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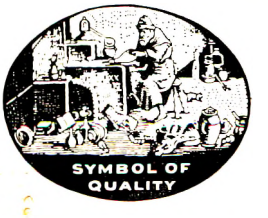

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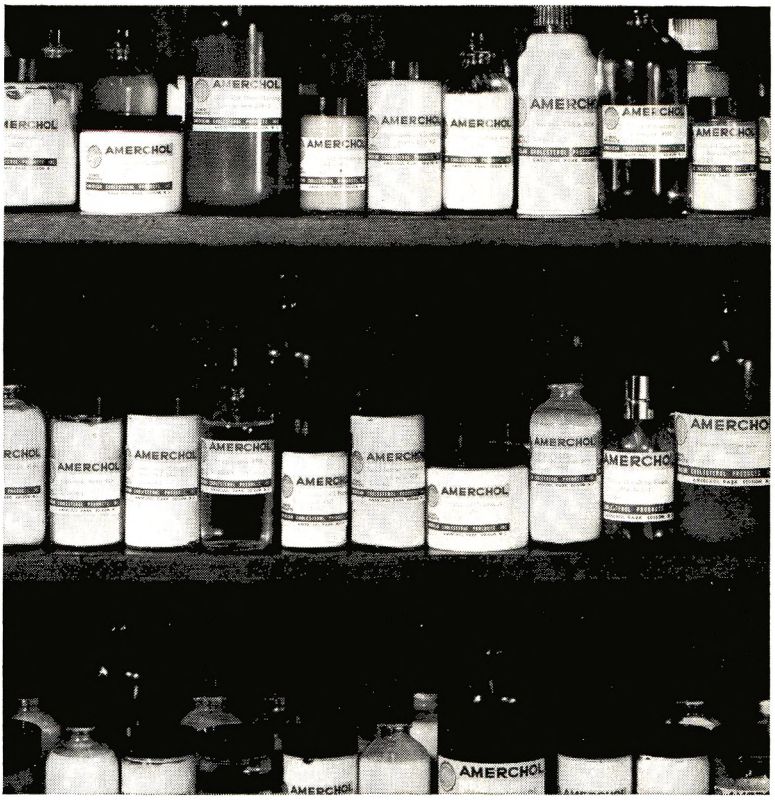

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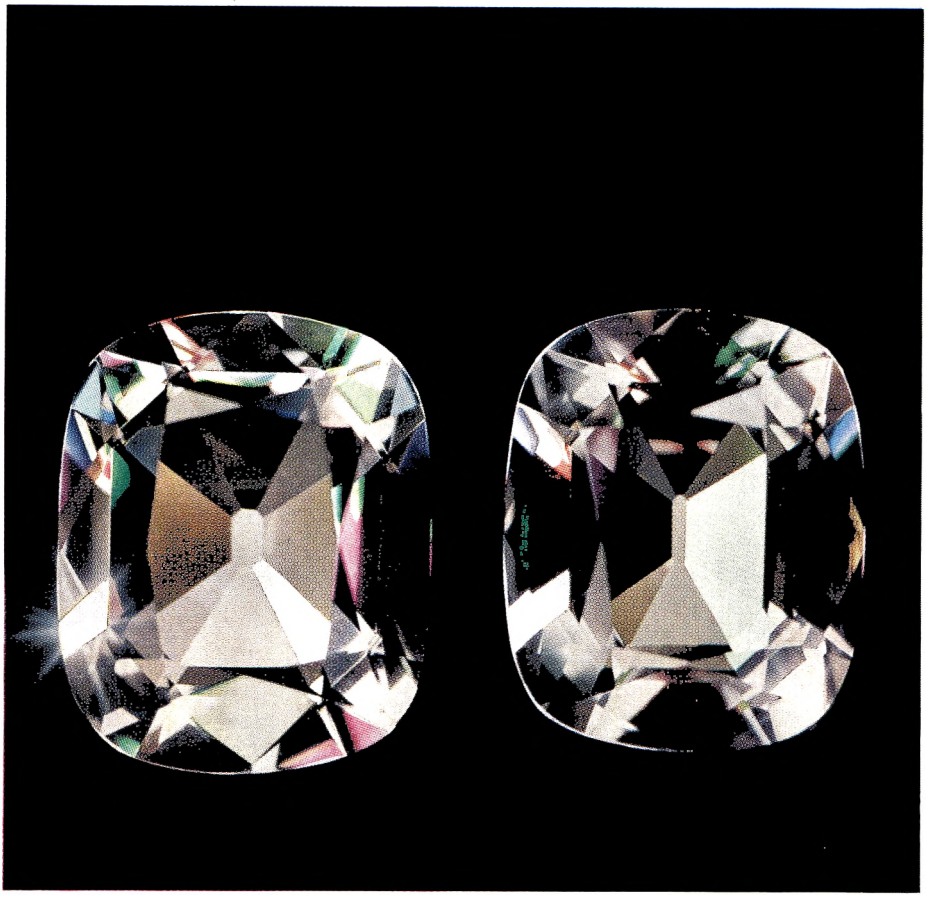

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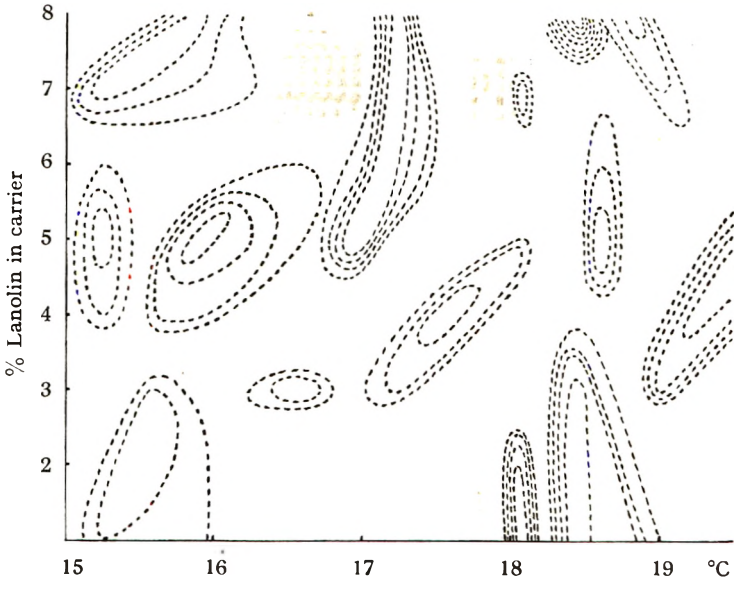

Figure 3
Fresh lanolin. Index contours 40 (outer) x 5

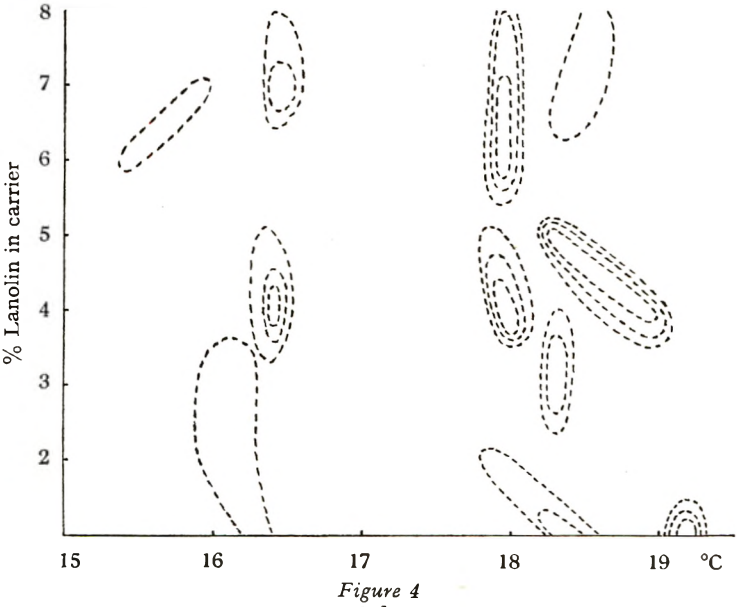

Figure 4
Degraded lanolin [100 hr—100 °C]. Index contours 40 (outer) x 5

as is shown by the contours lying at an angle to the concentration and temperature axes and across more than one critical peak temperature. In view of what is known of the constituents of lanolin this is exactly what would be expected. Again there is evidence of the formation of peroxides. Some traces of the carrier peak remain, but it is more broken up than in the case of the olive oil pointing to higher concentrations or high surface activity of some of the polar compounds, or possibly both. The two large peaks at low concentration between 18 and 19°C are due to free acid, probably produced by hydrolysis during the extraction and purification of the lanolin, and can be almost exactly reproduced by using a pure mineral oil to which a small quantity of lauric acid has been added. The sloping contours at 19 to 19.5°C suggest ketonic or hydroxy-acids.

The above sample of lanolin was degraded by heating a thin layer in an oven at 100°C for 100 hours. The resulting diagram (*Fig. 4*) is much simplified, and suggests that a certain amount of re-esterification has taken place. There is an increased and more definite indication of ketones (two peaks at 16.4°C) whilst the peroxide, hydroxyl and carrier peaks have largely disappeared. There are, however, still strong indications of acid constituents between 18 and 19°C, but at a lower concentration than in *Fig. 3* since the peaks appear at a higher lanolin concentration.

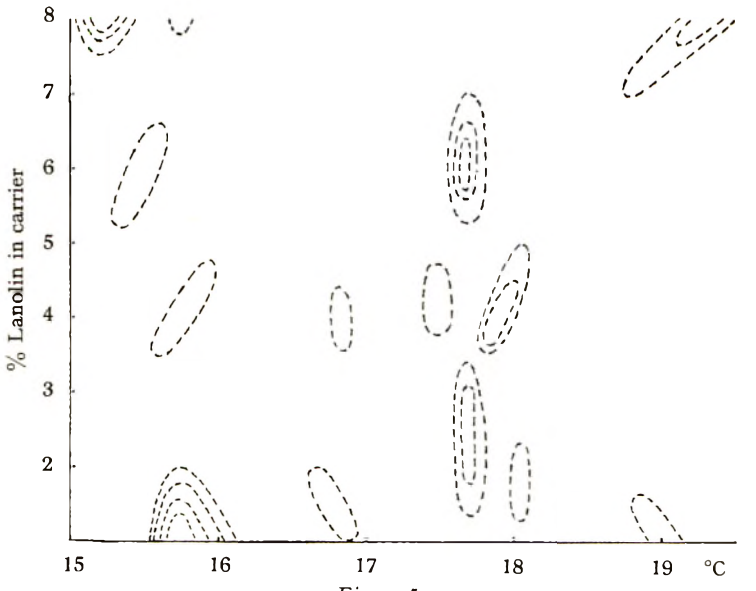

Figure 5
Lanolin, stored 10 years. Index contours 40 (outer) x 5

The main change appears to be oxidation of secondary hydroxyl to ketonic groups as might be expected.

Fig. 5 was obtained by using a sample of lanolin which had been in store for ten years. Here re-esterification has definitely taken place. The acid and hydroxyl peaks are markedly less in evidence, but no ketone has been produced.

There is a small amount of peroxide showing at the top, left-hand corner of the diagram. The reappearance of peaks in the 17.8 to 18°C range suggests the reforming of the carrier peak, a phenomenon to be expected if the concentration of the polar groups had been very considerably reduced.

Conclusion

Outline/area index chromatography appears to be a useful and sensitive technique for the examination of mixtures the nature and complexity of which make other methods difficult to apply.

At this stage it is presented as a potentially useful idea rather than as a completely developed method. There are still a number of points, particularly those associated with the influence of molecular structure, which are not clear, and, at the moment, no satisfactory theoretical explanation of the variation of the index can be put forward.

The obvious disadvantage is the amount of work involved in the production of the diagrams, but against this must be set the simplicity of the apparatus and technique, and the rapidity with which the results may be obtained.

While it is not suggested that the method would be used where other established techniques are satisfactory, except perhaps as a method of preliminary examination, it is of interest that an infra-red examination of the sample of lanolin did not yield anything like as much detailed information as the outline/area index diagrams.

Acknowledgements

The author wishes to thank Dr. G. F. Reynolds, Reader in Analytical Chemistry, and the Esso Petroleum Co. for the sample of mineral oil, and Dr. A. G. Briggs for advice and assistance with the I.R. examination of the lanolin.

(*Received : 2nd September 1964*)

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(4) Poxon, D. W., Agger, R. T., Beaumont, D. E. and Pugh, J. A. *J. Oil Colour Chemists Assoc.* **46** 339 (1963).

Introduction by the lecturer

The main point about the method described is that there is no need to separate the materials present in the mixture being chromatographed. The alterations in band shape indicate the particular groups which are present because these give peaks at definite temperatures and in the substances which we have investigated, this peak, say the hydroxyl peak, occurs at 15.8°C no matter what the compound is.

At the same time, if the results are plotted in a diagram having temperature and concentration as axes, the peak height being indicated by contours, different substances appear at different concentrations. This means that if one is investigating a mixture about which very little is known, at least as a preliminary examination, one does not have to search around for different methods of separating out the hydroxyl compound, the ketone or the carboxylic acids. The method indicates the presence of these substances and, within reason, how many of them occur. The apparatus (*Fig. 6*) consists of a 2l beaker with a Perspex cover which holds a thermometer fitting reasonably tightly into a copper cone which is never removed from the inside of the apparatus, and therefore provides good thermal contact. The cover also holds a length of glass rod bent into a circle at right angles to the stem which holds the actual paper by the tabs (between the sectors), which are bent down at right angles. After the application of the material the paper is held in an atmosphere of petroleum ether for between 1 and 2 min. This time is quite critical if reproducible results are to be obtained. The paper is then lowered by pushing down the glass rod so that

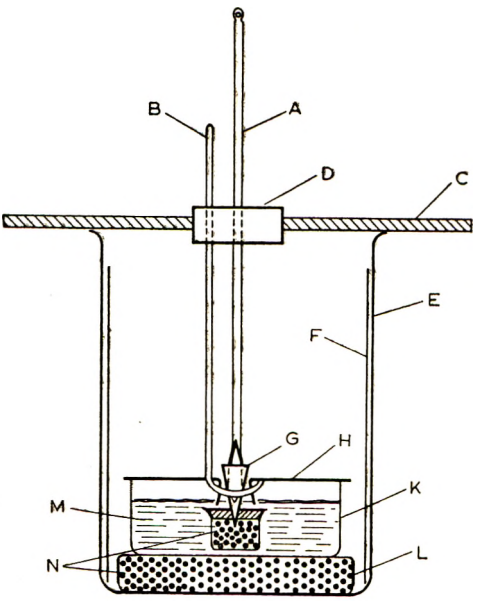

Figure 6
Apparatus used (4)

A. Thermometer
B. Paper holder
C. Perspex lid
D. Cork
E. Two-litre beaker
F. Paper lining
G. Copper cone
H. Chromatographic paper
K. Crystallising dish
L. Weight
M. Solvent
N. Lead shot

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Band shape on dyed paper as a method of chromatography for oils and fats: D. W. POXON. *Journal of the Society of Cosmetic Chemists* **16** 3-14 (1965)

Synopsis—Certain dyes, containing surface active groups, profoundly influence the shape of a chromatographic band in a manner related to the composition of the material being chromatographed.

Using paper impregnated with such dyes, together with the length of outline per unit area as a measure of the shape of the band, a method of chromatography has been evolved having a completely standardized development technique for all mixtures.

This outline/unit area index varies with temperature according to the nature of the polar groups of the compounds present in the mixture being chromatographed, and with the concentration of the individual compounds.

A three-dimensional diagram may therefore be constructed, having as axes temperature, concentration and index, which will be absolutely characteristic of a particular experimental material, and which does not require actual separation of the constituents of that material.

In the present investigation this method of chromatography has been applied to some materials likely to be of interest to the cosmetics industry.

The activity of antibacterials in two-phase systems: H. S. BEAN, S. M. HEMAN-ACKAH and J. THOMAS. *Journal of the Society of Cosmetic Chemists* **16** 15-30 (1965)

Synopsis—Bactericides in oil/water systems are partitioned between the two phases, the concentration in the aqueous phase being controlled by the overall concentration and the oil:water ratio. The antibacterial activity of the systems is determined both by these factors and by the enhanced concentration of bactericide at the oil/water interface. A change in temperature of the dispersions changes their antibacterial activity by virtue of the normal effect of temperature on bactericides, and by a change in the partition coefficient which may, in some cases, be appreciable.

The behaviour of lanolin derivatives in pressurized formulations II. A. HERZKA. *Journal of the Society of Cosmetic Chemists* **16** 31-38 (1965)

Synopsis—The solubility of ten *Golden Dawn* lanolin derivatives in seven different propellant/solvent systems, stored for six weeks at 0°C and 20°C, is described. The results indicate (1) that the solubility of four derivatives decreases on storage, (2) that the solubility of one derivative increases on storage, (3) that the solvent power of six of the propellant systems decreases on storage, and (4) that storage at 0°C does not materially affect the solubility of eight of the tested products.

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Band Shape on Dyed Paper as a Method of Chromatography for Oils and Fats

D. W. POXON*

Presented at the Symposium on "Preservatives and Antioxidants", organised by the Pharmaceutical Society of Great Britain and the Society of Cosmetic Chemists of Great Britain, in London on 17th November 1964.

Synopsis—Certain dyes, containing surface active groups, profoundly influence the shape of a chromatographic band in a manner related to the composition of the material being chromatographed.

Using paper impregnated with such dyes, together with the length of outline per unit area as a measure of the shape of the band, a method of chromatography has been evolved having a completely standardized development technique for all mixtures.

This outline/unit area index varies with temperature according to the nature of the polar groups of the compounds present in the mixture being chromatographed, and with the concentration of the individual compounds.

A three-dimensional diagram may therefore be constructed, having as axes temperature, concentration and index, which will be absolutely characteristic of a particular experimental material, and which does not require actual separation of the constituents of that material.

In the present investigation this method of chromatography has been applied to some materials likely to be of interest to the cosmetics industry.

Introduction

Although much progress has been made in the chromatographic examination of widely differing types of material, such an examination of complex mixtures of high molecular weight compounds still presents many difficulties. This is particularly so if it should be necessary to follow or detect changes, which may not be very extensive, resulting from manufacturing operations, oxidation, or deterioration of any type.

*Department of Industrial Chemistry, Loughborough College of Technology, Loughborough, Leics.

Since the complexity of the material to be examined may mean that all the components are not known, may not be separable by any one technique, or may be sensitive to such conditions as high temperature, type of column packing, etc. it is desirable to have a method of chromatography in which such factors are not of prime importance.

Certain azo dyes, of which *Dispersol Fast Scarlet B.* (C.I. Disperse Red 1.11110) has been found to be most suitable, have been shown to have a pronounced effect upon the shape of the chromatographic band of oils when the chromatography was carried out on paper impregnated with the dye, using petroleum ether as the eluting liquid.

At certain temperatures, characteristic of the polar group of the compound being examined, a sharp increase in the length of the outline of the band per unit area (the Outline/Area Index) was found (2,3,4).

All substances are not, of course, chromatographed by petroleum ether, and to overcome this difficulty the experimental material was dissolved in oleic acid which itself gives a single peak at 17.6°C.

It was found that characteristic peaks were still obtained, but in many cases these were rather small. The addition of a small amount of a saturated acid to the oleic acid markedly increased the size of the peaks without altering their appearance at characteristic positions along the temperature axis.

Although most saturated acids have this effect, palmitic acid gave the most satisfactory results and a solution of 2% palmitic acid in oleic acid (the carrier) was finally used.

The use of such a carrier allowed the effects of concentration to be assessed. It was found that each compound had a critical concentration at which the index peak was a maximum.

Thus, by making use of both the temperature and concentration effects it was possible to construct a diagram giving classification along the temperature axis and individual compounds along the concentration axis with peak height as a third dimension, and without separating the components of the mixture.

The third parameter, peak height, appears to be related chiefly to surface activity and, to a more limited extent, to concentration. With regard to surface activity, compounds more surface active than oleic acid suppress the carrier peak which remains intact for compounds less surface active, though here again, concentration, within the limits of the critical concentration, appears to play a minor role.

Table I gives the temperatures at which peaks are given by the main

polar groups likely to be found in oils and fats. Complexity of molecular structure may cause modifications of this simple pattern. If the compound is polyfunctional the peak area may lie across the relevant temperatures. Isomers may give peaks on opposite sides of the carrier peak.

Table I
Peak temperatures for various polar groups

Group	Temperature °C
COOH	15.6 16.9 18.1 18.8
CO	16.4
OH	15.8 (alcohols, enols, phenols)
Peroxides	15.0 – 15.3
Carrier	17.6

Although only a limited number of isomers have been examined, there is evidence that geometric, and sometimes optical, isomers give such an effect. Although there are four peaks for the carboxyl group, all acids do not give all of these ; some may be only minor peaks, or completely suppressed.

Some of these results for particular compounds are shown in *Table II*. The peaks occur at these positions whether the isomers are examined separately or as a mixture.

Table II
Effects due to molecular structure

Compound	Peak temperature
Maleic acid	16.9 only
Fumaric acid	18.8 only
Erythrodihydroxystearic acid	18.1 only*
Threodihydroxystearic acid	16.9 only*
Cis 1,2–dihydroxycyclohexane	15.8
Trans 1,2–dihydroxycyclohexane	18.5
Triphenylethyleneglycol (racemate)	15.8 19.5

*Racemates. No indication of peaks due to separate optical isomers.

Experimental

The full details of the method have been published elsewhere (4), but the essential points are as follows :—

Number one quality, 11 cm, circular filter papers were cut to give eight sectors with half length, narrow tongues between each sector, leaving a 1.5 cm disc in the centre.

These papers were dyed with a saturated solution of *Dispersol Fast*

Scarlet B in a 50/50 ethanol/ether solvent mixture to which was added 5 to 10% of a saturated solution of *Waxoline Green G* (C.I. Solvent Green 3.61565) in a similar solvent mixture. This latter dye is oil soluble, and there should be just sufficient of it to make the chromatographic band distinctly visible.

The carrier liquid was made by dissolving sufficient palmitic acid in the oleic acid to obtain a mixture with a setting point of 18°C and a liquifaction point of 23.5°C.

Not only did this method of preparation compensate for any small quantities of saturated acid present in the oleic acid as impurities, but the use of such a mixture, in addition to enhancing the effects which are the basis of the method, gave a liquid the viscosity of which could be so controlled by temperature as to make easy the application to the paper of a band of the desired width and uniformity.

The material to be chromatographed was applied as a continuous band, 1 to 2 mm wide, across the inner end of each sector. It was found that maintaining the carrier solution at 70–80°C gave the desired conditions for easy application. The paper was then placed in an oven at 70–80°C for approximately one minute, cooled, exposed in the chromatograph tank to the vapour of petroleum ether (boiling range 40–60°C) for not less than one and not more than two minutes, lowered so that the tongues, which had been bent at right angles to the main body of the paper, dipped into the petroleum ether, and development was allowed to proceed at constant temperature. The latter took only a few minutes. If the temperature altered more than 0.1°C the paper was rejected.

The outline of each band was then pencilled in, and the perimeter and area subsequently measured. Eight samples, one on each sector, could of course, be chromatographed at the same time.

For the purpose of the present investigation certain materials of interest to cosmetic chemists have been investigated, and three-dimensional chromatograms obtained covering a temperature range of 15 to 19.5°C and a concentration of the experimental material in the carrier liquid of from 1 to 8%.

The results are illustrated by *Figs. 1 to 5*. For the sake of clearness only those contours for index values of 40 and upwards are shown (except for *Fig. 2*, where some minor peaks are shown by dotted contours at index values of 30 and 35), the contour intervals being 5 units.

Points were however obtained for every 1% interval of concentration, and for approximately each 0.3°C interval of temperature, making 125–135

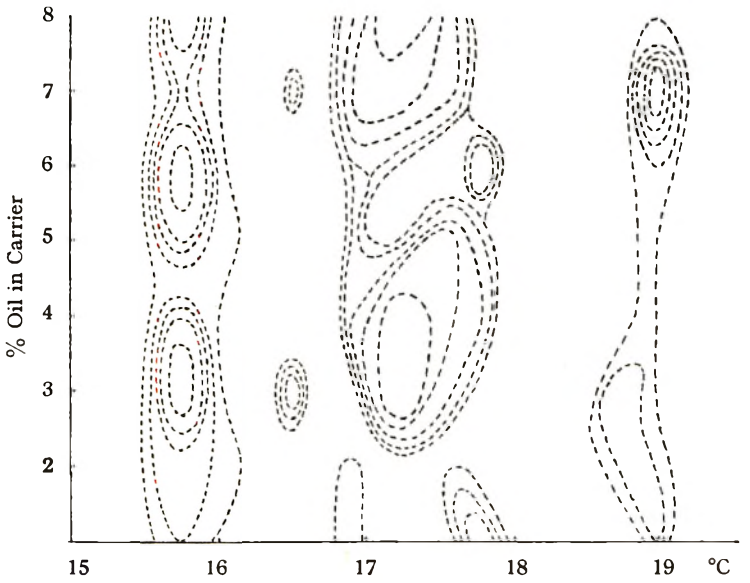

Figure 1
Olive oil. Index contours 40 (outer) x 5

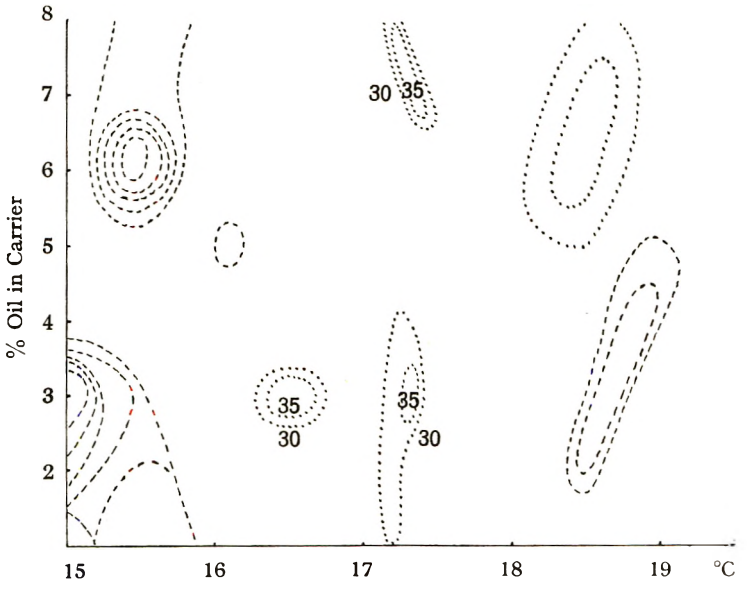

Figure 2
Pure mineral oil. Index contours 40 (outer) x 5

observations per diagram. The contours were then drawn, taking into account the disposition of all index values.

The amount of experimental material actually used per diagram is of the order of 0.05 g, and the accuracy with which the index could be repeated is within $\pm$ 5% of the actual index value.

The minimum concentration of a polar compound which will give a peak is difficult to estimate, and undoubtedly varies with the surface activity. Compounds having quite low surface activity gave peaks when present in a concentration of less than 1% in the experimental material, and it seems probable that compounds having a high surface activity would be detected at much lower concentrations.

Results and Discussion

Fig. 1 shows the type of diagram obtained for an oil consisting mainly of glycerides, in this case olive oil.

The series of peaks on the left of the diagram, at 15.8°C, must represent compounds with free hydroxyl groups, i.e. partially acylated glycerol. The two small peaks at 16.4°C are ketones, possibly produced by the oxidation of secondary hydroxyl groups.

The large, distorted, high index value area between 17 and 18°C is typical of the effect of very small quantities of polar substances on the carrier peak. This, in the absence of disturbing, polar groups, would be a narrow band across the diagram, parallel to the concentration axis at 17.6°C. This effect has been obtained with a very pure mineral oil.

The peak area to the right of the diagram, at 19°C, cannot be definitely identified. It is possibly due to isomers of compounds giving peaks in the lower temperature range.

The mineral oil, the diagram for which is shown in *Fig. 2*, has been solvent refined, but contains no antioxidants or other additives. The dashed contours are for index values of 40 and upwards, the dotted contours for 30 and 35. There are clear indications of peroxide formation (bottom left hand corner); of small amounts of hydroxy and ketonic compounds; of traces of the carrier peak, and possibly of hydroxy-acids (18–19°C), but, apart from the peroxides, the quantities are, as one would expect, very small.

The diagram for a sample of lanolin is shown in *Fig. 3*. Of the three types of material examined this represents the most complicated case. There are a large number of polar compounds, and all three types of polar group are represented. Some of the compounds are at least bifunctional

the tabs dip into the petroleum ether. Development takes place in less than 5 min so that there is no difficulty in maintaining the temperature, even with quite a crude type of thermostat, to within 0.1°C. The rod is then pulled up again, the paper removed, and the band pencilled in. The dye used is Dispersol Fast Scarlet B which has a $-CH_2.CH_2OH$ side chain attached to the amino group. This group is essential for the production of these large changes in band shape. If the –OH group is phenolic in character the changes are not produced. I have used this dye because it gives the simplest effect with the carrier mixture that I use, i.e. one single peak 35 units high at 17.6°C. Other dyes having a similar structure give multiple peaks.

In addition to the work described in the paper we have tried various phenols, all of which give a peak of 15.8°C whatever their nature. I am not suggesting that the method would detect such compounds present as antioxidants. I would not claim that it is so sensitive, and I think that the sensitivity, in fact, increases with the surface activity of the compounds present in the mixture. We made a series of fairly complicated molecules and one of the things we found was that if one has compounds with several polar groups, e.g. carboxyl, widely spaced over the molecule the peaks become flattened. The same thing happens when using dyes having surface active groups widely spaced in the molecule.

Although, using known compounds, the peak height is only about 35–40 units, irradiation of oils gives quite enormous peaks 80 to 90 units high. This means, of course, that a band of 1 in^2 area would have an outline 80 to 90 in long. These are quite large changes. One may think that the measurement of the outline and area of the band is not very accurate on such small bands and, of course, this is true, but I do not consider that the accuracy is any less than that involved in estimating the R_f value by orthodox chromatography. In most cases, the effects are so large, that there is no doubt where the actual peak occurs. One can exactly pinpoint the temperature and concentration at which the peak occurs, and one can reproduce it. I think that the ease of reproducing is shown by the fact that students after a few days' practice have no difficulty whatever.

DISCUSSION

MR. E. W. CLARK : Highly degraded or autoxidised lanolin invariably has greatly increased acid and peroxide values. Yet *Figs. 4* and *5* show reduced peaks for peroxides and for acids. Does this indicate that the method is not highly specific for the various group configurations and that it is more dependent on surface activity or some interaction between the sample and the carrier?

THE LECTURER : The point about surface activity is, I think, to a certain extent true, but the lanolin was degraded at 100°C and I think I am correct in saying that most peroxides decompose below this temperature. You will therefore get the decomposition products of the peroxides rather than the actual peroxides themselves.

MR. E. W. CLARK : Yes, but when the peroxide is decomposed, additional free fatty acids are formed and one might have expected that the peak due to the carboxyl group would then have been even more marked.

THE LECTURER : One of the peaks which is due to carboxyl groups occurs at 18.1°C. In *Fig. 4*, which is the degraded material, there is one sloping peak which covers the temperature 18.1°C, and two other peaks further up the diagram which are also at 18.1°C. It is true that one can get four peaks with carboxylic acids, but these do not always show for a particular acid ; one or the other of them may predominate.

MR. E. W. CLARK : Have you any comments to make on the small peak for peroxides in *Fig. 5* ?

THE LECTURER : The fact that the peak is right at the top of the diagram indicates that the concentration of peroxide is rather low. If one has a high concentration of peroxide, the peak will appear at the bottom of the diagram, but as the concentration falls the peaks move up the concentration axis. Even though this lanolin has been stored for ten years it suggests that there is no high concentration of peroxide.

MR. E. W. CLARK : Was the sample taken from the surface layer, or deep down ?

THE LECTURER : If the surface layer had been taken there would probably have been a much higher concentration of peroxide.

MR. J. D. CHESHIRE : Do you think that your method for group classification has any advantages for a laboratory already equipped with I.R. spectrophometers?

THE LECTURER : Yes, for a preliminary examination. If one is faced with a new sample, or a sample of a mixture which is suspect in any way, the method would be very useful when taken in conjunction with I.R. examination. I am not putting up this method as better in any way than established procedures where the analysis is straightforward. It does involve rather a lot of work as I have mentioned towards the end of the paper, and therefore would not be justified except in a particularly difficult case.

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The Activity of Antibacterials in Two-Phase Systems

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Presented at the Symposium on "Preservatives and Antioxidants", organised by the Pharmaceutical Society of Great Britain and the Society of Cosmetic Chemists of Great Britain, in London on 17th November 1964.

Synopsis—Bactericides in oil/water systems are partitioned between the two phases, the concentration in the aqueous phase being controlled by the overall concentration and the oil:water ratio. The antibacterial activity of the systems is determined both by these factors and by the enhanced concentration of bactericide at the oil/water interface. A change in temperature of the dispersions changes their antibacterial activity by virtue of the normal effect of temperature on bactericides, and by a change in the partition coefficient which may, in some cases, be appreciable.

During the past decade or so an increasing number of failures of so-called "preservatives" to protect creams from microbial spoilage has been reported. This period coincides with that during which, in general, there has been a change from the use of anionic emulsifiers to nonionic emulsifiers, and this reformulation unwittingly modified the resistance of the products to microbial attack. Many factors influence the effectiveness of a preservative in any formulation and there are some notable reviews on the subject (1,2,3,4).

Emulsions and creams contain many substances which collectively form excellent substrates for the growth of micro-organisms (5,6). Under some conditions the oil phase may be metabolised. Fungi have been reported to grow on fixed oils (7,8,9), and some oxidative and lipolytic bacteria can break down fixed oils and fats (10,11,12), while hydrocarbon oils can be

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metabolised by pseudomonads and other bacteria (13,14,15). Many emulsifying agents are liable to microbial attack. In low concentration many anionic emulgents can be used as energy sources (16,17), but the anionic soaps are normally used in creams in sufficient concentration to produce an unfavourably high pH for growth or indeed to be bactericidal. Sodium stearate is bactericidal at a concentration of 1 per cent, and other saturated and unsaturated fatty acid soaps at concentrations far below this level (18). Many nonionic emulgents, particularly the fatty acid ester type, can be utilized by a variety of organisms (19), but it is not certain whether this is due to the greater facility with which they can be metabolised or to their aqueous solutions being near to neutrality and less likely than aqueous solutions of anionic emulgents to interfere with the functioning of bacterial enzymes.

It is now well established that nonionic emulgents can inactivate a wide variety of commonly-used preservatives including the *p*hydroxybenzoates (20,21), and phenols (22). The mechanism of inactivation may be complex formation due to hydrogen bonding (22) or solubilization within micelles (23), the latter process being established in the case of inactivation of bactericides by anionic soaps (24,25,26).

Other important factors affecting the activity of preservatives in o/w systems are those controlling the availability of the preservative in the aqueous phase and in particular the o/w partition coefficient of the preservative, the phase-volume ratio and the temperature.

It has long been known that phenols dissolved in oils and fats possess no antimicrobial activity except when the oil is in contact with water (27), and that the inclusion of water in phenol ointment markedly increases the activity of the phenol (28,29). These observations indicate that the activity of phenol in the presence of both oil and water is controlled by its partitioning between the two phases. Indeed, Solution of Chloroxylenol B.P.C. which contains 5 per cent chloroxylenol has a Rideal-Walker coefficient of about 3, but 5 per cent chloroxylenol has been found inadequate to prevent mould growth in emulsified ointments (30). Whenever preservatives are more soluble in oil than in water, enough must be added to an o/w system to obtain a sufficient concentration in the aqueous phase (31). Thus a knowledge of the partition coefficient of the preservative is essential to ensure that the forementioned condition is met. A very useful table of partition coefficients of methyl *p*hydroxybenzoate in oils commonly used in creams has been prepared by Hibbott and Monks (32) but there is comparatively little data of this type in the literature.

Cutting oil emulsions used in the metal-working industry are particularly prone to microbial attack and attempts have been made to preserve them and eliminate a large annual financial loss. Pivnick and Fotopoulos (33), and others (34) found that the concentration of preservative needed varied with the oil : water ratio, an observation which must be linked with the larger bacterial population which can develop in cutting oil emulsions (1). Obviously the effectiveness of a preservative in an emulsion must be controlled both by its partitioning between the two phases, and the phase-volume ratio since both factors will determine the concentration in the aqueous phase.

Studies on the influence of temperature on chemical sterilization processes have been confined to aqueous solutions, but the literature reveals several studies on the influence of temperature on partition coefficients (31,35). In general, a rise in temperature produces an increase in the solubility of a preservative in both the oily and aqueous phases of an emulsion, but the partition coefficient must be presumed to change with temperature. Thus the overall effect of temperature on preservative activity in o/w systems will be determined, (i) by the normal influence of temperature on the disinfection rate, and (ii) by the alteration in the distribution of the preservative between the two phases.

The experiments described in this paper were undertaken to gain an understanding of the major physical factors controlling the activity of preservatives in o/w systems and, in particular, of the interactions between them. To this end, simple o/w systems containing no emulsifying agent were used. This permitted a study of the major controlling factors, but it is acknowledged that when, at a later stage, emulgents are included, our conclusions about the relative importance of each factor may have to be modified and certainly additional factors will be introduced. Our assessments of antimicrobial activity of the systems were, for expedience, made using *Escherichia coli* but it is conceded that the results might have been of greater interest had we elected to use a mould.

The Partition Coefficients

The very wide range of the partition coefficients of preservatives in some of the systems we have studied is shown in *Table I*. The range of coefficients is about 6600-fold or about 10 times the range recorded by Hibbott and Monks (32), for the partitioning of methyl *p*hydroxybenzoate between water and a variety of oils. The partition coefficients for arachis oil/water systems are high compared with those for liquid paraffin/water

Table I
Partition coefficient at 25°C of some preservatives in liquid paraffin (S.G. 0.830 to 0.870) and arachis oil

Preservative	Partition coefficient	
	Liquid paraffin/ water	Arachis oil/ water
Phenol	0.067	5.6
Chlorocresol	1.53	116.7
Thymol	—	447
Phenylmercuric acetate	0.23	—

systems. This is one reason why creams prepared with a vegetable oil are more difficult to preserve than those prepared with a mineral oil.

The concept of the partition coefficient is universally understood. It is usually determined in a static system, and it is not always appreciated that it is the interaction between partition coefficient and phase-volume ratio which determines the concentration of a preservative in the two phases of an o/w system (*Table II*).

Table II
Influence of partition coefficient and phase-volume ratio on concentration of preservative in aqueous and oil phase of a two-phase system

	$K°_w$ at 25°	Oil/water ratio	0.2	1.0	2.5	5.0	10.0
0.4% w/v phenol in liquid paraffin/water dispersions	0.067	Preservative in oil %	0.031	0.050	0.080	0.080	0.176
		,, ,, water %	0.474	0.750	1.199	1.799	2.636
1% hypothetical preservative	1.000	,, ,, oil %	1.000	1.000	1.000	1.000	1.000
		,, ,, water %	1.000	1.000	1.000	1.000	1.000
4.0% w/v chlorocresol in arachis oil/water dispersions	116.7	,, ,, oil %	22.96	7.93	5.60	4.79	4.40
		,, ,, water %	0.197	0.068	0.048	0.0411	0.038

For any given overall concentration of preservative the concentration in the aqueous phase may be calculated from the expression

$$C_w = C \frac{(\emptyset + 1)}{(K°_w \emptyset + 1)}$$

where C_w = concentration in the aqueous phase % w/v
C = overall concentration % w/v
$K°_w$ = oil:water partition coefficient
$\emptyset$ = oil:water ratio

When the partition coefficient is less than 1.0, the majority of the preservative is in the aqueous phase and an increase in the oil:water ratio increases the aqueous phase concentration. When the partition coefficient is greater than 1.0, most of the preservative is in the oil, and an increase

in the oil:water ratio reduces the concentration in the aqueous phase. It is only when the partition coefficient is exactly 1.0–and in practice this is only very rarely, if ever, the case–that changing the oil:water ratio has no effect on the preservative concentration in either phase, but as will be indicated later, this may still influence the antimicrobial activity of the system.

When the partition coefficient is very close to 1.0 the inclusion of an amount of oil within a system with a given overall concentration of preservative has only a very small effect on the aqueous phase concentration, but when the coefficient is far removed from unity the inclusion of a similar volume of oil has a considerable effect on the aqueous phase concentration. For example, the concentration of phenol in the aqueous phase of a liquid paraffin/water dispersion ($K°_w = 0.067$) having an oil:water ratio of 10.0:1.0 is 6.6 times that of an aqueous solution of phenol of the same overall concentration. The concentration of chlorocresol in the aqueous phase of an arachis oil/water dispersion ($K°_w = 116.7$) having the same oil:water ratio is only $\frac{1}{153}$ that of the aqueous solution with the same overall concentration. For dispersion having an oil:water ratio of 0.2:1.0 the corresponding changes are 1.2-fold and 1/20.4-fold respectively.

Thus when selecting a compound for study as a possible preservative for a product, both the partition coefficient, and the proportion of oil in the product must be considered.

The Interfacial Factor

The selection of a preservative for a two-phase system fixes one parameter of the system, i.e. the partition coefficient. The antimicrobial activity of the preservative is then determined by the aqueous phase concentration which is controlled by the overall concentration and the oil:water ratio.

The activity, expressed as the extinction time, is related to the oil:water ratio by the expression

$$\log t = A - p\sqrt{\emptyset}$$

where t = extinction time

A = constant

$\emptyset$ = oil:water ratio

p = slope of regression log extinction time on $\sqrt{\emptyset}$

Since the concentration of antibacterial in the aqueous phase is related to $\emptyset$ and, therefore, to $\sqrt{\emptyset}$, the slope of p of the regression of log extinction

time on $\sqrt{\phi}$ is analogous to the concentration exponent of bactericides in aqueous solution, and is called the *phase-volume* coefficient.

Our early experiments with *E. coli* showed that a further factor was of greater significance in determining the activity than had hitherto been presumed. A quantity of liquid paraffin was included within a 100 ml volume of inoculated aqueous phenol solution, the necessary adjustments being made to restore both the aqueous phase concentration and the number of organisms/ml to that of the original aqueous solution. The two-phase system was slightly but significantly more antibacterial than the aqueous solution. We have shown (36) that a further increase in the oil:water ratio considerably increased the activity (*Fig. 1*). The only difference between the aqueous solution and the two-phase systems was

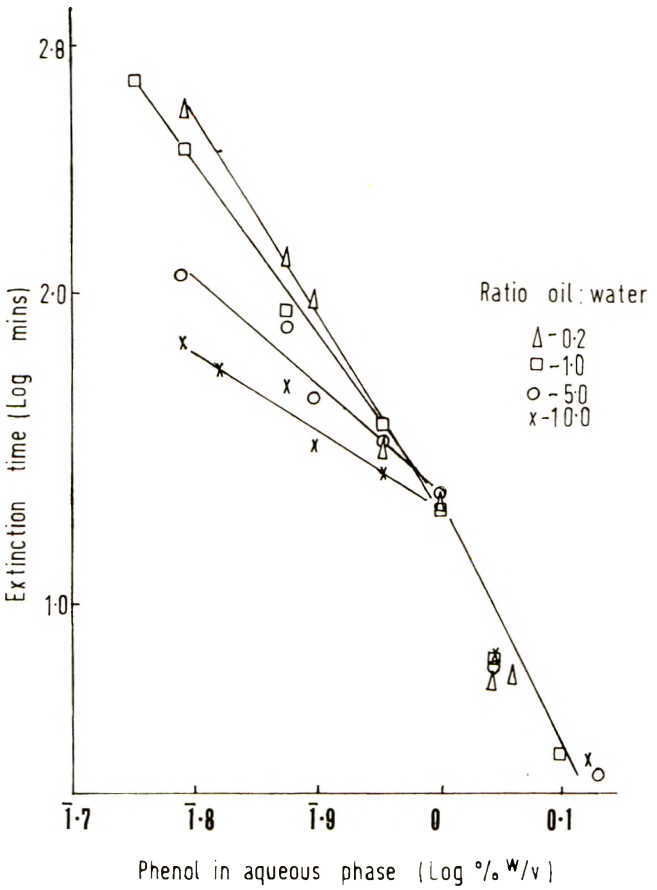

Figure 1. Activity against *E. coli* of liquid paraffin—phenol—water mixtures

the volume of oil contained within the latter and the oil/water interface created by the latter. Photographs show that a proportion of the organisms are adsorbed at the interface and, indeed, probably help to stabilize it. The o/w systems containing bacteria were shaken intermittently, and it is possible that organisms which at one moment were in the bulk aqueous phase were at another adsorbed at the aqueous side of the interface.

It may also be deduced from interfacial tension measurements and the Gibb's adsorption equation, that the concentration of phenol at the interface is greater than in the bulk of the aqueous phase. What is uncertain is precisely how the phenol molecules and the organisms are aligned at the interface, but it may be assumed that the molecules at the interface penetrate the aqueous phase to a smaller depth than do the cells. It is therefore probable that part of the bacterial surface at the interface is in contact with a higher concentration of phenol, but this is certainly not true for the whole cell. A complicating factor is the concentration of phenol in the oil phase which is always related to that of the aqueous phase (*Table II*), and may behave as a reservoir for the latter. Our knowledge of all the factors operating to control the activity of preservatives in two-phase systems is undoubtedly far from complete. Nevertheless, the role played by the interface in determining the antimicrobial activity of a system seems too real to be ignored and has, in fact, been underlined by all the subsequent experiments we have carried out.

An analysis of 400 experiments arranged in a randomized block design to determine the importance of the concentration of bactericide in the aqueous phase, and of the oil:water ratio in determining the antibacterial

Table III Analysis of variance of extinction-time data for *E. coli* in phenol/liquid paraffin/water dispersions at 25°

Source of variation	Sums of squares	°F	Variance	F	P
Between oil-water ratios (R)	404730.48	6	67455.08	$\frac{6}{358}$ 409.94	<0.001
Between aqueous phase phenol concentrations (P)	1945585.19	5	389117.04	$\frac{5}{358}$ 52364.73	<0.001
Interaction (RP)	865299.78	30	28843.33	$\frac{30}{358}$ 175.286	<0.001
Residual	58910.19	358	164.55		
Total	3274525.64	399			

activity of the systems again indicated the importance of the interface. An analysis of variance of the data is shown in *Table III*.

The two sources of variation shown are the only two assignable causes of variation in the extinction times, and both are highly significant ($p = <0{\cdot}001$). The F-ratio indicates that the concentration of phenol in the aqueous phase has a greater effect in determining extinction time than does the oil:water ratio. This conclusion is in line with that reached by intuitive reasoning since for any given overall concentration of phenol, the oil:water ratio controls the phenol concentration in the aqueous phase, and it is the latter which largely determines activity. Of considerable interest is the interaction item which, when tested against the residual, is highly significant. It is sometimes difficult to understand the meaning of interactions in analyses of variance, but in this case the interaction between oil:water ratio and the concentration of phenol in the aqueous phase is in all probability the interfacial effect. Thus the statistical analysis of the data supports the conclusion derived from direct experimentation but additionally permits some estimate of importance to be assigned to the interface. Though highly significant it is statistically the least important of the three factors determining activity ; the magnitude of its effect is dependent on the other two factors.

The Effect of Temperature

The effect of temperature on the activity of bactericides in o/w systems may be complex. Any temperature in excess of about 50°C will, *per se*, produce the death of vegetative cells either by protein coagulation or enzyme inactivation. There are, additionally, the indirect effects of temperature on the partition coefficient, and on the velocity of the bactericidal reaction.

Our experiments studied the effects of temperature over the range 5–45° and revealed that the partition coefficient of some preservatives increases, and for others falls, as the temperature is raised (*Table IV*).

Table IV Changes of partition coefficients as temperature of oil/water dispersions is increased from 5–45°C.

Preservative	Oil phase	Approximate ratio $\frac{K°_w \text{ at } 45°}{K°_w \text{ at } 5°}$
Phenol	Liquid paraffin	260 per cent
Chlorocresol	Liquid paraffin	170 ,, ,,
Chlorocresol	Arachis oil	103 ,, ,,
Phenylmercuric acetate	Liquid paraffin	50 ,, ,,

Even if the four preservatives referred to in *Table IV* all had the same intrinsic antimicrobial activity and the same temperature coefficient in aqueous solution–and they certainly have not–the big differences in the change of partition coefficients with temperature change would inevitably produce big differences in the preservative ability of the compounds in two-phase systems during storage at different temperatures. And yet how often are such parameters determined during the formulation of two-phase systems ?

The bactericidal activity of phenol in liquid paraffin/water dispersions,

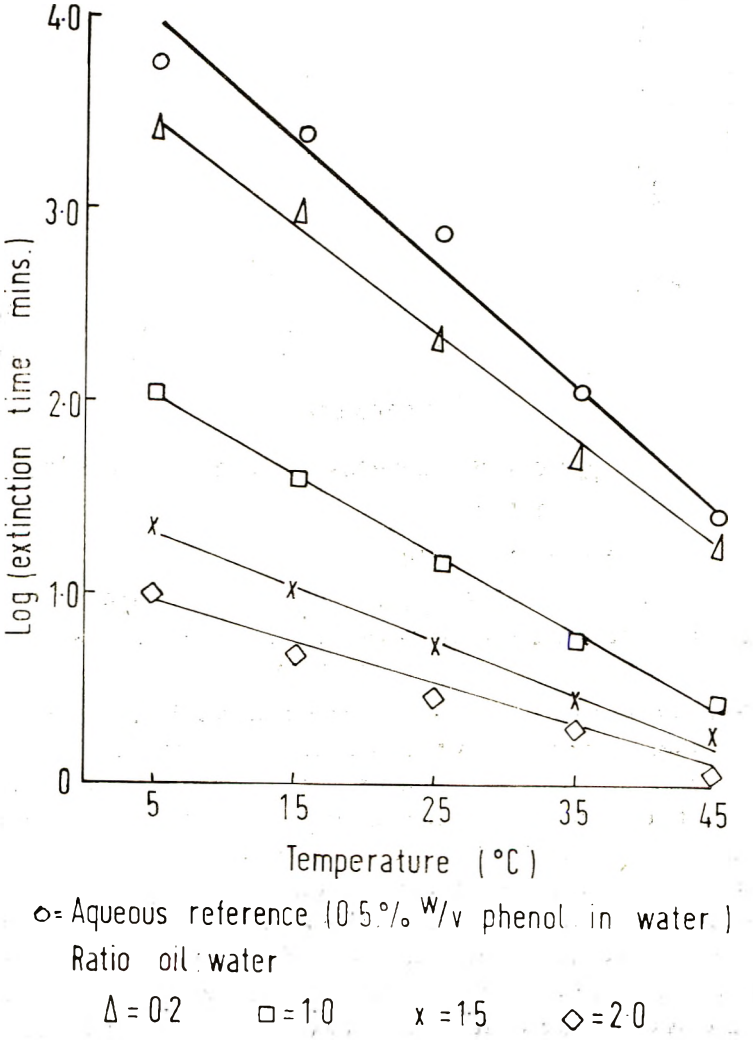

Figure 2. Activity against *E. coli* of 0.5% w/v overall concentration of phenol in light liquid paraffin—water dispersions

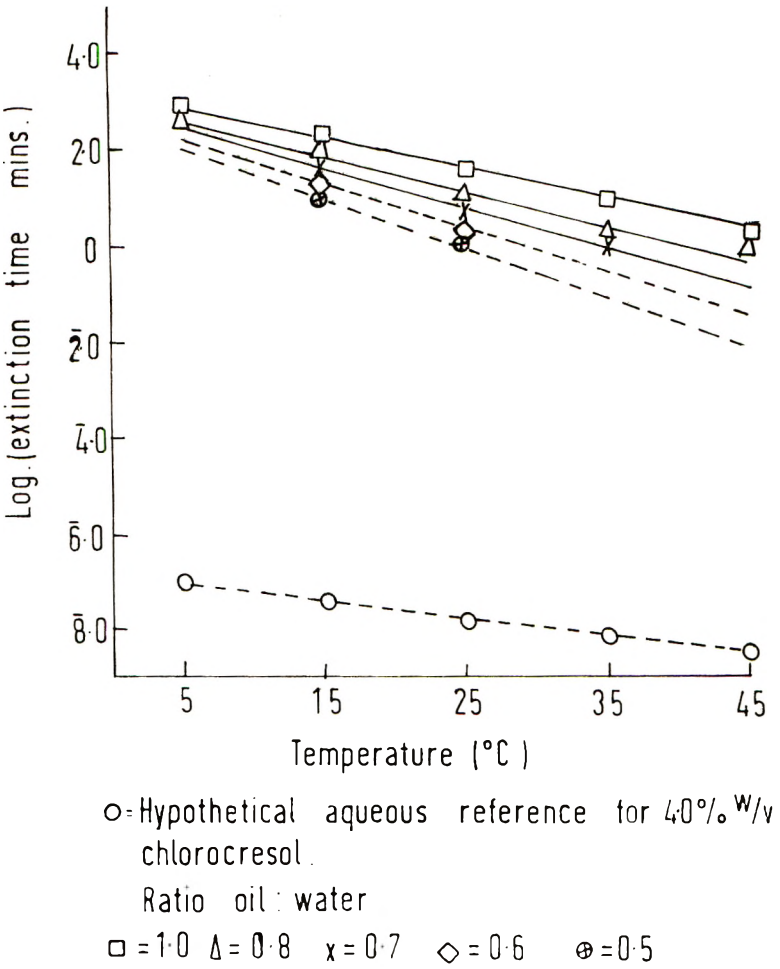

Figure 3. Activity against *E. coli* of 4.0% w/v overall concentration of chlorocresol in arachis oil—water dispersions

and chlorocresol in arachis oil/water dispersions has been examined (37,38) and is shown in *Fig. 2* in which reference lines for aqueous solutions having the same overall concentration of bactericide are also shown. The aqueous reference for chlorocresol in *Fig. 3* is, and can only be, a hypothetical line because the 4 per cent chlorocresol necessary to produce measurable activity in the dispersions exceeds by far the limit of aqueous solubility. It is immediately observable that at all temperatures at which observations were made, the dispersions of phenol in liquid paraffin and water are more active, and the dispersions of chlorocresol in arachis oil and water are less active than the aqueous reference. This difference is

a manifestation of the partition coefficients (*Table I*). The slopes of the regression lines of log extinction time on temperature are log temperature coefficients, and as the oil:water ratio is increased the temperature coefficients are decreased. That is, the activities of oil:water dispersions containing preservatives are influenced less by temperature changes than is an aqueous solution of the same preservative. The temperature coefficient for a dispersion containing a given overall concentration of preservative is related to that of the aqueous reference solution of the same concentration by the expression

$$K = \frac{\log \theta_a - \log \theta_s}{\emptyset}$$

where θ_a = temperature coefficient of aqueous solution

θ_s = temperature coefficient of dispersion

$\emptyset$ = oil:water ratio

K = constant for each preservative/oil/water system

Since the temperature coefficients of the dispersions decrease with increasing oil:water ratio, it is possible by using the constant *K* to calculate a ratio at which the temperature coefficient is 1.0, and the antibacterial activity of the dispersion is independent of temperature. For the very few systems studied so far, this ratio has been of the order of 3.0 to 3.5 and in two cases it was possible to verify this value experimentally. The reason for this unique ratio is as yet uncertain but it is possibly determined by the concentration of preservative at the oil/water interface. It is hoped that further experiments will help to establish this.

The conclusion which must be drawn from the experiments is that it is utterly futile to spend time and labour formulating a cream, and as a final gesture add an *ad hoc* concentration of a preservative. The formulation must be approached *ab initio* from the points of view of both physical and microbiological stability. The two aspects cannot be divorced one from the other, and any attempt to do so is an invitation to failure.

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DISCUSSION

MR. S. A. HILL : One of the most interesting points brought out in these experiments of the simple two-phase systems is that the interface, although the least important of the factors affecting the preservative activity, does have statistically a highly significant effect. I agree that, in the emulsion systems which you will eventually be investigating, other factors affecting preservative activity will certainly arise. I would like to know if, on the basis of the results gained so far, you would anticipate that the interface could become more important than either the phase/volume ratio or the partition coefficient of the preservative owing to the very large interfacial area in emulsions.

DR. H. S. BEAN : In emulsified systems the oil/water ratio will control the concentration of preservatives in the aqueous phase and will always be an important

factor. Phenol or preservative molecules are absorbed at the interface together with bacteria, and also emulgent particles, otherwise no emulsion would be obtained.

Exactly what will happen to the activity is uncertain especially as some of the surface active molecules will be absorbed on the bacterial surface. At the bacteria/water interface there will be a build up of micelles and some of the preservative may go into a solution in the interior of the micelles thereby changing its availability to the micro-organism. At the moment it is very difficult to see how all these factors are going to interact.

DR. E. E. BOEHM : In page 16 there is the statement "it has long been known that phenols dissolved in oils and fats possess no antimicrobial activity except when the oil is in contact with the water". This statement is not in agreement with the experimental results obtained by Sabalitschka and Priem (39) where it was shown that a 10% solution of phenol in oil, in the absence of water, kills Staphylococci within 7 hr. On mixing with 2% water the same organism was killed within 50 min. Under otherwise similar conditions a solution of 2% phenol in water killed this organism within 5 min. In fact, phenol dissolved in oil in the absence of water was about 20 times less effective, and in the presence of traces of water about 10 times less effective than an aqueous solution of phenol. Resorcinol in oil, under similar test conditions, appeared to be about 10 times weaker than an aqueous solution of the same concentration, but after mixing with 2% water was twice as strong as an aqueous solution of the same concentration. The authors explained this in terms of the different distribution of both phenols between the oil and water.

THE LECTURER: Our observations are in no way different from those of Sabalitschka; the difference lies in the interpretation. Prof. Sabalitschka grew his organisms on agar and they were not dried organisms. There was a proportion of water there and the preservative in the oil was partitioning into this small quantity of water. When he added water to 2% the activity increased, which is absolutely compatible with the statement made in our paper. He reduced the phase volume ratio, and therefore increased the preservation concentration in the aqueous phase indicating that he was using a system in which the partition coefficient was high.

DR. N. D. HARRIS : It seems to me that one of the real problems in this discussion is whether one can decide what the antibacterial activity of materials is in oil?

THE LECTURER : Yes, of course, this is one of the difficult things, which no one has solved satisfactorily so far. I think Professor Bullock was getting very near it but he had to use a factor to allow for a very high mortality in his organic solvents.

MR. R. SMART : We have infected arachis oil by spraying a water suspension of spores or cells into air, and "sampling" volumes of the air by a slit-sampler. If the usual nutrient agar plate is replaced by a plate containing a membrane filter moistened with oil, the cells adhere to the oil layer. The cells can be taken into a volume of oil by inverting the membrane in a filter holder and washing with more oil.

To estimate the number of viable cells on a membrane filter the latter is placed in the filter holder the right way up, washed with about 10 ml polyethylene glycol 300 10% v/v solution in water and cultured in the usual way.

The number of cells impinging on the membrane can be estimated in the slit sampler by counting the colonies produced on a nutrient agar plate in the same area as the membrane filter.

(39) Sabalitschka. T. and Priem, A. *Fette Seifen Anstrichmittel* **46** 277 (1939).

PROF. M. DONBROW : The materials dealt with are all of a semipolar nature and might be expected to dimerise in the oil phase, so that the partition coefficient would not be constant. This does introduce the problem of calculating partition coefficients for each concentration and each phase volume which is being studied.

THE LECTURER : In the aqueous phase one is likely to have dissociation, and association is likely to occur in the oil phase. If association were occurring in the oil then our partition coefficient would have been wrong and therefore our estimate of the phenol in our aqueous phase would have been wrong because a greater quantity would have gone into the oil. We would have picked this up by the position of our time survivor curves because the kill would have been very much slower than we had anticipated. In fact, our estimates of extinction time came very close to the observed extinction times.

As for dissociation in the aqueous phase, we know that the pKa of these phenols is in the region of 10. We were working at about pH 6.5—7 so there was not much dissociation.

MR. A. H. FENTON : Have you considered the addition of a material such as propylene glycol which, when added to the aqueous phase, would increase the concentration of the preservative?

THE LECTURER : We have examined a number of systems in which additives both increase and decrease the partition coefficient. The activity goes roughly as one would expect. We have looked at glycols and at the effect of salts, because the latter is of interest in sterilisation. The process of heating with a bactericide depends in part upon the shift of the partition coefficient in favour of increased concentration of bactericide in the bacterial cell.

MR. J. T. REES : You state that cream formulations must be approached, from the start, from the points of both physical and microbiological stability. We have seen that the partition coefficient effect is only one of several factors which will influence preservative efficiency. These other factors may in fact be stronger than the above partition coefficient effect. The method of packaging, and the type of pack employed, will also influence the rate of microbial growth. A point is reached, however, when the preservative of choice must be tested. Such testing should relate to conditions closely approximating those found in practice. To be of use to the development chemist the tests should also yield the required information in a short time. The method should also be simple to operate.

Serial dilution methods have been described by several authors. Conditions here, however, can hardly be described as relating to those found in the cream itself. The agar cup-plate method is also well known; the cream, containing the preservative, is placed in a central cavity in a seeded agar plate. These conditions again do not approximate closely to the true ones. A water soluble preservative may diffuse from the cream into the surrounding agar. The oxygen balance of the system must also differ markedly from that found in a normal cream jar or collapsible tube.

Other authors have proposed inoculating the sample itself with the test organisms, incubating, and later plating the samples for colony counts. This method is both lengthy and complicated.

I agree that cream development should embrace both physical and microbiological testing from the start but I am not sure that present test methods are always appropriate.

THE LECTURER : There are some guiding rules when one starts looking for a suitable preservative. If you plot phase volume ratio against the reciprocal of the fraction of preservative in the aqueous phase you get a linear regression line the slope of which depends upon the partition coefficient. If the partition coefficient is low, a change in oil/water ratio will not materially alter the proportion of preservative in the aqueous phase. If you have selected a preservative with a very low slope you can assume that you are going to have some activity in your product. But if you went to the other extreme, and selected a preservative with a high partition coefficient, as you change your phase volume ratio the proportion of preservative in the aqueous phase falls very rapidly. I presume you would start off in selecting a preservative with the aid of a graph and then calculate the concentration in the aqueous phase because the activity resides there. Your final product can be tested by inoculating and including a dye, which changes colour with changes in metabolic by-products from the organism, or just with a change of oxidation reduction potentials. After 18 hr some change in the indicator colour in the system could be seen long before it is possible to detect growth by inspection. With a semi-solid cream it might be perhaps several weeks before one detected mould growth.

MRS. D. WEDDERBURN : Short term product inoculation tests can be misinterpreted, for although they will reveal those preservatives which are *not* likely to be active, they cannot be used to predict those which *will* be effective. I say this because it is known that over a long period of storage certain organisms, particularly the *Pseudomonads*, can adapt themselves to their environment. I have experience of this. On one occasion, during a prolonged product inoculation test, for the first four or five months the preservative seemed to be holding the inoculation in a quiescent state, but after six months vigorous growth began and deterioration of the emulsion set in.

For this reason I see no substitute for long term tests which provide the opportunity for adaptation to be detected.

DR. J. B. M. COPPOCK : May I draw attention, from the food field in synthetic creams and even margarines and butter, that freeze-dried organisms do not necessarily behave, indeed often multiply faster, than organisms in their natural habitat. Thus I would not accept tests on o/w, or other systems, using freeze-dried organisms alone but would still require the more classical "suck it and see" experiment more related to practical conditions.

MR. G. SYKES : I agree wholly with Mrs. Wedderburn. This is one of the occasions in which the microbiologist must not be rushed. Short term tests will give an approximate result, but this is not good enough for commercial products. Adaptations can take place rapidly in a generation or two, or very slowly during several weeks, and it is the latter which one has to watch. It can only be controlled by long term observations. There is also the question of the types and strain of organism which may be involved.

MR. D. W. POXON : It does strike me that of the factors you were talking about, the interfacial phenomena were of considerable importance. If your micro-organisms are absorbed at the interface, surely the antibacterial, which was also strongly absorbed at the interface would be most effective.

THE LECTURER : This is correct.

MR. D. W. POXON : Is there any relationship between surface activity and antibacterial activity?

THE LECTURER : We are not quite sure how the molecules are orientated at the interface, but we shall assume that the polar portion of the molecule is in the water, and the non-polar is in the oil. They will only be measurable in Ångstroms whereas your cell is going to be microns in diameter. The organism is going to project into the aqueous phase very much further than the molecules, and it would seem that possibly only a portion of the cell surface is in contact with the higher concentration of bactericide or preservative. It is possible and, indeed very likely, that absorption of preservative molecules does also occur at the cell-water interface.

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The Behaviour of Lanolin Derivatives in Pressurized Formulations II

A. HERZKA*

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Synopsis—The solubility of ten *Golden Dawn* lanolin derivatives in seven different propellant/solvent systems, stored for six weeks at 0°C and 20°C, is described. The results indicate (1) that the solubility of four derivatives decreases on storage, (2) that the solubility of one derivative increases on storage, (3) that the solvent power of six of the propellant systems decreases on storage, and (4) that storage at 0°C does not materially affect the solubility of eight of the tested products.

Introduction

Having established the solubility and spray performance of ten lanolin derivatives in nine different propellant and propellant alcohol systems (1), further experiments were carried out with propellant/liquid paraffin systems, and with propellant/aqueous alcohol systems. The performance of these various compounds in a butane/aqueous alcohol system was also tested. The tests were carried out at 0°C and 20°C as previously (1). The following *Golden Dawn* products were tested :

i. Anhydrous lanolin, B.P.
ii. Wool alcohols, B.P.
iii. Liquid lanolin "A.C.E."
iv. Alcohol soluble lanolin.
v. Liquid lanolin "50 Super."
vi. Liquid lanolin "I.S.O."

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vii. Liquid lanolin "R.I.C.2."
viii. Liquid lanolin "L.I.N."
ix. Water soluble lanolin "75."
x. Water soluble wool alcohols "20."

Full details concerning these products are to be found elsewhere (1).

Experimental

The following systems were tested :

K — propellant 12/liquid paraffin B.P.* (2:1 w/w)
L — propellant 114/liquid paraffin, B.P.* (2:1 w/w)
M — propellants 11/12/liquid paraffin, B.P.* (1:1:1 w/w)
N — propellants 11/12/liquid paraffin, B.P.*/IMS** (1:1:1:1 w/w)
O — propellant 12/IMS**/water (1:5:3 w/w)
P — propellant 114/IMS**/water (1:10:5 w/w)
Q — butane mixture (40 psig)/IMS**/water (1:10:5 w/w)

*liquid paraffin, B.P.—S.G. 0.885.

***industrial methylated spirits, DM7 grade, 74 o.p.
Density at 15.6°C=0.7974. American proof 98.9.

Systems *O* and *Q* give homogeneous solutions at 20°C. System *P* gave a homogeneous solution at 20°C. After six weeks' storage at 0°C, however, separation into two liquid layers had taken place, i.e. a small second liquid layer had formed at the bottom of the container.

5% by weight of each product was predissolved in each solvent system, with heating where necessary. All filled dispensers were allowed to stand for 1 hour at 20°C before being examined for solubility. The results observed are detailed in *Table I.*

Dispensers filled with each variable were re-examined at 6 weeks' storage at both 0°C and 20°C, in the following manner :

Dispensers stored at 20°C

(1) Visible assessment of solubility.

(2) Assessment of sprayability, and observation of valve blockage, if any.

(3) Spray characteristics, if considered significant.

(4) As shaking before spraying is common with water-based formulations, the ease of re-dispersion–by shaking–of any separated matter was examined, particularly in systems *O*, *P* and *Q*.

Dispensers stored at 0°C

(1) Visible assessment of solubility at 0°C, and after being allowed to stand for 24 hours at 20°C.

(2) Assessment of sprayability, and observation of possible valve blockage, at 0°C, and after being allowed to stand for 24 hours at 20°C.

(3) Spray characteristics, if considered significant.

(4) Ease of re-dispersion, by shaking, of any separated matter, particularly in systems *O*, *P* and *Q*.

Containers and valves

Plastic coated glass containers were utilised for all tests, fitted with Precision valves of British origin, ref. 1792. Specification of valves (international part nos.) : 14/23/12/3/standard buna/6/19/2. There was no definite preference for Precision valves, but other valves suitable for glass containers were not in stock when these experiments were commenced.

Results

Table I Solubility of 10 lanolin products after 1 hour at 20°C.

Lanolin products	Propellant systems							Total
	K	L	M	N	O	P	Q	
i	2	2	1	1	4	5	4	19
ii	3	2	1	1	5α	6	4	22
iii	1	1	1	1	4	4	4	16
iv	4	5	2	1	5	5	4	26
v	1	1	1	1	5	5	5	19
vi	1	1	1	1	5	5	6	20
vii	1	3	1	1	5	5	6	22
viii	1	1	2	1	5	5	4	19
ix	4	4α	4α	3	2	2	2	21
x	4α	4α	4α	1	2	2	2	19
Total	22	24	18	12	42	44	41	

Key to Tables I, II and V.

1 = Soluble, no precipitate.
2 = Soluble with negligible precipitate (visible only on close scrutiny).
3 = Soluble with slight precipitate (readily seen on inverting bottle).
4 = Partially soluble, with either a dense precipitate or an insoluble liquid layer floating on the surface.
5 = Partially soluble, with either a dense precipitate or an insoluble liquid layer at the bottom of the container.
6 = Totally insoluble.
α = Agglomeration of precipitate.
β = Crystal formation.

Table II Solubility of 10 Lanolin Products after 6 weeks

Lanolin products	Propellant systems																				Overall Total	Average
	20°C								0°C								0°C + 24 hours at 20°C									
	K	L	M	N	O	P	Q	Total	K	L	M	N	O	P	Q	Total	K	L	M	N	O	P	Q	Total		
i	3	3	3	1	5	5	4	24	3	4α	5	5	5	6	4α	32	3	3	3	1	5	6	4α	25	81	27
ii	4	4	3	1	5β	5β	4α	26	4	4α	3	1	5β	5	4α	26	4	4	3	2	5α	6αβ	4	27	79	26.3
iii	3	3	1	1	5	5	4	22	2	3	1	1	5	5	4	21	2	4	1	1	5	5	4	22	65	21.7
iv	4	4	4	1	5	5	4	27	4	4	4	1	5	5α	4	27	4	4	3	1	5	6	4	27	81	27
v	1	1	1	1	5	5	4	18	1	2	1	1	5	5	4	19	1	1	1	1	5	5	4	18	55	18.3
vi	1	1	1	1	5	5	6	20	1	1	1	1	6	6	6	22	1	1	1	1	6	5	6	21	63	21
vii	1	2	1	1	5	5	4	19	1	4	2	1	6	6	6	26	1	4	2	1	5	6	4	23	68	22.7
viii	1	1	1	1	5	5	4	18	1	1	1	1	6	6	6	22	1	1	1	1	5	6	4	19	59	19.7
ix	5	6α	4	5	2	2	2	26	4α	6α	4	5	3	3	3	28	4	4α	4α	5	3	2	3	25	79	26.3
x	4α	4α	4α	3β	3	3	2	23	4α	4α	4αβ	3	2	3β	2	22	4α	4α	4α	3	2	3	2	22	67	22.3
Total	27	29	23	16	45	45	38		25	33	26	20	48	50	43		25	30	23	17	46	50	39			

Table III Spray results after 6 weeks

Lanolin products	Propellant systems																			
	20°C							0°C								0°C + 24 hours at 20°C							
	K	L	M	N	O	P	Q	K	L	M	N	O	P	Q	K	L	M	N	O	P	Q
i	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
ii	S	S	S	S	S	S	Sf	S	S	S	S	S	U	S	S	S	S	S	S	Sf	Sqf
iii	S	S	S	S	S	S	S	S	S	S	S	S	U	S	S	S	S	S	S	S	S
iv	S	S	S	S	S	Sqf	Sqf	S	S	S	S	S	U	S	S	S	S	S	S	Sqf	Sqf
v	S	S	S	S	S	U	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
vi	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
vii	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	Sf	S	S
viii	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
ix	S	U	S	S	S	S	S	S	U	S	S	S	S	S	S	S	S	S	S	S	S
x	Sqf	S	S	S	S	S	S	Sqf	U	S	S	S	S	S	S	Sqf	U	S	S	S	S	S

Key: S = satisfactory spray
U = no spray
f = stable foam on impingement
qf = quick-breaking foam.

Table IV Propellant systems

	Propellant systems						
	K	L	M	N	O	P	Q
Overall total	77	92	72	53	139	145	120
Average	25.7	30.7	24	17.7	46.3	48.3	40

Table V Appearance of controls after 6 weeks

Propellant system	20°C	0°C	0°C + 24 hours at 20°C
K	1	1	1
L	1	1	1
M	1	1	1
N	1	1	1
O	1	1	1
P	1	5	5 (irreversible)
Q	1	1	1

Table VI Effect of shaking (on separated matter) after 6 weeks' storage.

Lanolin products	Propellant systems													
	20°C							0°C + 24 hours at 20°C						
	K	L	M	N	O	P	Q	K	L	M	N	O	P	Q
i	½f	½f	–	–	x	x	x	1½f	1½f	1½f	–	x	x	x
ii	1	1½	1	–	½	½	x	1½	1½	1½	–	x	x	½
iii	–	–	–	–	1	½	1½	1½f	x	–	–	x	x	1
iv	–	x	x	–	½	½	1f	1½	½f	1½	–	½	x	½
v	–	–	–	–	¼	x	½	–	–	–	–	x	x	1
vi	–	–	–	–	1	x	½	–	–	–	–	½	1½	½
vii	–	2f	–	–	x	1½	½	–	2f	2f	–	x	1	x
viii	–	–	–	–	½	1	1	–	–	–	–	½	½	½
ix	1f	xf	xf	1½f	1½	1½	1½	2f	½f	1f	1½f	2f	2f	2f
x	x	xf	xf	xf	1½f	1½f	1½	1½f	xf	½f	½f	4f	xf	4

Key: f = foaming inside container after shaking.
x = separated matter does not redisperse.
Separated matter redisperses.
¼ = Stability of 25 seconds.
½ = ,, ,, ½ minute.
1 = ,, ,, 1 minute.
1½ = ,, ,, 1½ minutes.
2 = ,, ,, 2 minutes.
4 = ,, ,, 4 minutes.

Summary

After 1 hour at 20°C

Liquid paraffin, B.P. (Systems K–N; Table I)

The solubility of the lanolin products is fair to good, with the exception of alcohol solution lanolin (iv), water soluble lanolin "75" (ix), and water soluble wool alcohols "20" (x). The addition of industrial methylated spirits effects complete solution of all products, with the somewhat surprising exception of water soluble lanolin "75" (ix). The different effects of the various propellant systems are not as marked as might have been expected from the results obtained previously (1). In two instances—wool alcohols, B.P. (ii) in the propellant 12/liquid paraffin, B.P. system (K), and liquid lanolin "L.I.N." (viii) in the propellants 11/12/liquid paraffin, B.P. system (M), the results are contrary to expectation.

Water (Systems O–Q; Table I)

The solubility of the lanolin products is poor in the water-based systems with the exception of water soluble lanolin "75" (ix), and water soluble wool alcohols "20" (x), but even the last two products are not completely soluble. The system containing propellant 114 (P) is inferior to that containing propellant 12 (O) with only one product, wool alcohols, B.P. (ii). Replacing the halogenated propellants with butane does not have a consistent effect. With three products—wool alcohols, B.P. (ii), alcohol soluble lanolin (iv), and liquid lanolin "L.I.N." (viii), the solvent power of system *Q* (butane/IMS/water) is superior to that of system *O* (propellant 12/IMS/water) and system *P* (propellant 114/IMS/water). With two products, liquid lanolin "ISO" (vi) and liquid lanolin "R.I.C.2" (vii), its solvent power is inferior to those of systems *O* and *P*. With four of the products its solvent power is equal to those of systems *O* and *P*, and with one product, anhydrous lanolin, B.P. (i) its solvent power is equal to that of system *O* which in turn is superior to that of system *P*.

Storage for 6 weeks at 20°C

Liquid paraffin, B.P. (Systems K–N; Table II)

The solvent power of these systems has decreased somewhat. Some changes in solubility of the lanolin products have occurred, notably with anhydrous lanolin, B.P. (i), with wool alcohols, B.P. (ii), and with liquid lanolin "A.C.E." (iii). The solubility of liquid lanolin "R.I.C.2" (vii) in the propellant 114/liquid paraffin B.P. system (L) has improved on storage.

Crystal formation occurred with water soluble wool alcohols "20" (x) in the paraffin/alcohol system (N).

Water (Systems O–Q; Table II)

The solvent power of the two systems incorporating halocarbon propellants (O,P) has decreased marginally, while that of the butane system (Q) has increased to a similar extent. In general there have been only minor changes in solubility during the test period. Crystal formation occurred with wool alcohols, O.P. (ii) in both halocarbon propellant systems (O,P).

Storage for 6 weeks at 0°C

The solvent power of all seven propellant systems decreased (*Table II*). In all propellant systems there is a marked decrease in the solubility of anhydrous lanolin, B.P. (i), and a moderate decrease in the solubility of liquid lanolin "R.I.C.2" (vii). With the other eight products, the decrease in solubility is almost negligible when compared with that obtained after six weeks' storage at 20°C. Agglomeration of separated matter is more prevalent.

Storage for 6 weeks at 0°C, followed by storage for 24 hours at 20°C

In general, the solubilities are similar to those noted after storage for 6 weeks at 20°C, with the exception of liquid lanolin "R.I.C.2" (vii) in the presence of propellant 114 systems (L) and (P).

Spray results (Table VI)

Spray failure occurred only in systems incorporating propellant 114 (L),(P), and was generally caused by agglomerated particles jamming in the valve housing. Anhydrous lanolin, B.P. (i), liquid lanolin "A.C.E." (iii), liquid lanolin "I.S.O." (vi), liquid lanolin "L.I.N." (viii) did not cause valve blockage in any of the propellant systems tested.

Spray characteristics

Table III indicates that quick-breaking foams were obtained with wool alcohols, B.P. (ii), and alcohol soluble lanolin (iv) in propellant 114/IMS/water (P), and in butane/IMS/water (Q) systems. Also with water soluble alcohols "20" (x) in the propellant 12/paraffin system (K). With wool alcohols, B.P. (ii) a stable foam on impingement was obtainable.

Effect of shaking on separated matter

All containers were shaken for $\frac{1}{2}$ minute and the time taken for the

settling of the redispersed matter was noted. The results are detailed in *Table VI.*

Conclusions

By adding the numbers in *Tables I* and *II* both horizontally and vertically, a total for each individual lanolin product in all propellant systems is obtained, together with a total for each individual propellant system containing all lanolin products. The lower the total, the better the solubility or solvent power.

Liquid lanolin "50 Super" (v) was found to be the most soluble product, whereas anhydrous lanolin, B.P. (i), wool alcohols, B.P. (ii), alcohol soluble lanolin (iv), and water soluble lanolin "75" (ix) are almost identically least soluble. The solubility of anhydrous lanolin, B.P. (i), wool alcohols, B.P. (ii), liquid lanolin "A.C.E." (iii), and water soluble lanolin "75" (ix) in the various propellant systems decreases on storage, whereas that of liquid lanolin "50 Super" (v) increases during the storage period. As might be expected, only water soluble lanolin "75" (ix), and water soluble wool alcohols "20" (x) are moderately soluble in the aqueous systems (O–Q), but the limited extent of this is somewhat disappointing.

Storage at 0°C materially affects the solubility characteristics of anhydrous lanolin B.P. (i), and of liquid lanolin "R.I.C.2" (vii).

Propellants 11/12/liquid paraffin/IMS (N) was found to be not only the best propellant system in respect of solvent power, but also far superior to all the other six propellant systems, the solvent powers of which decreased on storage, as will be seen from *Tables I* and *II*,

The results obtained with the propellant 114/IMS/water system (P) should be treated with a certain amount of reserve because of the irreversible separation into two liquid phases recorded after 6 weeks' storage at 0°C (*Table V*).

(*Received: 14th June 1964*)

Reference

(1) Herzka, A. *J. Soc. Cosmetic Chemists* **14** 331 (1963).

Book reviews

ANNUAL REPORTS ON THE PROGRESS OF CHEMISTRY FOR 1963. Vol. LX. Pp. 681 (1964). *Chemical Society, London.* 40s.

The Chemical Society's "Annual Reports" must be familiar reading to most chemists. This annual publication presents a superb, very concise review of the world's literature on chemistry compiled by a panel of distinguished contributors. Inevitably the latest edition is a little larger than its predecessors. This volume follows the general pattern of earlier editions in being divided into six main sections: General and Physical, Inorganic, Organic, Biological, and Analytical Chemistry, and Crystallography.

The first two reports are concerned with ionic solutions: Firstly the equilibrium properties of electrolyte solutions, and secondly, kinetic process in solution particularly electron and proton transfers. The third report deals with photochemical and photosensitized reactions and is the first since 1950 in this field where exciting new developments are taking place. Reports follow on homogeneous liquid phase polymerization, infra-red and raman spectroscopy, mass spectrometry and microwave spectroscopy of gases.

The section on Inorganic Chemistry gives an overall coverage of the typical elements, the transition elements and complexes.

The usual chemical groups and synthetic methods are surveyed in the section on Organic Chemistry and, in addition, reports deal with reaction mechanisms, studies of equilibria, physical properties and structure, alkaloids, steroids, carbohydrates, amino-acids and peptides. Steroids are dealt with rather more thoroughly than they have been in recent years, and notable developments include those in allene chemistry, synthesis in the terpene field and recent work on pituitary hormones.

Four reports are presented under Biological Chemistry which now includes proteins and peptides, formerly dealt with in the organic section. Other reports are on heteromeric saccharides, action of thyroid hormones, and the structure and function of ribosomes in protein biosynthesis.

Analytical Chemistry is reviewed under the classical inorganic/organic headings. In this field the most notable features are the expanding use of instrumental techniques and of automation, particularly in the control of industrial products, and the prominence of the several forms of chromatography.

Finally two reports deal respectively with the crystallography of inorganic and organometallic structures and of organic structures.

Comprehensive author and subject indexes conclude this excellent little volume which is an essential reference book for every chemist who wishes to keep abreast of new developments. R. P. REEVES.

SCIENCE AND THE SKIN. A. Jarrett. Pp. xv + 167 + Ill. (1964.) *The English Universities Press, London.* Paperback: 12s. 6d.; boards: 21s.

It is a special pleasure to review a book written by a fellow-member of the Society and the more so when the author is a British dermatologist who is also a keen research investigator. This is not a treatise, but a fairly simple exposition aimed at those who work on the fringes of the skin field. Part One presents the elementary facts of morphology and physiology, which are developed in Part Two as an excursion through present-day research ; this is naturally concerned to a large extent with frankly pathological conditions but nevertheless sheds a good deal of light on the problems encountered in cosmetic science.

Jarrett does not draw a hard and fast distinction between the proper sphere of cosmetics and the realm of the dermatologist, though one acquires the feeling that he might not like to see too much transgression into clinical problems. His plea for the evaluation of protective creams and the like by dermatologists in collaboration with cosmetic chemists, is thoroughly commendable. An injunction might well have been added to stress the need for a rigidly critical approach to the setting-up, conduct and analysis of such clinical trials.

The text, by avoiding obscure terminology, is unusually readable ; however, the scientific reader may encounter the difficulty of subsequently meeting the conventional terms without recognizing their significance. For example, Jarrett distinguishes between the sweat glands and the apocrine gland. He does not generally employ the customary designation of the eccrine gland as the source of sweat, and rather confuses the issue by occasional reference to "apocrine sweat glands." In a small volume like this, abbreviation to the point of serious inaccuracy might have been

anticipated but there is practically no sign of this. In the section dealing with antiperspirant and deodorant action, for example, it would have been easy just to give a brief resumé of the traditional half-truths, but the author's intellectual honesty is even here readily apparent. Cosmetic chemists seeking an introduction to the physiological background of their pursuits will find a great deal to interest them. Nowadays they need to be seriously concerned with potential side-effects and Jarrett demonstrates the correct perspective in relation to irritation, sensitization and carcinogenicity.

We could find no reference to dandruff anywhere, which seemed a pity, but obviously Jarrett could not deal with every aspect of dermatology. Perhaps one day he will see fit to compile a larger and more embracing volume with the same type of enquiring approach; in the meanwhile, the present one will certainly help to fill the gaps in many readers' understanding of the skin and its behaviour. N. J. VAN ABBE.

ELSEVIER LEXICA 4: LEXICON OF PRESSURIZED PACKAGING (AEROSOLS). Compiled by A. Herzka. Pp. x + 159 + Ill. (1964). *Elsevier Publishing Company, Amsterdam/London/New York.* 70s.

It is perhaps not too widely known that Elsevier publish a series of dictionaries each restricted to a distinct scientific field. These are intended to supply to technical interpreters and translators a reliable and comprehensive glossary of terms within a particular discipline or technology and to assist the worker seeking to abstract the essential features of a highly specialised publication in an alien tongue. Elsevier select for such Lexicons those languages in which a significant volume of relevant literature exists. The general editor, himself a former chief interpreter to the United Nations Organisation, usually entrusts the preparation to an acknowledged expert in the appropriate field, with assistance from similar authorities in those other countries in whose tongues equivalent terms are desired. The latest "pocket-book" in this series is devoted to the very new realm of aerosol technology.

There are two obvious linguistic consequences of the remarkable development of the pressurized packaging industry. Semantic problems have arisen from the extended use of existing terms and the undisciplined coining of new ones, whilst the preponderance of specialist publications in the English language directly results from the pioneering roles of the U.S.A. and, subsequently, the United Kingdom. Many terms, borrowed

from engineering practice or the packaging industry itself, are of colloquial origin and, as the compiler points out in his preface, it may be "exceedingly difficult to determine the appropriate equivalent in many of the other languages." Alternatively, artificial creations—which may be meaningless if literally translated without reference to the rationale of their evolution—are frequently assimilated, subject only to inflexion, into the technical patois of other tongues.

The compiler has selected 263 English words and phrases that he considers basic to the field. In the first part of the Lexicon these are marshalled into six groups, thus (I) contains general terms including varieties of phase and spray systems; these are followed by detailed terminology for (II) containers, (III) valve assembly and (IV) filling techniques; group (V) has a few terms for propellants, and in (VI) are to be found 55 phrases likely to be employed in discussing laboratory equipment and methods. Each term as it appears is immediately rendered into, in most cases, 20 other languages, viz. French, Italian, Spanish, Rumanian, German, Dutch, Norwegian, Swedish, Danish, Russian, Czech, Serbo-croatian, Slovenian, Bulgarian, Hungarian, Finnish, Greek, Hebrew, Arabic and Japanese. The printers are to be complimented on the clarity with which they have set the variety of non-Roman scripts included in this list.

In the next section, short definitions are given for 38 of the more important phrases appearing in the Lexicon. Many of these definitions were first published in the compiler's earlier book (1) or in an industrial glossary (2) but are usefully reiterated. Whilst the majority would find general acceptance, a few minor criticisms could be made. Thus, the choice of 105°F (40°C) as an arbitrary temperature for distinguishing "propellant" and "non-volatile ingredients" is debatable. Then, in defining "headspace" and "ullage," it would have been better to qualify "volume-in-dispenser" as "available volume," with suitable cross-references to the more precise definition given for "capacity." Finally, at the risk of appearing to cavil, it may also be observed that the now truly international word "aerosol" is given only its strict, scientific definition, despite current usage as both noun and adjective in connection with the release of coarse or fine sprays and pressurized powders. The wider sense is, however, implicit in the subsequent definition of "aerosol packs."

The definitions are followed by eight neat drawings, which clearly illustrate many of the structural terms translated in the first section. Then to complete the book, there is for each of the 21 languages an alpha-

betical index giving the code number under which every term appears. These indices materially enhance the value of the Lexicon to the non-English-speaking user.

Whilst it is difficult to check the accuracy of the cited equivalents in many of the tongues, it is in the relevance and adequacy of the words and phrases selected that the Lexicon demonstrates its worth. All are terms recognized in United Kingdom practice ; whilst some purists might object to such American infelicities as "go-no-go gauge" and "unscrambler," these phrases are nevertheless widely used. In passing it is noted that the compiler acknowledges, as indeed does Webster (3), both spellings of propell(a/e)nt. As is well known the "a" version, traditionally employed for solid fuel propulsion, has been widely adopted by the aerosol filling industry and followed by a number of the specialist journals, whilst the alternative spelling is recommended by the British Standards Institution and has been used for many years by the largest producers of halocarbon propellents (sic) both in the United Kingdom and the U.S.A.

Today, authorities in many countries are showing interest in regulations regarding safety in production, distribution and use of pressurized (aerosol) dispensers (4–6), whilst national and international agencies variously consider standard specifications and procedures. Accurate and unambiguous translation of specialised terms at international meetings and working groups is therefore particularly important ; this Lexicon has already proved useful for that purpose. It might be even more helpful if the compiler could be persuaded to include the equivalent abbreviations for some of these terms when a revised edition appears.

It is concluded that this Lexicon is a timely and valuable contribution to the consolidation and further expansion of a vigorous industry.

G. F. PHILLIPS.

REFERENCES

(1) Herzka, A. and Pickthall, J. *Pressurised Packaging* (*Aerosols*). 2nd edn. (1961) Butterworth & Co : London.
(2) *Glossary of Technical Terms* The Metal Box Company : London.
(3) *Webster's New International Dictionary*, 2nd edn. (1961) G. Bell & Sons Ltd. : London.
(4) Phillips, G. F. *J. Soc. Cosmetic Chemists* **14** 357 (1963).
(5) *Specialities* **1** 34 (December 1964).
(6) Schuddeboom, L. J. *Mfg. Chemist Aerosol News* **35** 82 (July 1964).

Society of Cosmetic Chemists of Great Britain

DIPLOMA EXAMINATION

Brunel College

PAPER I

(Wednesday, 24th June 1964.)

Candidates should answer FIVE questions only; one question from each section. Each answer must be commenced on a separate sheet of paper or book.

Where possible, express relationships and reactions in equations with a full explanation of symbols used, and give simple sketches where appropriate.

SECTION A

1. With particular emphasis on the nature and importance of side chains, describe the way in which the polypeptide chains of hair keratin can be built up from simple compounds.
   To what extent and under what conditions are the cross linkages modified during a permanent waving process?

2. A coloured substance is of value in hair colourants only if it can be attached to the hair. Discuss the various methods which can be used to ensure satisfactory uptake with reference to (a) the chemical constitution of the dye molecule and (b) the environment of the dye molecules.
   To what extent can the present dye products be considered as ideal hair colourants.

3. Discuss with specific examples the formulation of the following hair-dressing products :—

   (i) Liquid brilliantines.

(ii) Two layer lotions.
(iii) Water in oil emulsions.
(iv) Aerosol hair sprays.

Section B

4. Discuss the uses of the separate ingredients in the face powder formula given below.
Suggest any improvements which you think could be made to this formula.

	%
Talc	15.0
Kaolin	65.0
Precipitated chalk	5.0
Titanium dioxide	4.5
Magnesium stearate	5.0
Magnesium carbonate	1.0
Perfume	0.5
Iron oxide colours	4.0

5. You are given the job of setting up a department for the manufacture of lipsticks. Discuss the items of equipment required, and considerations of services, storage and layout.

6. Discuss considerations of safety-in-use of lipsticks. What studies are required in order to estimate the toxicity of a raw material for use in lipsticks ?

Section C

7. Why is it necessary to have more than one propellant available for pressure packs ? Illustrate your answer by describing how the right balance of propellants would be arrived at in formulating a pressure packed hair spray.

8. What factors would you take into account in determining the shelf life of a brushless shaving cream to be packed into a collapsible tube ? How would you assess these factors ?

Section D

9. Give an account of the structure and properties of the bacterial spore with particular reference to those features which make it resistant to the action of antiseptics.

10. Outline the factors which must be considered in selecting a preservative for a toilet preparation.

11. What are the important micro-organisms occurring on the skin? Discuss the relative importance of each and the selection of a germicide which may be used to inhibit their development.

Section E

12. Describe with appropriate diagrams the structure of human epidermis.

13. Define the term 'emulsifying agent'. Describe, with specific examples, the properties and uses of the principal classes of emulsifying agents.

Paper II

(Monday, 29th June 1964.)

Candidates should answer FIVE questions only; one question from each section. Each answer must be commenced on a separate sheet of paper or book.

Where possible, express relationships and reactions in equations with a full explanation of symbols used, and give simple sketches where appropriate.

Section A

1. Describe the experimental set up and operation of a surface balance, commenting on some typical relationships between surface pressure and areas occupied by molecules of insoluble materials.
When 1×10^{-4} cc of a certain gum (density 0.9 g/cc) was compressed on a surface balance at 19°C the following values of surface pressure were obtained.

Area per molecule ($A^{\circ 2}$)	Surface pressure (dynes/cm)
2800	0.10
2061	0.15
1700	0.20
1480	0.25
1333	0.30

By making a suitable plot assuming the film is gaseous in nature and given that the Gas Constant (R) $= 8.31 \times 10^7$ ergs/mole degree calculate the molecular weight of the gum.

2. Explain what is meant by the "spreading coefficient." If the surface tension of water is 72.8 dynes/cm at 20°C, the surface tension of benzene is 28.9 and the interfacial tension between benzene and water is 35.0, what is the initial spreading coefficient ?
Discuss the practical importance of wetting and spreading during the action of cosmetic preparations.

SECTION B

3. Describe *either* the extraction and refining of fats and oils with reference to those of cosmetic interest *or* a classification of the lipids indicating the uses of those of cosmetic interest.
4. Infra-red and ultra-violet absorption spectroscopy are of value for the identification of organic chemical substances.
Write a short account of the physico-chemical principles upon which this statement is based.
5. What contribution would you expect chromatographic methods to make to the analysis of (1) an essential oil, (2) a fixed oil, and (3) a dyestuff ?
Discuss the experimental procedure in *one* instance.

SECTION C

6. Write short notes on what you know about six of the following perfumery raw materials :—

Amyl cinnamic aldehyde	Sandalwood oil East Indian
Lavandin oil	Terpineol
Citronellol	Lemongrass oil
The nitro musks	Linalyl acetate

7. (a) Suggest one simple formula of at least four constituents for each of the following types of floral perfume :—
Lilac, violet, rose, and lavender
Include if possible an approximate indication of the proportions of those constituents mentioned.
(b) What factors need to be taken into account in formulating a perfume for a white toilet soap ?

SECTION D

8. Discuss the limitations of the falling sphere method of measuring viscosity.
A glass ball weight 0.157 g and 0.5 cm in diameter was allowed to

fall through an oil contained in a very wide tube. The mean time taken to fall through a distance of 25.0 cm was 11.3 sec. Given that the acceleration due to gravity is 981 cm sec^{-2} and that the density of the oil at the temperature of the experiment was 0.85 g cc^{-1}, calculate the viscosity of the oil.

9. Describe the drop-weight for the determination of surface tension. When a given volume of olive oil was dropped through a standard tip into a 0.001M NaOH solution, 44 drops were formed and the interfacial tension between olive oil and 0.001M NaOH was computed to be 7.3 dynes/cm. When the same volume of olive oil was dropped through the same standard tip into an aqueous solution 0.001M in NaOH and 0.15M in NaCl, 300 drops were formed. Calculate the interfacial tension between olive oil and an aqueous solution 0.001M in NaOH and 0.15M in NaCl.

10. Describe in detail a series of simple laboratory tests which can be used for subjective and objective assessment of cosmetic emulsions.

SECTION E

11. Write short notes on each of the following :—
    Dental caries.
    Enamel.
    Calculus.
    Denture cleansers.

12. Using diagrammatic flow curves distinguish between Newtonian, pseudo-plastic and thixotropic materials. State the factors which govern the viscosity of an emulsion and use mathematical expressions where possible to indicate their significance.

Successful Candidates

In the new course, eleven out of seventeen candidates were successful. In the old two-year course, five out of seven candidates were successful. Diplomas were awarded to the following :

Miss G. Bellamy	P. J. Hunt	B. Price
Miss A. Colls	G. Ibbott	W. W. F. Scotland
J. S. Crombie	Miss J. M. Johnson	K. B. Shipp
C. D. Foley	Miss V. J. Lavin	Miss P. Vassalli
E. J. Fowler	P. A. Mawbey	Miss M. Woodward
	Miss S. V. Peters	

£5 prizes were awarded to Mr. W. W. F. Scotland (new course), and Miss V. J. Lavin (old course).

SOIRÉE

The Society recently broke fresh ground by holding a Soirée on 16th November 1964 at the School of Pharmacy, Brunswick Square, London, W.C.1. The Soirée took place immediately prior to the Symposium on "Antioxidants and Preservatives," and was well attended by members and their friends and those participating in the Symposium.

The Guest of Honour for the evening was Mr. R. W. Murphy, Immediate Past-Chairman of the Toilet Preparations Federation, who stood in at short notice for Mr. A. Stafford May, Chairman of the Toilet Preparations Federation. The Society's President and Mrs. Herzka with Mr. and Mrs. Murphy received the guests. Other guests included Dr. A. C. Bevan, Acting Head of the Chemistry Department, Brunel College, Dr. K. R. Capper, Pharmaceutical Society, Professor A. C. Frazer, Professor of Medical Biochemistry and Pharmacology in the University of Birmingham, Dr. Frank Hartley, Dean of the School of Pharmacy and Mrs. Hartley, Professor E. Shotton, School of Pharmacy, Mr. J. B. Wilkinson, President of the International Federation of Societies of Cosmetic Chemists, and Mrs. Wilkinson, and Mr. V. H. Williams, High Sheriff of Merionethshire, and Mrs. Williams.

During the course of the evening, 14 of the candidates who had been successful in the Society's 1964 Diploma Examinations were presented with their Diplomas. Miss V. J. Lavin and Mr. W. W. F. Scotland were awarded £5 prizes for the best results in these examinations.

The rest of the evening was taken up with dancing and with an interesting and amusing interlude arranged by Max Factor, and presented by Miss E. Gardiner.

The Soirée provided a pleasantly informal occasion for members and their friends to meet, and it is expected that it will become a regular feature in the Society's calendar.

Symposium on "PRESERVATIVES AND ANTIOXIDANTS"

A Symposium on "Preservatives and Antioxidants" organised by The Pharmaceutical Society of Great Britain and The Society of Cosmetic Chemists of Great Britain took place on Tuesday, 17th November 1964, at The Connaught Rooms, Great Queen Street, London, W.C.2. It was attended by 258 participants, including delegates from Germany, Holland, Japan, Switzerland and the U.S.A.

Mrs. Murphy being presented with a bouquet by Mrs. Herzka, while Mr. Murphy looks on.

(left to right) Prof. A. C. Frazer, The President, Mrs. Herzka, Mr. R. W. Murphy, Mrs. Murphy.

Miss Eve Gardiner and assistants.

Miss V. J. Lavin receiving her Diploma and the £5 prize from the President.

SOIRÉE 1964

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SYMPOSIUM ON EMULSIONS

A Symposium on Emulsions will be held at the Royal Hall, Harrogate, Yorks, from 30th March–1st April 1965. Participation is permitted only when application has been made on the appropriate form, and the fee duly paid. This is £3 3s. for each participant who is a member of one of the Societies of Cosmetic Chemists affiliated to the I.F.S.C.C. The registration fee for non-members is £6 6s. Registration forms giving all details are available from Mr. R. F. L. Thomas, c/o Gibbs Pepsodent Ltd., P.O. Box No. 167, Leeds, 1, Yorks. **The closing date for registration is 28th February 1965.**

Wednesday, 31st March 1965

9.30 a.m. Civic Welcome by the Mayor of Harrogate.

9.40 a.m. "Fundamental Methods of Predicting Changes in Rheological Properties of Emulsions on Ageing" P. SHERMAN, M.Sc., F.R.I.C. (*Unilever Research Laboratory, Welwyn*).

10.20 a.m. "The Importance of Perfumes in Emulsion Formulations" J. KLAP (*Proprietary Perfumes Ltd., Ashford*).

11.00 a.m. COFFEE.

11.20 a.m. "Hydrolysis of Wax-Esters in Emulsions" E. V. TRUTER, B.Sc., Ph.D., A.R.C.S., D.I.C. and C. A. ANDERSON, B.Sc., Ph.D. (*Department of Textile Chemistry, Leeds University*).

12.00 noon "Cationic Surfactants as Emulsifying Agents" K. M. GODFREY, B.Sc., A.R.I.C. (*Armour Hess Chemicals Ltd.*).

Thursday, 1st April 1965

9.30 a.m. "An Alternative Approach to the Principle of Emulsion Formulation" W. BURT, F.P.S. (*Chelsea School of Pharmacy*).

10.10 a.m. "The Influence of Lanolin Derivatives on Dispersed Systems" L. I. CONRAD, H. F. MASO and S. A. DERAGON (*American Cholesterol Products, Inc., New Jersey*).

10.50 a.m. COFFEE.

11.10 a.m. "Difficulties of Topical Treatment in Dermatology" G. HODGSON, M.B.E., M.D. (*Cardiff Royal Infirmary*).

11.50 a.m. "An Evaluation of the Requirements and Problems in the Packaging of Emulsions" C. E. HIGGS, B.Sc., A.R.C.S. (*Gibbs Pepsodent Limited, Leeds*).

EXHIBITION AND SOCIAL EVENTS

Tuesday, 30th March 1965

11.00 a.m. Exhibition opened by the Mayor of Harrogate.

8.30 p.m. Civic Reception at the Lounge Hall. Buffet refreshments and informal concert (free to symposium and exhibition participants).

Wednesday, 31st March 1965

1.00 p.m. Informal Lunch at the Old Swan Hotel (included in registration fee).

7.30 p.m. for 8.00 p.m. Banquet at the Old Swan Hotel.

Thursday, 1st April 1965

Exhibition closes 6.00 p.m.

1965 LECTURE PROGRAMME

Venue : The Royal Society of Arts, John Adam Street, London, W.C.2.

Time : 7.30 p.m.

Lectures will be given on the following dates:

Tuesday, 26th January 1965. *Storage Testing in the Cosmetic Industry.* A discussion, with contributions by D. E. Butterfield, M.A. (I.F.F. [Great Britain] Ltd.), E. K. Clarke, B.Sc. (Beecham Group, Ltd.) and J. J. Mausner, B.Sc., Ph.D., F.R.I.C. (Helena Rubinstein, Ltd.).

Thursday, 25th February 1965. *The Sebaceous Gland.* F. J. G. Ebling, M.Sc., Ph.D., M.I.Biol. (University of Sheffield).

Thursday, 13th May 1965. *Film evening.*

1965 MEDAL LECTURE

The Council has agreed to institute an Annual Medal Lecture, and it was decided that

> "the recipient of the Annual Medal shall be a leading personality who has made an outstanding contribution to science, public life or the arts. The lecture shall be on cosmetics or allied subjects."

The 1965 Medal Lecture entitled *The hormonal background of the skin*, will be given on Wednesday, 14th April by Professor Sir Edward Charles Dodds, M.V.O., F.R.S., F.R.I.C., Courtauld Professor of Biochemistry, University of London.

Admission by tickets, obtainable from the General Secretary.

FUTURE SYMPOSIA

A symposium on **Physical Methods** will take place in Bristol, on 16th November 1965. *Programme Secretary :* Mr. N. J. Van Abbé, Beecham Toiletry Division, Beecham House West, Great West Road, Brentford, Middex.

A Symposium on **Colour** will take place in Eastbourne, Sussex, during the week commencing 25th April 1966.

DINNER AND DANCE

This will take place on Saturday, 13th February 1965, at the Connaught Rooms, Great Queen Street, London, W.C.2.

ANNUAL GENERAL MEETING

This will take place on Monday, 24th May 1965, at 7 p.m., at 55 Park Lane, London, W.1.

4TH I.F.S.C.C. CONGRESS

The 4th I.F.S.C.C. Congress will take place in June 1966, in Paris. Anyone wishing to submit a paper should contact the Societe Francaise de Cosmetologie, 65, Bld. Lannes, Paris 16e, France.

GENERAL NOTICES

Publication dates: The "Journal of the Society of Cosmetic Chemists" is published every four weeks.
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Two issues by the Gesellschaft Deutscher Kosmetik-Chemiker, e.V.
from Hamburg-Grossflottbek, Beselerstrasse 1, Germany.

Issue No.	*Publication Date*	*Country of Origin*
1	8th January	Great Britain
2	5th February	U.S.A.
3	4th March	U.S.A.
4	1st April	Great Britain
5	29th April	Germany
6	27th May	U.S.A.
7	24th June	Great Britain
8	22nd July	Great Britain
9	19th August	U.S.A.
10	16th September	U.S.A.
11	14th October	Great Britain
12	11th November	Germany
13	9th December	U.S.A.

Advertisements : All enquiries regarding advertisements in the British Editions of the Journal should he addressed to Mr. J. V. Robinson, Pergamon Press, Ltd., 4 & 5 Fitzroy Square, London, W.1.

Subscription : All members of the Society of Cosmetic Chemists of Great Britain receive one copy of each edition free. Further copies at non-member rates. Non-members: £10 per annum, post free, or £1 per issue, post free.

Missing Numbers : **Journals are despatched at Printed Paper rate. Claims for missing numbers can be entertained only from subscribers in the country of origin of the particular issue, and must be made within 30 days from date of issue.** Members and subscribers are urged to give notice of change of address to the Publication Offices.

Responsibility for statements published : The Society of Cosmetic Chemists of Great Britain and its Hon. Editor assume no responsibility for statement or opinions advanced by contributors to this Journal.

Lectures : The Society shall have the right of first publication of any lecture or address delivered before it, but does not undertake to publish any given matter.

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Manuscripts : These should be in accordance with the "Directions for Preparation of Manuscripts," copies of which are available from the Hon. Editor, Ashbourne House, Alberon Gardens, London, N.W.11 (see next page).

DIRECTIONS FOR PREPARATION OF MANUSCRIPTS

1. General Policy

The copyright of papers presented before the Society of Cosmetic Chemists of Great Britain belongs to the Society. A presented paper may only be published, in the first instance, in another journal of the author(s)' choice if the *Journal of the Society of Cosmetic Chemists* is unable to publish it.

2. Nature of the Text

2.1. When submitting the manuscript of his text the author shall specify, as far as possible, in which category of original scientific literature this text is to be classified, i.e.

2.1.1. original scientific paper;

2.1.2. provisional communication or preliminary notes ; or

2.1.3. subject review article.

2.2. A text is regarded as belonging to the category "*Original scientific paper*" when it is written in such a way that a qualified research worker, specializing in the same branch of science, is able, simply on the basis of the information given

2.2.1. to produce the experiments and secure the results described with equal accuracy or within the limits of experimental error specified by the author ;

or

2.2.2. to repeat the author's observations and judge his findings ;

or

2.2.3. to check the accuracy of the analyses and deductions on which the author's findings are based.

2.3. A text is regarded as "*Provisional communication or preliminary notes*" when it contains one or more novel items of scientific information, but is insufficiently detailed to allow readers to check the said information in the ways described in paragraph 2.2 above.

2.4. A "*Subject review article*" is not designed for publication under the heading of "new scientific information" ; it is a survey of one particular subject, in which information already published is assembled, analyzed and discussed.

3. Drafting of the Text

3.1. The introduction, of a historical or critical character, although often useful, shall be kept as short as possible ; in particular the author shall refrain from producing a critical review of the type of the subject review article described above.

3.2. The syntax shall be as simple as possible and the words used should be those to be found in any ordinary dictionary. If this is not feasible, the author shall make certain that the neologisms he uses form part of the international scientific and technical vocabulary. The author is recommended to state the origin of the neologisms he uses. Should he be obliged to create some himself, he should say how this has been done, giving the etymology and definition. Finally, the author shall make sure that he does not distort the meaning of the terms belonging to the specific vocabulary of the branch of knowledge with which he is dealing.

3.3. In drafting the text, the author shall describe fully the methods employed and significant results obtained. Should industrial or national security considerations lead him to restrict the amount of scientific information that he wishes to publish on the subject he is dealing with, the text shall be presented as belonging to class 2.1.2. *provisional communication* or *preliminary notes*, and not to class 2.1.1. *original scientific paper*. This is an absolute moral obligation for the author. It goes without saying that, in any publication, the facts observed or the methods employed must not be wilfully misrepresented.

3.4. Explicit reference shall be made to any work previously published by the same author, or by another, when a knowledge of such works is essential in order to see how the text presented fits into the general picture of scientific progress. It should be stated whether these previous publications duplicate, completely or partially, the text presented.

4. Opening Page

It is suggested that this include :

4.1. The title.

4.2. Name(s) of the author(s), and titles.

4.3. Names and address of author(s)' laboratory, etc.

4.4. The *Synopsis :* This should comprise a brief and factual summary of the contents and conclusions of the paper, refer to any new information which it may contain, and give an indication of its relevance. It should enable the busy reader to decide more surely than he can from the mere title of the paper whether it merits his reading it. The author of every paper is therefore requested to provide also a synopsis of it, in accordance with the following suggestions.

4.4.1. Use complete sentences rather than a mere list of headings. Any reference to the author of the article should be in the third person. Standard rather than proprietary terms should be used. Unnecessary contractions should be avoided. It should be presumed that the reader has some knowledge of the subject but has not read the paper. The synopsis should therefore be intelligible in itself without refer-

ence to the paper. (For example it should not cite sections or illustrations by their numerical references in the text.)

4.4.2. *Content.* As the title of the paper is usually read as part of the synopsis, the opening sentence should be framed accordingly so as to avoid repetition of the title. If, however, the title is not sufficiently indicative, the opening sentence should indicate the subjects covered. Usually, the beginning of a synopsis should state the objects of the investigation. It is sometimes valuable to indicate the treatment of the subject by words such as—brief, exhaustive, theoretical, etc.

4.4.3. The synopsis should indicate newly observed facts, conclusions of an experiment or argument, and if possible, the essential parts of any new theory, treatment, apparatus, technique, etc. It should contain the names of any new compound, mineral species, etc., and any new numerical data, such as physical constants ; if this is not possible, it should draw attention to them. It is important to refer to new items and observations, even though some may be incidental to the main purpose of the paper ; such information may otherwise be hidden although in fact it might be very useful.

4.4.4. When giving experimental results the synopsis should indicate the methods used ; for new methods, the basic principle, range of operation, and degree of accuracy should be given.

4.4.5. *References, citations :* If it is necessary to refer in the synopsis to earlier work, the reference should always be given in the same form as in the paper ; otherwise, references should be omitted.

4.4.6 *Length.* The synopsis should be as concise as possible. Only in exceptional cases should it exceed 200 words.

5. Preparation of Manuscripts

These should be submitted in duplicate (original and one carbon copy), double-spaced typescript, leaving a 1″ (2½ cm) margin on the left-hand side, with formulæ in careful manuscript. Only one side of the paper should be used. The author should always retain a carbon copy for his own use.

To assure typographical correctness in the printed proofs, the following suggestions should be carefully observed in the preparation of manuscripts.

5.1. Matters that are to be set in Greek type should be clearly indicated. Some Greek letters, when handwritten, are difficult to distinguish from similar-looking English letters. In case of both Greek and English letters if not typewritten, it should be made clear if they are to be set in capital or small (lower case) type.

5.2. The letter l should be looped when typewritten in equations, etc., to avoid confusion with the figure 1. There should also be a clear

differentiation between zeros and the letter O (or the small o) which often look the same when typewritten or handwritten.

5.3. Superior and inferior positions should be clearly indicated.

5.4. In general, equations and formulations should be clearly and carefully written, taking care to have all figures and symbols, especially in fractions and equations, in the alignment in which they are to be printed.

5.5. *Footnotes.* These as distinct from literature references should be avoided as far as possible. Where they are essential, reference is made by the symbols * † ‡ § ‖ ¶ in that order.

6. Illustrations

All photographs should be black and white, glossy, and unmounted. Diagrams, graphs, charts, etc., should be about twice the final size required, and should be drawn in Indian ink on tracing paper, or on white drawing paper. All letterings and figures should be large enough to be capable of reduction to the required size. The following standard symbols should be used on line drawings since they are easily available to the printers.

▲ △ ■ □ ● ○ ⊙ + × ◆

On the back of each illustration, photograph, etc., should be written the author's name and the figure number. Legends for the figures should be supplied on a separate sheet.

6.1. Each illustration, figure, graph, or chart, should be numbered consecutively using *Arabic* numerals, e.g. Figure 1.

6.2. Tables should be numbered consecutively, using *Roman* numerals, e.g. Table I.

7. References

The names of all the authors of papers to be cited should be given when reference is first made in the text. In cases where there are more than two authors subsequent citations should give the first-named author followed by the words et al underlined once. References should be indicated in the text by bracketed numbers, and the full reference should be given at the end of the paper in the following form.

7.1. Journals.

It is essential that the authors' names and initials, the abbreviated title of the journal, the volume number, the page number, the month and year of publication should be given. Abbreviations of journals should be in accordance with the practice followed by *Chemical Abstracts* (cf. list of periodicals, abstracted by *Chemical Abstracts*). If access to that publication is not available then the name of the journal should be given in full.

7.2. Books.
It is essential that the authors' names and initials, the full title of the book, the page number, the year of publication, the name and address of publishers should be given.

7.3. Patents.
Only the patent number should be cited, e.g. Brit. Pat. 805,202.

7.4. **Please note that in the typewritten manuscript**

7.4.1. the abbreviated or full title of the journal, or the title of the published book, should be underlined once ;

7.4.2. the volume of the journal should be underlined twice ;

7.4.3. the month of publication, the year of publication, and the publisher in the case of books, should be bracketed independently of each other, e.g.

(1) Fregert, S. Acta Dermato-Venereol. 40 206 (July) (1960).

(2) Baer, R. L. in McKenna, R. M. B. Modern Trends in Dermatology 232 (1954) (Butterworths, London).

If references are likely to be accessible only with difficulty, the inclusion of a suitable abstract reference is also desirable.

8. Trade Names

All trade names cited in the text should be underlined once.

9. Acknowledgements

Acknowledgements for permission to reproduce illustrations, photographs, graphs, etc., should be listed at the end of the text. Acknowledgement to the author(s)' employers for permission to publish should not be made, because publication is deemed to indicate that such permission has been granted.

10. Proofs

Page proofs will be sent to the first-named author for correction. In the interest of speedy publication galley proofs are no longer submitted to authors. The difficulty and expense involved in making corrections to page proofs makes it essential for authors to prepare their manuscripts carefully so that alteration is not required.

11. Reprints and Preprints

The first-named author will automatically receive 25 reprints, or preprints where applicable, free of charge. Additional reprints must be ordered at the time the proofs are returned.

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