the standards applicable in this country and the requirement of labelling. Apart from standards, we have in this country Codes of Practice and accepted standards which are not in the Statute Book and these are less likely to be known abroad. In a number of cases the additives permitted abroad for colour, preservatives, raising agents, etc., are different from those permitted in this country and difficulties can easily arise unless full co-operation is obtained before deliveries are made. Warranty is often not applicable to imported products.

Where pre-packing is carried out, a number of packaging materials are necessarily permeable and this may lead to a loss of weight.

Chemical aspects

Apart from compositional standards, a number of other checks must be carried out for such effects as can corrosion, stripping of lacquer, loss of vitamin potency, oxidation of fats or flavours, possible alteration of colour and similar chemical effects. The methods used for chemical analysis are generally agreed, although the choice of solvents, quantities used, etc., can give some variations in results. With imported food, it is very important that the details of the method of analysis and of the calculation of results be agreed and checked by replicate analyses. The calculation of the analytical results to presumed constituents may be affected by the manufacturing process and assumptions are usually made in the calculation. Natural products vary in composition according to origin, type, climate, soil, time of harvesting, etc., and these factors can appreciably affect the calculated result. Where no standards of composition exist for the individual food, the distributor must ensure that the quality of the product satisfies the enforcement authorities as well as the consumer.

Bacteriological aspects

This subject is of special importance in connection with the distribution and sale of

cooked meats which, although sterile if packed in cans, are sliced and necessarily handled for display and sale, and during the interval between removal from the can and the time of sale may be liable to contamination which can develop extremely rapidly. 'Open pack' products are, of course, the most sensitive and extreme care must be taken from the manufacturing stage, through delivery, to the selling point, and close liaison is essential between the manufacturer and the distributor. Date coding by the distributor is, therefore, an important safeguard. Pre-packing of these products may reduce some hazards, but this can also give a false sense of security. Also in the grocery trade it is accepted that a good display is an important selling point.

Again, the method of test may be important with special attention being given to details; as an example, a number of bacteriologists have had difficulty in finding *Salmonella*.

The method of reporting bacteriological results is often not uniform. The amount of sample is usually definite but when testing equipment there is often little information as to the area tested and whether the swab was of cotton-wool or of a soluble substance. Agreement on these points would greatly help the comparison of results obtained by different workers.

Although desirable standards for the bacteriological quality of meat products have been formulated, enforcement of these standards is a difficult matter. Meat from slaughterhouses and imported meat often contains a high bacterial population, including some pathogens, and the handling entailed in the preparation of the meat for consumer requirements can allow the spread of this contamination to hands, clothing, equipment and premises, with the danger of subsequent re-contamination of the processed product. The rapid rate of growth and reproduction of these organisms under favourable conditions must be constantly borne in mind and the necessary action taken by the use of low temperatures and bactericides, and the distributor must be prepared to limit his range of products to those which conform to his possibly arbitrary standards.

Hygiene aspects

Hygiene is of supreme importance in connection with food—from the raw material to the shop counter. Pre-packing has greatly reduced many hazards, but adequate measures must be taken to maintain premises at a high standard of general and bacteriological cleanliness by the adequate use of suitable detergents and bactericides as well as by a suitable layout of the premises, the use of guards, covers and cool storage. Precautions must also be taken against possible infestation by rodents, insects and birds and also against contamination by micro-organisms and foreign matter. Again the identification of foreign matter, and how it came to be present, are important in order to prevent future trouble.

As a general rule, the amount of stock held at the shop should be such that all is sold within the period of safety and the amount on display should be the minimum possible.

This seems an obvious truism, but it must be remembered that weather can greatly influence purchasing. It is also important to clear packaging materials frequently and the 'empties' room requires attention, while doubtful or condemned stock should be isolated from saleable products. Imported products such as dried fruits and nut products may require extra care as they are often packed under conditions where hygiene is not as good as in this country.

Training of the personnel is necessary in the awareness of ways in which organisms spread, e.g. handles, wiping cloths, overalls, equipment, towels and handkerchiefs. This awareness must be present through the whole chain from Management to the counter hand. Food hygiene is a continuous and progressing process and this must be brought home to all personnel by periodic refresher courses and the use of posters and other visual aids.

Control

This is effected by the examination, testing and analysis of samples which are assumed to represent a reasonable cross section of the possible variables. 'Long Life' products are manufactured on a large scale and all precautions such as retorting, pH, solids, moisture, etc., are taken by the manufacturer to yield a stable product. These products, therefore, do not in general require such frequent testing as 'Medium Life' or 'Limited Life' products.

Chemical analysis will indicate compliance or otherwise with the legal requirements, standards and specifications, and bacteriological tests and examination of the product will indicate quality, but the significance of these results will depend on the sampling. The frequency, size and location of sampling is, therefore, of very great importance.

With regard to the bacteriological examination of samples, the results can and do vary from site to site and from sample to sample. Even under good hygiene conditions, chance contamination from hands, equipment and airborne sources can build up rapidly. Tests on equipment and premises can indicate the danger spots and personnel are trained to recognize these hazards and to reduce them by frequent cleaning and the use of suitable bactericides.

Co-operation with authorities

The control methods outlined above are only one aspect of the range of controls in which Licensing Authorities, Research Associations, Manufacturers, Public Health Departments, the Public Health Laboratory Service, Port Authorities, Public Analysts and others all play a part and I freely acknowledge the help and co-operation we have received from these sources in the endeavour to supply satisfactory food and to prevent waste and make the best use of available resources.

Summing up and closing remarks

J. G. DAVIS

President of the Institute of Food Science and Technology

MR CHAIRMAN, Ladies and Gentlemen, I feel now rather like the Bishop who is called on to pronounce the benediction at the end of a very long service; the congregation know that they cannot dodge it, they merely hope that he will not take too long about it. Now let me put your minds at rest straight away. I am not proposing to endeavour to sum up all the papers that have been given at this most excellent *Symposium*, in fact I think that it is quite unnecessary for the papers have been given so very clearly and precisely and the very fruitful discussions have brought out so many points in these papers, that I think there is no need for me to attempt this. We shall publish all the papers in some form or other and that will give you an opportunity to read them at your leisure and go through any points which you may have missed. We have had some questions put to us about the Institute so I would like to deal with some of these enquiries because the Institute is still a new organization and a lot of people do not know about it yet. We get the inevitable questions: Why have another scientific society? Why have another scientific journal? and so on. Let me make a few points clear. We are not a scientific society in the usual sense and we are not a rival organization to the Food Group of the Society of Chemical Industry. I make that clear because so many people ask: Why have a Food Group as well as an Institute of Food Science and Technology? There are two fundamental differences between our Institute and the Food Group. Firstly, we are not a scientific society. We expect certain qualifications for applications for membership, and we have different grades of membership; fellow, associate, licentiate and student. These are graded to quite different degrees corresponding fairly closely to the requirements of the Royal Institute of Chemistry, the Institute of Physics, the Institute of Biology and similar organizations. The second major difference between the Food Group and ourselves is whereas the Food Group consists almost entirely of chemists, we include in our membership all sorts of scientists and technologists. We interpret the terms 'food scientist' and 'technologist' very broadly: we include mainly chemists, but we also have physicists, mathematicians, statisticians, biologists, botanists, microbiologists, engineers and we can accept any other type of discipline or person who can be classified as a food scientist or technologist. If you are in doubt as to whether you conform to these requirements, put in an application and see what happens.

Now what do members get for their money? The subscriptions vary from £2 for

students up to £5 a year for fellows. You get the opportunity of attending all meetings of the Institute, and we already have branches in the Midlands, the North of England, Northern Ireland and Scotland. It is highly probable that a branch will also be formed in the Republic of Ireland and there may later be one in the West of England. All members may attend any meetings. In addition we publish a journal which is already well known and has a high reputation—the *Journal of Food Technology*. As soon as we can afford it, we shall also publish *Proceedings** which will be an amplified form of the present *News Letter* which gives general information and news about the activities of the Institute and our members individually. If any of you are interested to have further information, you will see the various officers of the Institute roundabout the hall with their badges and you can ask them any questions you like.

Now it leaves me only to perform a very pleasant duty. That is, to thank all those who have contributed so magnificently to the success of the symposium. First of all I would like to express our appreciation of our Chairmen, Lord Kings Norton, the first morning, Lord Sainsbury in the afternoon, Lord Trenchard this morning and Sir Samuel Salmon this afternoon. I think the fact that men of such distinction and leaders of our food industry have been willing to take the time off from their many duties to come along here and preside over our meetings shows the importance they attach to the functions of scientists and food technologists in the industry today.

Next I would like to mention all the speakers who have given us such excellent papers. We are greatly indebted to them and hope we will be seeing them on other occasions and have other excellent papers from them. Lastly, but not least, I would like to express our thanks to those who have been responsible for organizing the symposium, Mr McLachlan the Chairman of the Programme Committee, who is also our Vice-President, Dr Herschdoerfer, who has been the Convener of the Symposium Committee and who I may say has done most of the detailed work; and in addition Mr Heath and Mr Spencer, two members of the Committee, who have also very ably assisted Mr McLachlan and Dr Herschdoerfer on many of the details. Only those who have ever been responsible for organizing a symposium like this can appreciate the terrific amount of time and effort which has to be put in to make such a function efficient. May I, therefore, ask you to express your appreciation in the usual way?

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JOURNAL OF FOOD TECHNOLOGY: NOTICE TO CONTRIBUTORS

The *Journal of Food Technology* publishes original contributions to knowledge of food science and technology and also review articles in the same field. Papers are accepted on the understanding that they have not been, and will not be, published elsewhere in whole, or in part, without the Editor's permission. Papers accepted become the copyright of the Journal.

Typescripts (two complete copies) should be sent to the Editor, Dr E. C. Bate-Smith, A.R.C. Institute of Animal Physiology, Babraham, Cambs. Papers should be typewritten on one side of the paper only, with a 1½ inch margin, and the lines should be double-spaced. In addition to the title of the paper there should be a 'running title' (for page headings) of not more than 45 letters (including spaces). The paper should bear the name of the author(s) and of the laboratory or research institute where the work has been carried out. The full postal address of the principal author should be given as a footnote. (The proofs will be sent to this author and address unless otherwise indicated.) The Editor reserves the right to make literary corrections.

Arrangement. Papers should normally be divided into: (a) Summary, brief, self-contained and embodying the main conclusions; (b) Introduction; (c) Materials and methods; (d) Results, as concise as possible (both tables and figures illustrating the same data will rarely be permitted); (e) Discussion and conclusions; (f) Acknowledgments; (g) References.

References. Only papers closely related to the author's work should be included; exhaustive lists should be avoided. References should be made by giving the author's surname, with the year of publication in parentheses. When reference is made to a work by three authors all names should be given when cited for the first time, and thereafter only the first name, adding *et al.*, e.g. Smith *et al.* (1958). The '*et al.*' form should always be used for works by four or more authors. If several papers by the same author and from the same year are cited, a, b, c, etc., should be put after the year of publication, e.g. Smith *et al.* (1958a). All references should be brought together at the end of the paper in alphabetical order. References to articles and papers should mention (a) name(s) of the author(s); (b) year of publication in parentheses; (c) title of journal, underlined; (d) volume number; number of first page of article. References to books and monographs should include (a) name(s) and initials of author(s) or editor(s); year of publication in parentheses; (b) title, underlined; (c) edition; (d) page referred to; (e) publisher; (f) place.

Standard usage. The *Concise Oxford English Dictionary* is used as a reference for all spelling and hyphenation. Verbs which contain the suffix

ize (ise) and their derivatives should be spelt with the z. Statistics and measurements should always be given in figures, i.e. 10 min, 20 hr, 5 ml, except where the number begins the sentence. When the number does *not* refer to a unit of measurement, it is spelt out except where the number is greater than one hundred.

Abbreviations. Abbreviations for some of the commoner units are given below. The abbreviation for the plural of a unit is the same as that for the singular unless confusion is likely to arise.

gram(s)	g	second(s)	sec
kilogram(s)	kg	cubic millimetre(s)	mm ³
milligram(s)	mg	millimetre(s)	mm
(10 ⁻³ g)	mg	centimetre(s)	cm
microgram(s)	µg	litre(s)	l
(10 ⁻⁶ g)	µg	millilitre(s)	ml
nanogram(s)	ng	pound(s)	lb
(10 ⁻⁹ g)	ng	gallon(s)	gal
hour(s)	hr	milliequivalent	mEq
minute(s)	min	R _F values	R _F

Figures. In the text these should be given Arabic numbers, e.g. Fig. 3. They should be marked on the backs with the name(s) of the author(s) and the title of the paper. Where there is any possible doubt as to the orientation of a figure the top should be marked with an arrow. Each figure must bear a reference number corresponding to a similar number in the text. Photographs and photomicrographs should be unmounted glossy prints and should not be retouched. Line diagrams should be on separate sheets; they should be drawn with black Indian ink on white paper and should be about four times the area of the final reproduction. Lines and lettering should be of sufficient thickness and size to stand reduction to one-half or one-third. Letters and numbers must be written lightly in pencil. Whenever possible, the originals of line diagrams, prepared as described above, should be submitted and not photographs. The legends of all the figures should be typed together on a single sheet of paper headed 'Legends to Figures'.

Tables. There should be as few tables as possible and these should include only essential data; the data should not be crowded together. The main heading should be in capitals with an Arabic number, e.g. TABLE 2. Each table must have a caption in small letters. Vertical lines should not be used.

Page proofs will be submitted to the contributors for minor corrections and should be returned to the Editor within 3 days. Major alterations to the text cannot be accepted.

Offprints. Fifty offprints will be issued free with each paper but additional copies may be purchased if ordered on the printed card which will be sent to the senior author with the proofs.

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