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Journal of the Society of Cosmetic Chemists

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Effective with the start of the year 1979, the *Journal of the Society of Cosmetic Chemists* will publish seven issues per year as follows:

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No. 6 November
No. 7 December

As you know, during the past years we have published our Journal in conjunction with the Journal of the British Society of Cosmetic Chemists; the U.S. Society published seven issues while the British Society published five issues per year. With our increasing membership and technical growth we are now in a position to begin publishing a Journal entirely under the auspices of the U.S. Society of Cosmetic Chemists. The British have agreed in principle to continue publication of their Journal as a separate, new Journal and we will supply each member of our Society and each subscriber to our Journal with copies of the British Journals as they are received by us. In this way, there will be no loss to our members or subscribers in that they will receive 12 issues of the Journal during 1979.

We take this opportunity to thank our British colleagues for their past cooperation and wish them well as they continue to publish their own Journal. We are ready to offer them our assistance in any way we can.

I also take their opportunity to announce that Dr. Leszek Wolfram of Clairol Incorporated will assume the Editorship of the Journal on January 1st, 1979. I thank you for the cooperation you have given to me during my tenure as Editor of your Journal.

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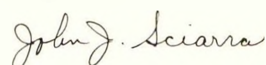

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Journal of the Society of Cosmetic Chemists®

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DECEMBER 1978

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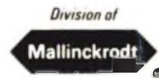

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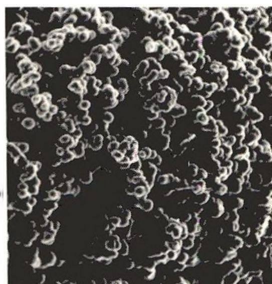

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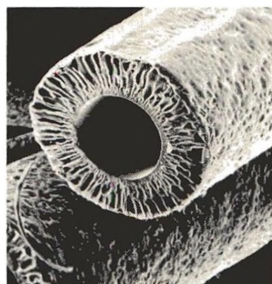

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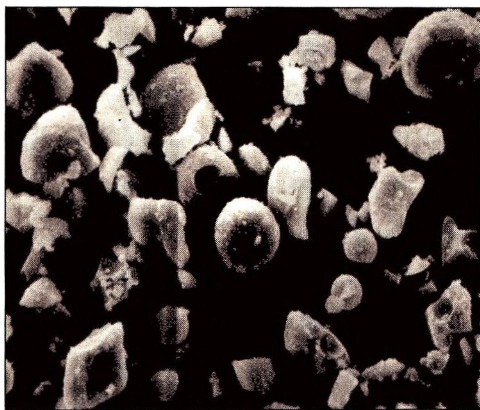

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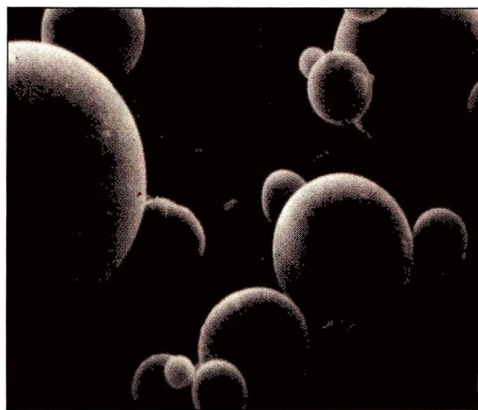

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SYNOPSES FOR CARD INDEXES

The following synopses can be cut out and mounted on 3 x 5 index cards for reference, without mutilating the pages of the Journal.

Low energy emulsification II: evaluation of emulsion quality: T. J. Lin. *Journal of the Society of Cosmetic Chemists* 29, 745 (December 1978)

Synopsis—Low-energy emulsification (LEE), an emulsification technique proposed by Lin to conserve mechanical and thermal energies in the processing of emulsions, was examined in terms of emulsion quality and compared with similar emulsions made with a conventional hot process.

Experimental data obtained from prototype cosmetic formulations consisting of W/O and O/W emulsions stabilized with various cationic, anionic and nonionic surfactants and their mixtures indicate that the technique is extremely flexible and is capable of producing emulsions with varying droplet sizes. The key to success in applying the technique lies in understanding and controlling the physical variables responsible for causing a droplet size variation.

The key variables found were: first stage emulsification temperature, mixing intensity, ratio of external phase added during the first stage of emulsification to that of the second stage dilution, rate and the mode of phase combination. By effectively controlling these variables, it has been demonstrated that it is possible to produce emulsions with smaller and more uniform droplet size distribution using the low-energy technique than similar emulsions obtained with the conventional hot emulsification. In many instances, withholding of a large amount of external phase for later addition resulted in a sharp reduction of the droplet size. This effect is apparently related to the solubilization effect observed by Lin, Kurihara and Ohta and a proper control of this effect allows processing of finer emulsions with a substantial reduction of not only thermal and mechanical energies but also processing time.

Imidazolidinyl urea activity against Pseudomonas: Philip A. Berke and William E. Rosen. *Journal of the Society of Cosmetic Chemists* 29, 757 (December 1978)

Synopsis—*Pseudomonas* contamination of cosmetics is a major concern in the cosmetic industry because pseudomonads are so widely distributed in nature, so adaptable, and so resistant to most antimicrobials. Eleven ATCC-type pseudomonads, representing ones of concern for contaminating cosmetic products, and seventeen "wild" pseudomonads, isolated from a variety of contaminated cosmetic products, were screened and were found to differ in their vulnerability to imidazolidinyl urea alone or in combination with parabens. Screening experiments were carried out to study variables such as incubation time, incubation temperature and pH with the purpose of learning how to design and interpret *Pseudomonas* screening experiments. Experimental data are presented contrasting inadequate or marginal preservation of cosmetic lotions and shampoos with adequate preservation of these products. It was found, for example, that the parabens alone provided inadequate protection against pseudomonas growth in several creams, lotions, and shampoos. Addition of Imidazolidinyl Urea to these formulations is shown to enhance the range of effectiveness and to give adequate protection against *Pseudomonas* contamination.

Eine methode zur bestimmung der verteilungsgleichgewichte von konservierungsmitteln in emulsionen: Gerhard Sauermann, Wolfgang Hofeditz and Walter Engel. *Journal of the Society of Cosmetic Chemists* 29, 767 (December 1978)

Synopsis—Partial phase separation of emulsions was achieved by ultrafiltration and ultracentrifugation without noticeable changes in partition coefficients and adsorption characteristics. The amount of preservatives in the continuous phase is then determined with the aid of HPLC. Data obtained by this technique are in good agreement with the results of microbiological challenge testing.

Effects of harvesting techniques on hydration dynamics: gravimetric studies of stratum corneum: Robert L. Rietschel and William A. Akers. *Journal of the Society of Cosmetic Chemists* **29**, 777 (December 1978)

Synopsis—Stratum corneum specimens harvested by several methods commonly used for in vitro studies have been compared for hygroscopicity. Cantharidin blisters give superior data when compared to heat, trypsin and ammonia fume separated specimens. As the cantharidin data represent skin from a different age group and body site, a comparison to animal data in the literature is made.

Prediction of hair assembly characteristics from single fiber properties: C. R. Robbins and G. V. Scott. *Journal of the Society of Cosmetic Chemists* **29**, 783 (December 1978)

Synopsis—An hypothesis is developed with relationships that predict how changes in the behavior of hair assemblies (tresses or heads) depend on changes in single fiber properties that are measurable, i.e., how changes in combing ease, flyaway, body, managability and style retention of hair assemblies relate to changes in fiber friction, stiffness, static charge, curvature, weight and diameter. From these relationships desired changes in assembly characteristics may be approached through changes in the fiber properties.

J. Soc. Cosmet. Chem., **29**, 745–756 (December 1978)

Low-energy emulsification II: evaluation of emulsion quality

T. J. LIN *628 Enchanted Way, Pacific Palisades, CA 90272;*
Toshiyuki Akabori, Shoji Tanaka and Katsuyuki Shimura,
Arimino Chemical Co., Ltd., Tokyo, Japan.

Received December 26, 1977. Presented at Annual Scientific Meeting,
Society of Cosmetic Chemists, December 1977, New York, New York.

Synopsis

LOW-ENERGY EMULSIFICATION (LEE), an emulsification technique proposed by Lin (1, 2) to conserve mechanical and thermal energies in the processing of emulsions, was EXAMINED in terms of EMULSION QUALITY and compared with similar emulsions made with a conventional hot process.

Experimental data obtained from prototype cosmetic formulations consisting of W/O and O/W emulsions stabilized with various cationic, anionic and nonionic surfactants and their mixtures indicate that the technique is extremely flexible and is capable of producing emulsions with varying droplet sizes. The key to success in applying the technique lies in understanding and controlling the physical variables responsible for causing a droplet size variation.

The key variables found were: first stage emulsification temperature, mixing intensity, ratio of external phase added during the first stage of emulsification to that of the second stage dilution, rate and the mode of phase combination. By effectively controlling these variables, it has been demonstrated that it is possible to produce emulsions with smaller and more uniform droplet size distribution using the low-energy technique than similar emulsions obtained with the conventional hot emulsification. In many instances, withholding of a large amount of external phase for later addition resulted in a sharp reduction of the droplet size. This effect is apparently related to the solubilization effect observed by Lin, Kurihara and Ohta (3, 4) and a proper control of this effect allows processing of finer emulsions with a substantial reduction of not only thermal and mechanical energies but also processing time.

INTRODUCTION

The basic principles and the economical advantages of low-energy emulsification (LEE) have been thoroughly discussed by Lin in his earlier publications (1, 2). Basically the technique involves withholding a portion of the emulsion's external phase and first making an emulsion concentrate at an elevated temperature. The withheld external phase is kept at a lower temperature and added to the concentrate with mixing during the second stage (diluting) operation.

Since a portion of the external phase is added at a lower temperature (usually room

temperature), a significant amount of thermal energy can be conserved particularly if the amount of withheld external phase is substantial relative to the amount of emulsion concentrate processed in the first stage. In addition to conserving thermal energy, the technique also allows a considerable reduction in processing time by effectively reducing the time required for batch cooling. Mechanical energy expended during the cooling period is, as a result, also reduced.

It should be emphasized that LEE differs substantially from ordinary, low-temperature emulsification in that the entire process is not carried out at a constant, low temperature. First-stage emulsification can be carried out at almost any desired temperature to obtain the necessary sterilization, dispersion, blending or promotion of a chemical reaction in the low-energy method. The diluting liquid to be added to the batch at the second stage is usually kept at ambient temperature but may be adjusted to any desired temperature, if necessary. LEE is, therefore, much more versatile compared to the conventional, low-temperature method as it allows processing of a wide variety of cosmetic emulsions even when they contain waxy substances such as cetyl alcohol, stearic acid and beeswax. By applying thermal energy only when and where needed, LEE offers a great flexibility with a definite economical advantage.

Whereas the economy of LEE cannot be disputed for the mass production of emulsions when applicable, the quality of emulsions so produced has not been critically examined. The main purpose of this investigation is to systematically evaluate the qualities of typical O/W and W/O cosmetic emulsions prepared by LEE against similar emulsions prepared by a conventional hot process. The quality of a cosmetic emulsion is an ambiguous term and is obviously dependent on its end purpose and one's definition. For the purpose of this presentation, nevertheless, the quality of an emulsion is defined such that the finer the droplet size, the better the quality. This definition is consistent with most applications of cosmetic emulsions as a finer droplet size is usually associated with a finer texture, higher gloss and, generally, but not absolutely, with a better stability. The definition is an arbitrary one, however, as a finer droplet size does not always guarantee a better stability or better performance. Emulsion stability is dependent not only on the droplet size distribution of the internal phase but also on many other factors such as rheological and electrical properties. Moreover, in some cosmetic applications, an excessive stability may not even be desirable from the product performance viewpoint.

EXPERIMENTAL

All emulsions were prepared in a 500-ml glass beaker equipped with four baffles as shown in Figure 1. A six-blade, stainless steel turbine with 50 mm diameter and 20 mm height, set 15 mm above the bottom of the beaker, was used for mixing. The mixer, driven by a powerful motor, rotates at exactly preset speeds virtually unaffected by viscosity variations during the mixing operation. In most instances, the internal phase of the emulsion was first heated to a desired temperature in the 500-ml beaker. A predetermined amount of the external phase, heated to the same temperature as the internal phase, was then added to start first-stage emulsification at a preset speed. The portion of the external phase withheld for second-stage addition was kept at approximately 20°C and later added to the emulsion concentrate at an approximately constant rate of 150 ml/min through a funnel. The finished emulsion was then mixed to uniformity and the droplet size distribution was

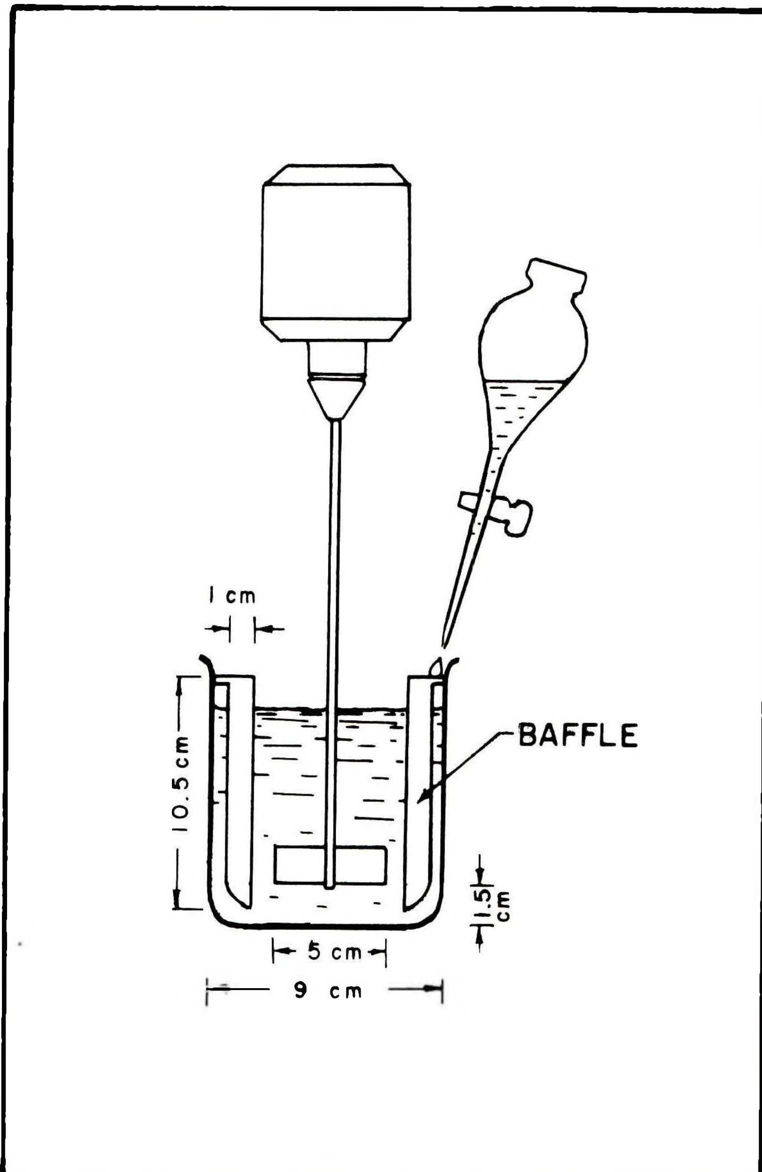

Figure 1. Experimental apparatus

obtained photomicrographically. The average droplet size represents an arithmetic average of approximately 200 droplets.

A parameter, α, was defined as the percentage of a given phase withheld for a later addition. Thus $\alpha = 0$ represents no withholding, i.e., conventional hot processing. Specifically, α_O and α_H represent, respectively, the percentages of the oil and aqueous phases withheld.

A thermometer placed in the beaker registered temperature changes and viscosity of the finished emulsion was measured at room temperature with a Brookfield-type viscometer

(Type B Viscometer, Model BL, by Tokyo Keiki Seizosho, Tokyo, Japan) at 30 rpm using spindle No. 3. Viscosity measurements were taken after a 1-min rotation. All emulsification operations were done carefully to assure good reproducibility. All surfactants, oils and waxes used in the formulations were cosmetic grade materials without further purification, and deionized water was used in all experiments.

RESULTS AND DISCUSSIONS

Although a great number of formulations representing a wide range of cosmetic emulsions and nonemulsions were tested by LEE in this series of investigations, because of the space limitation only the results from several representative formulations will be shown. These formulations are simplified prototype cosmetic emulsions including a cationic O/W emulsion, a nonionic W/O emulsion, a nonionic O/W emulsion and an anionic/nonionic O/W emulsion.

The cationic O/W emulsion shown in Table I represents a prototype cationic hair rinse emulsion stabilized with a popular quaternary surfactant, stearyl dimethyl benzyl ammonium chloride.

The results obtained with this cationic O/W emulsion are shown in Figure 2 where the arithmetic mean droplet diameters are plotted against α_H, the percentages of water withheld for second-stage dilution. The initial temperatures of first-stage emulsification, T_e, are also indicated in the figure. It is clear from Figure 2 that the emulsion becomes coarser as the emulsification temperature, T_e, is lowered. The mean droplet size also increases somewhat as α_H is increased beyond 50%. Below 50% α_H, the variation in the mean droplet sizes was within the experimental error.

The result is not surprising since it is expected that as emulsification temperature is lowered, emulsification becomes less efficient due to a viscosity build-up. However, for this system, no significant increase in the mean emulsion droplet size is observed until α_H is well over 50%. This means that as much as 50% of the external phase (water) of this emulsion could be withheld for a later addition at room temperature to save a considerable amount of thermal energy without adversely affecting the emulsion quality.

In general, it is easier to carry out LEE on O/W emulsions containing low solids such as a moisturizer with 70% or more external, aqueous phase. However the applicability of LEE is by no means restricted to O/W emulsions. It also works satisfactorily for W/O emulsions containing a large amount of mineral oil. An example of such a W/O emulsion is given in Table II.

Table I
Cationic O/W Emulsion

	Wt. %
Stearyl Dimethyl Benzyl Ammonium Chloride (21% active)[a]	4.0
Light Mineral Oil	4.0
Stearyl Alcohol	1.6
Water	90.4
	100.0

[a] Rohm & Haas Co., Philadelphia, Pennsylvania.

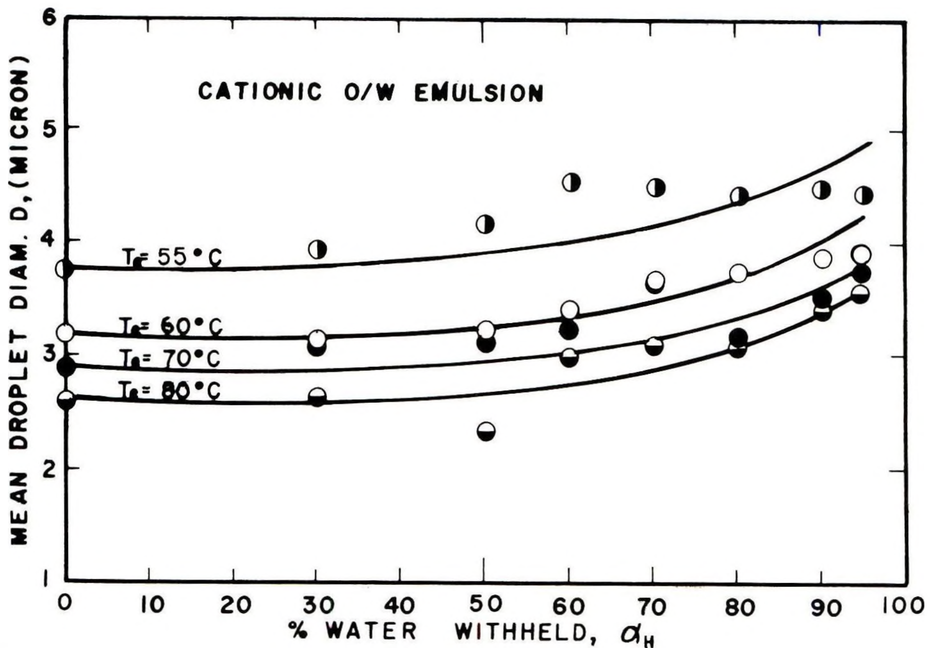

Figure 2. Effect of α_H and emulsification temperature on droplet size of the cationic O/W emulsion

The external phase of this W/O emulsion is mostly mineral oil. Varying amounts of pure mineral oil were withheld for later dilution at room temperature after completion of the first stage emulsification. It is clear from Figure 3 that LEE worked satisfactorily at an emulsification temperature of 80°C up to $\alpha_O = 70$.

A sharp increase in mean droplet size, indicating a degradation of the emulsion, was observed between $\alpha_O = 70$ and $\alpha_O = 80$. From conductivity measurements it was found that the emulsion inverted from a W/O type to an O/W type at α_O values above 70. Since the intended emulsion was a W/O type, the phase inversion resulted in the formation of coarse emulsions.

The reason for the phase inversion at a high α_O value is readily understood from the illustration in Figure 4. The dashed line in the figure represents the boundary between the first portion of the external phase used for the emulsification and the second portion used for dilution. As α_O is increased, the boundary is lowered and the ratio of the first-stage

Table II
Nonionic W/O Emulsion

	Wt. %
Water	30
Diethyleneglycol Distearate	1
Polyoxyethylene (20) Sorbitan Monostearate[a]	4
Sorbitan Sesquioleate[a]	8
Light Mineral Oil	57
	100

[a] Kao-Atlas Co., Ltd., Tokyo, Japan.

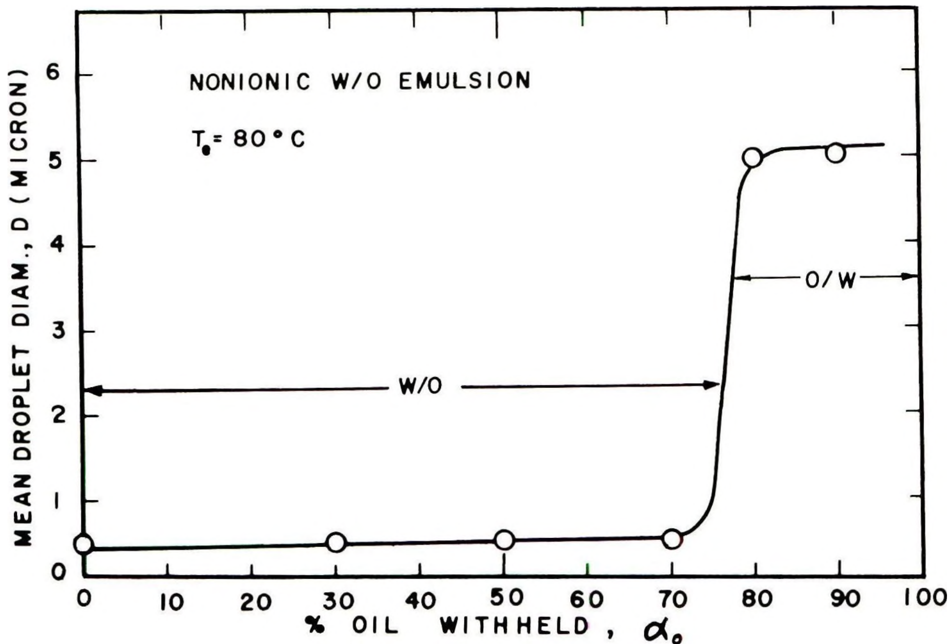

Figure 3. Effect of α_O on droplet size of the nonionic W/O emulsion

external phase to internal phase reduces accordingly. Eventually the ratio becomes too small to form the desired W/O emulsion concentrate initially and a phase inversion occurs. The subsequent addition of the cold external phase to the inverted emulsions, shown in Figure 3 for $\alpha_O > 70\%$, did not reinvert the emulsion and the final emulsion remained O/W type in high α_O range. Generally, when an inverted emulsion concentrate

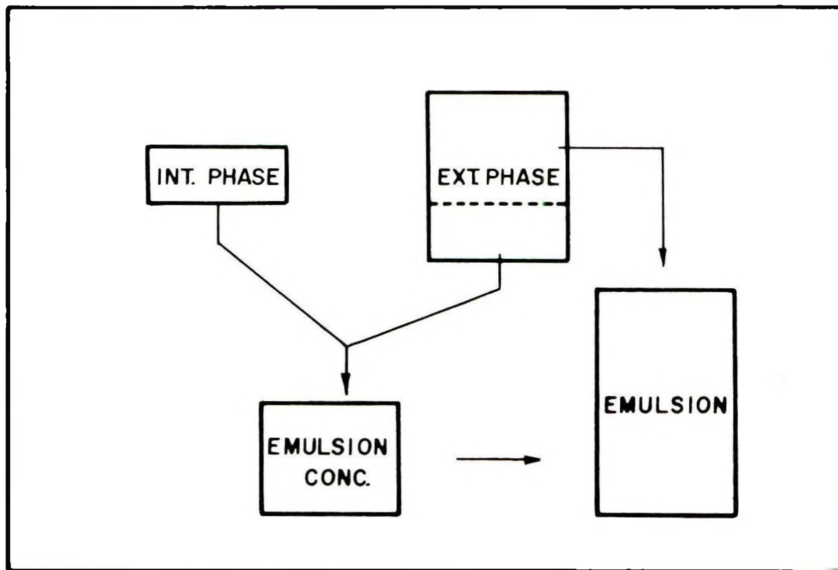

Figure 4. Illustration on α variation in LEE

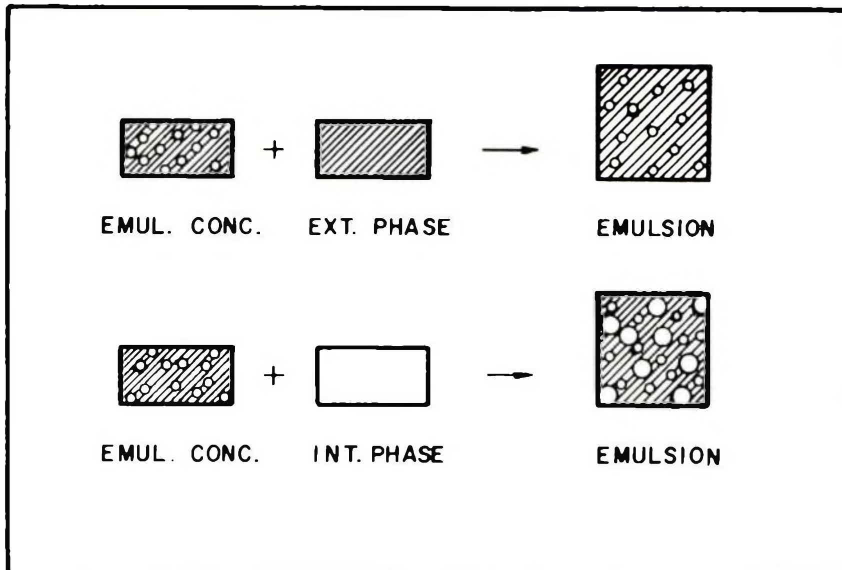

Figure 5. Illustration of adding external phase and internal phase liquids to the emulsion concentrate

fails to reinvert to form the intended type, an emulsion with poor stability or large droplets will form. This happens more frequently with emulsions having a fairly large amount of internal phase and is true with both W/O- and O/W-type emulsions.

Figure 5 illustrates the difference in diluting an emulsion concentrate with an external phase liquid and an internal phase liquid. In general a dilution with an external phase liquid at the second stage is more desirable as dilution proceeds more smoothly and the droplet size of the final emulsion does not change significantly from that of the emulsion concentrate. On the other hand, when an emulsion concentrate is diluted with the internal phase liquid, as illustrated by the lower emulsion of Figure 5, a coarse emulsion or an emulsion with a wider droplet size distribution can result. This is easily understood since, during the addition of the cold phase, the temperature of the batch is lowered considerably and unless a high-shear mixer or a homogenizer is used in the second stage the resulting emulsion will be coarse.

As a general rule in carrying out LEE, it is advisable to add the external phase liquid in the second stage after the completion of the first stage emulsification. If a homogenizing operation is desired, it is generally best to carry it out during the first stage since total batch volume at this stage is smaller and temperature is higher, making homogenization more effective.

In executing LEE, the higher the value of α, the greater is a conservation of energy expended to process the emulsion. Thus, it is of interest to determine the limit of α within which emulsions can be prepared without significantly sacrificing the emulsion quality. In most LEE applications, the greater the α value, the more concentrated and more viscous will be the emulsion concentrate. A practical limit of α is thus dependent not only on the formulation but also on the process equipment, particularly the geometry of the kettles and the type and power of the mixers. For example, a marine-type propeller mixer can handle low-viscosity emulsions adequately, but not moderate- to high-viscosity emulsions.

Table III
Nonionic O/W Emulsion

	Wt. %
Light Mineral Oil	10.0
Stearyl Alcohol	3.0
P.O.E. (5.5) Cetyl Ether	1.2
P.O.E. (10) Cetyl Ether	2.0
Propylene Glycol	5.0
Water	78.8
	100.0

A turbine mixer will handle a moderately high viscosity emulsion but probably not a heavy cream. A paddle-type mixer will handle a fairly viscous cream and allows LEE processing at a relatively high α, although the rate of shear provided by such a low-speed mixer may not be sufficient for an adequate dispersion. Thus, in a practical operation, the limit of α will be decided by a number of factors.

It is, nevertheless, of interest to determine the upper limit of α by carrying out experiments up to a very high α region in the laboratory where a sufficient mixing can be provided. Table III shows an example of a low-solids, O/W emulsion stabilized with nonionic surfactants used in this series of experiments.

A rather surprising result was obtained with this emulsion at high α_H range as shown in Figure 6. As α_H increased beyond 50%, the emulsion droplets became smaller and extremely fine emulsions having averaged droplet diameter in submicron range were obtained for α_H values greater than 70%. The sharp improvement in the emulsion quality at

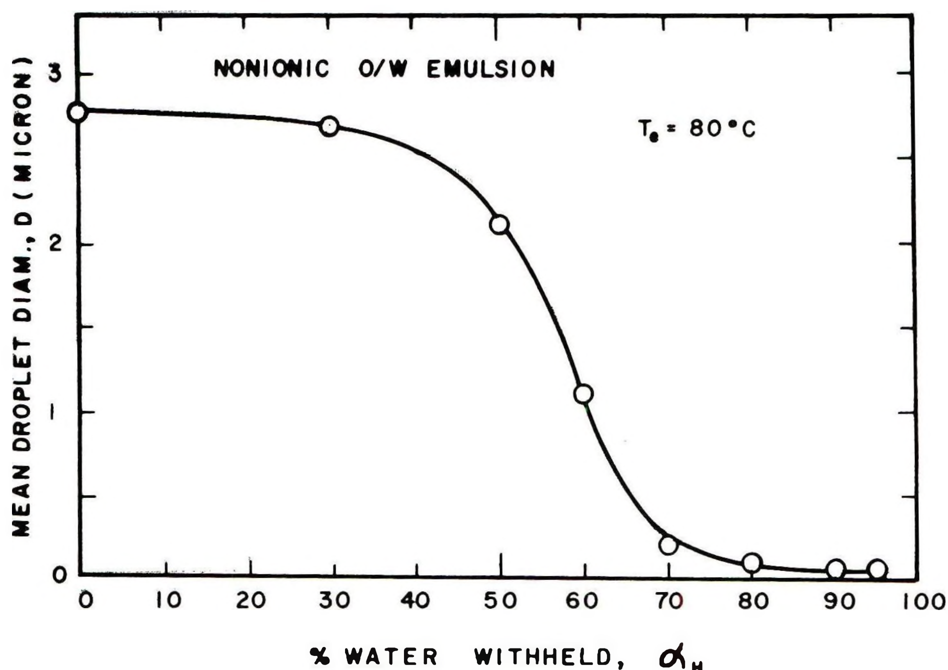

Figure 6. Effect of α_H on droplet size of the nonionic O/W emulsion

Table IV
Anionic/Nonionic O/W Emulsion

	Wt. %
Light Mineral Oil	10.0
Stearic Acid	5.0
Cetyl Alcohol	0.2
P.O.E. (5.5) Cetyl Ether[a]	0.6
P.O.E. (10) Cetyl Ether[a]	1.0
Triethanolamine	1.0
Propylene Glycol	5.0
Water	77.2
	100.0

[a] Nikko Chemicals Co., Ltd., Tokyo.

$\alpha_H = 50 \sim 70\%$ is particularly surprising and interesting. This is in contrast to the result of the W/O emulsion shown in Figure 3 where the droplet size shows an increase with α_O.

The remarkable reduction of droplet size at high α value was observed, not only in this nonionic O/W emulsion, but also in many other kinds of emulsions. Another example of this effect can be seen from the data obtained with an O/W emulsion stabilized with a mixture of anionic and nonionic surfactants, shown in Table IV.

As it is clearly shown in Figure 7 for emulsions obtained at an initial emulsification temperature of 70°C, a sharp decrease of the mean droplet size of the emulsion occurred when α_H increased beyond 50%. The viscosities of the emulsions, shown by the broken

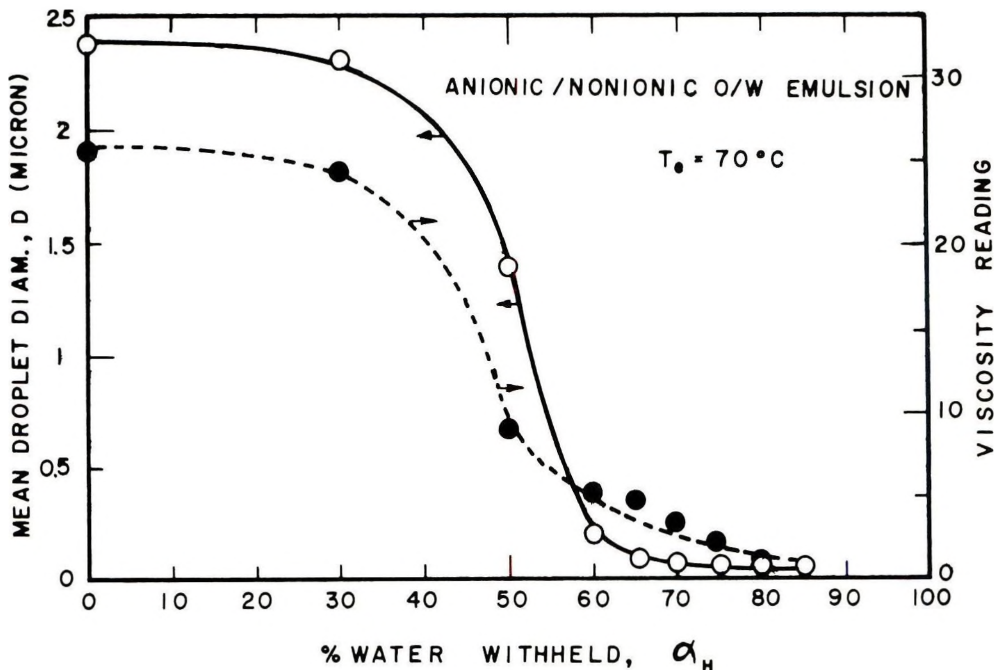

Figure 7. Effect of α_H on droplet size of anionic/nonionic O/W emulsion

line, also showed a sharp drop at about the same α_H value. The viscosities in Figure 7 are the viscosities of the final emulsions after they were cooled to room temperature approximately 2 hr after emulsification and they are represented by the Brookfield viscometer reading obtained with No. 3 spindle at 30 rpm. In making this series of emulsions, propylene glycol was placed with the first portion of the water. All other ingredients, including the nonionic surfactants, were initially placed in the oil phase.

The remarkable improvement of the emulsion at high α values is rather intriguing as it offers a possibility of making better emulsion while conserving energy. In practical applications, emulsification at a high α value means there is practically no need for cooling the batch after dilution. Not only a great saving of energy is possible, but a substantial reduction in processing time can also be achieved.

Further investigation of this effect revealed that it occurred in many systems, particularly in O/W emulsions. The cationic O/W emulsion of Table I did not show the effect at high α_H values. However, when this system was reexamined by using a similar but more concentrated cationic surfactant, a very different result was obtained. The revised formula used in this study is shown in Table V.

Stearyl dimethyl benzyl ammonium chloride (80%) is similar to stearyl dimethyl benzyl ammonium chloride (21%), except that the content of active stearyl dimethyl benzyl ammonium chloride is 80% instead of 21%. The constituents of the other components, however, may be significantly different in these two surfactants. The amount of stearyl alcohol was increased in the revised formula to compensate for the lowered viscosity.

The data for this emulsion obtained at emulsification temperature of 80°C are shown in Figure 8. Here again a sharp decrease in the droplet size is observed in the high α_H region. Interestingly, an optimum point appears to exist at α_H value of about 83% for this cationic emulsion. This means that above or below 83% α_H, the emulsions became coarser.

The existence of an optimum point is not completely surprising in view of a previous finding by Lin, Kurihara and Ohta (3). The authors pointed out the importance of a small amount of solubilized water present in the oil phase prior to forming O/W emulsions under a low mixing speed. They discovered that by initially solubilizing a small amount of water in the oil phase containing the surfactant, a remarkable improvement of emulsification efficiency could be achieved in some systems. The amount of the presolubilized water was found to be very critical, as an insufficient or excessive amount would make emulsification less efficient. They suggested that the solubilization was the first step in forming a (W/O)/W type double emulsion which allowed a more efficient emulsification mechanism to function. This mechanism was said to be responsible for the

Table V
Cationic O/W Emulsion, Revised

	Wt. %
Stearyl Dimethyl Benzyl Ammonium Chloride (80% active)[a]	2.0
Light Mineral Oil	4.0
Stearyl Alcohol	2.5
Water	91.5
	100.0

[a] Toho Chemical Industry Co., Ltd., Tokyo, Japan.

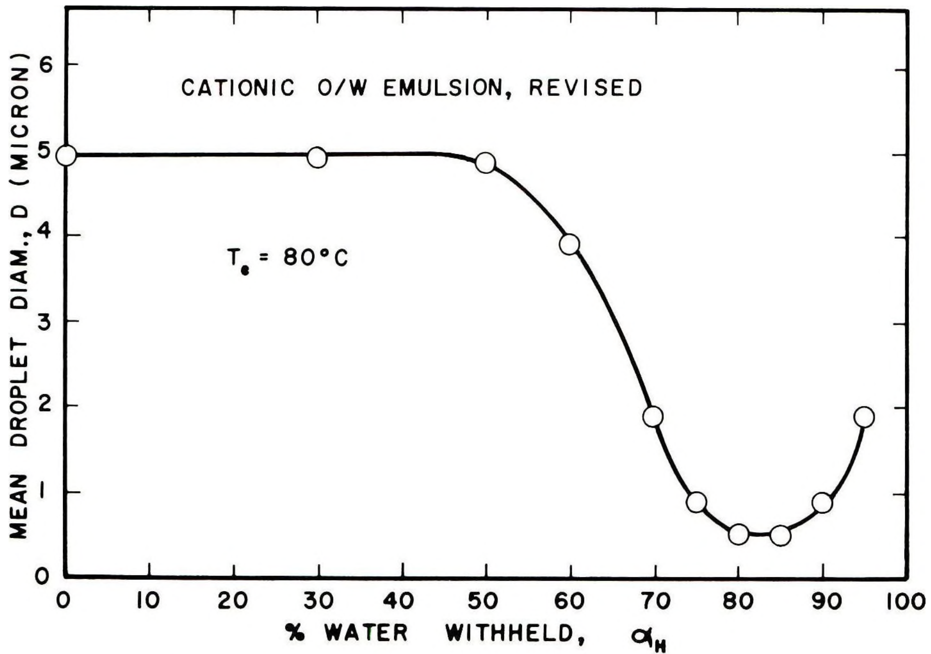

Figure 8. Effect of α_H on droplet size of cationic O/W emulsion

observed marked difference in the droplet size distributions of the emulsions made with different initial surfactant locations.

In their later work, Lin, Kurihara and Ohta (4, 5) further examined the role of the solubilized water in O/W emulsification. They reported that in many emulsified systems stabilized with various surfactants, the point of optimum emulsification corresponded to the point of maximum solubilization. It was suggested that a solubilization measurement could be utilized to predict the location of the optimum emulsification point.

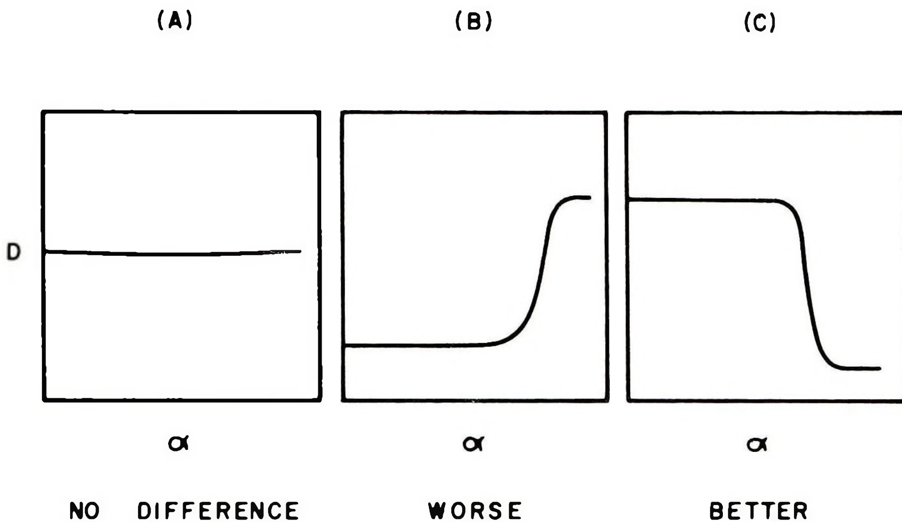

Figure 9. Three types of emulsions

When LEE is carried out for O/W emulsification in a high α_H region, the amount of the initial water in the emulsion concentrate will be relatively small. It is probable that at some point corresponding to a certain α_H value, the mechanism mentioned above will function most effectively, resulting in a formation of very fine emulsions.

In summary, the effect of α varies greatly depending on the emulsion system, but may be roughly classified into the three categories shown in Figure 9. The emulsion (A) shows little or no difference in the emulsion droplet size with respect to α. Emulsion (B) becomes coarse at a certain α value due to a phase inversion. In some systems such a degradation of the emulsion is not due to a phase inversion, but rather to the excessive viscosity build-up of the concentrate in high α region making mixing and first-stage emulsification ineffective. Emulsion (C) illustrates a sharp improvement of emulsification in higher α range. In some systems, optimum points were observed at a high α value, which may be regarded as a variation of the type (C).

CONCLUSIONS

It has been demonstrated that LEE can be applied effectively in commercially processing a wide variety of emulsions. An investigation of the qualities of emulsions made by such a technique revealed that not only the initial emulsification temperature is an important factor, but also the amount of the diluting phase withheld and the extent of mixing. A proper control of these variables will enable one to process a desired emulsion by LEE with a definite economical advantage. The finding of a marked reduction of droplet size in high α region opens an intriguing possibility of making a very fine emulsion with LEE while conserving a great deal of energy and reducing considerably the required processing time.

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Imidazolidinyl urea activity against Pseudomonas

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Synopsis

Pseudomonas contamination of cosmetics is a major concern in the cosmetic industry because pseudomonads are so widely distributed in nature, so adaptable, and so resistant to most antimicrobials. Eleven ATCC-type pseudomonads, representing ones of concern for contaminating cosmetic products, and seventeen "wild" pseudomonads, isolated from a variety of contaminated cosmetic products, were screened and were found to differ in their vulnerability to IMIDAZOLIDINYL UREA[1] alone or in combination with parabens. Screening experiments were carried out to study variables such as incubation time, incubation temperature and pH with the purpose of learning how to design and interpret *Pseudomonas* screening experiments. Experimental data are presented contrasting inadequate or marginal preservation of cosmetic lotions and shampoos with adequate preservation of these products. It was found, for example, that the parabens alone provided inadequate protection AGAINST PSEUDOMONAS growth in several creams, lotions, and shampoos. Addition of Imidazolidinyl Urea to these formulations is shown to enhance the range of effectiveness and to give adequate protection against *Pseudomonas* contamination.

INTRODUCTION

Pseudomonas contamination of cosmetic products continues to be a serious problem in the cosmetic industry (1, 2). Essentially all types of water-containing products are vulnerable to this versatile, adaptable, sometimes pathogenic, gram-negative bacterium (3). Because its nutritional requirements are minimal (3), it often survives and multiplies under conditions where other microorganisms cannot. Even in distilled water, *Pseudomonas* has been reported to grow to counts of one million per ml or more (4, 5). Contamination with the single species *Pseudomonas aeruginosa* has alone accounted for numerous recalls of products from the market (6).

Pseudomonads are difficult to avoid because they are widely distributed in water and soil (3) and are commonly found on the skin and on particles of dust in the air (6). The solution to the problem of *Pseudomonas* contamination of cosmetics is good manufacturing procedures plus the incorporation of an effective preservative system, but the choice of a preservative system is complicated by several factors: most commonly used preservatives are not effective against *Pseudomonas;* preservatives are frequently inactivated by other

[1]GERMALL 115, registered trademark of Sutton Laboratories, Inc., Roselle, NJ 07203.

components of a cosmetic formulation; and there are hundreds of *Pseudomonas* species and an almost limitless number of strains, which may differ in their vulnerability to antimicrobials.

It was therefore of interest to examine the behavior of a variety of pseudomonads in the presence of Imidazolidinyl Urea[1] (7), a commonly used (8) cosmetic preservative, in a laboratory screening test. Of course no screening test can substitute for the testing of a finished product. Only by challenge testing the final cosmetic formulation can one hope to establish whether or not a cosmetic is adequately preserved. Nevertheless, screening experiments can sometimes provide general information, which the authors felt might help cosmetic chemists and microbiologists to recognize the complexities and limitations of all testing procedures with *Pseudomonas*.

EXPERIMENTAL

METHODS

Since different screening methods often give different results (9), a modification of an AOAC screening test (10) was chosen which was consistent with general practice, although somewhat more rigorous than usual. The choice was made to ignore bacterial counting and to require complete kill. This unusually rigorous approach meant that a 99.9% kill would be recorded in this study as "no kill," because growth would occur in a subculture even if only one organism survived. Nevertheless, the authors felt that this adaptation of the phenol coefficient method (10), spread out over several days, more closely represented the needed activity against *Pseudomonas*. An incubation temperature of 37°C for *P. aeruginosa* is recommended by AOAC (10) and others (11), and is considered by many (12) as the optimum temperature for growth of most bacteria. Since the USP XIX (13) recommends an incubation temperature for bacteria of 30–35°C, the choice was made in the study reported here to routinely incubate at 35°C. A third basic choice was to incubate in dilute nutrient media (see below), following the often-repeated suggestion by Goldman (14) that such dilute media best simulate conditions of growth in cosmetics.

Initial screening was done with 11 pseudomonads purchased from ATCC (15), including those types most likely to be found in cosmetics. A 0.3% solution (16) of Imidazolidinyl Urea (Germall 115-Sutton Laboratories, Inc., Roselle, N.Y. 07203) was challenged with approximately 10^6 pseudomonads/ml. All pH's were adjusted to 7.0, because for most bacteria the optimum pH for growth generally lies between 6 and 8 (3, 12, 17). Table I shows the ATCC pseudomonads tested and includes strains of *P. aeruginosa* recommended by the USP (13) for testing antimicrobial effectiveness (i.e., *P. aeruginosa* ATCC 9027), by the AOAC (10) for disinfectant testing (i.e., *P. aeruginosa* ATCC 15442), by the CTFA Preservation Subcommittee (11) for testing of lotions (i.e., *P. aeruginosa* ATCC 13388), by the FDA (18) for testing of antiseptics (i.e., *P. aeruginosa* ATCC 14502) and the neotype strain (19) which is also used for antibiotic testing (i.e., *P. aeruginosa* ATCC 10145). The other pseudomonads tested (*P. cepacia, P. putida, P. stutzeri, P. fluorescens* and *P. aureofaciens*) are neotype strains of pseudomonads of interest to cosmetic microbiologists, and are species generally found in soil and water.

Cultures were maintained on Trypic Soy Agar (TSA, Difco Laboratories, Detroit, Michigan) at 5°C. They were transferred approximately once a month.

[1]GERMALL 115, registered trademark of Sutton Laboratories, Inc., Roselle, NJ 07203.

Table I
Challenge of 0.3% Imidazolidinyl Urea Solutions with ATCC Pseudomonads
(+ = Growth, − = No Growth)

Species	ATCC Number	Subculture After Incubation Times (Days) 1	2	3	7	14	21	28
P. aeruginosa	9027	−	−	−				
P. aeruginosa	15442	−	−	−				
P. aeruginosa	13388	−	−	−				
P. aeruginosa	14502	−	−	−				
P. aeruginosa	10145	−	−	−				
P. cepacia	10856	−	−	−				
P. cepacia	25416	−	−	−				
P. putida	12633	+	+	+	−	−	−	−
P. stutzeri	17588	−	−	−				
P. fluorescens	13525	−	−	−				
P. aureofaciens	13985	+	+	+	−	−	−	−

The screening procedure was an adaptation of a phenol coefficient procedure commonly used for disinfectants (10). Each inoculum was prepared by inoculating the organism into AOAC Letheen Broth (BBL 10914) and incubating for 24 hr at 35°C. A 0.5-ml aliquot of the 24-hr broth culture was added to 4.5 ml of the aqueous Imidazolidinyl Urea solution to be challenged. Diluting the broth culture tenfold by mixing with the Imidazolidinyl Urea solution resulted in a microbial count of approximately 10^6 organisms per ml. The mixture to be incubated also contained dilute (1:10) broth as nutrient and had an antimicrobial concentration reduced below the nominal concentration by 10% (e.g., the so-called 0.3% Imidazolidinyl Urea solution was actually 0.27% after addition of the 24-hr broth culture).

Challenged solutions or products were sampled with a 4-mm id transfer loop and subcultured in AOAC Letheen Broth. After incubating for 48 hr at 35°C, the decision of "growth" vs. "no growth" in the subculture was made by inspection.

RESULTS OF SCREENING

Inoculated solutions were routinely sampled after 1, 2 and 3 days (d), and the samples subcultured in nutrient broth to determine whether viable organisms were still present. As shown in Table I, 9 of the 11 ATCC pseudomonads were killed within 24 hr. Viable organisms of *P. putida* and *P. aureofaciens* were present for the first 3 d, but when samples were taken after longer incubation times, it was clear that both organisms were killed within 7 d.

When 0.5% solutions of Imidazolidinyl Urea were challenged with *P. putida* and *P. aureofaciens*, both organisms were killed within 2 d (Table II). When a frequently used (20) preservative system, 0.3% Imidazolidinyl Urea plus 0.2% methylparaben plus 0.1% propylparaben, was used, both *P. putida* and *P. aureofaciens* were killed within 1 d (Table III).

Screening experiments were then extended to "house" pseudomonads, organisms that had been found at various times in cosmetic manufacturing plants or in cosmetic products. Microbiologists in many cosmetic companies kindly supplied samples or "slants" of these wild, possibly mutated organisms, all of which had been identified as *Pseudomonas*. The

Table II
Challenge of 0.5% Imidazolidinyl Urea Solutions with ATCC Pseudomonads
(+ = Growth, − = No Growth)

Species	ATCC Number	Subculture After Incubation Times (Days) 1	2	3	7	14	21	28
P. putida	12633	+	−	−				
P. aureofaciens	13985	+	−	−				

traditional wisdom in the field is that mutated types are often harder to kill than the "tame" ATCC types.

As shown in Table IV, a 0.3% solution of Imidazolidinyl Urea was challenged with 17 different "house" pseudomonads in the usual way (10^6/ml, pH 7.0, etc.). Twelve of the seventeen were completely killed within 1 d, one survived for 1 d, but not for 2 d, and four survived for 3 d. Extending the incubation time gave complete kill of three of the four resistant strains, but one "house" pseudomonad, number 37-3, survived for the full 28 d.

The four "house" pseudomonads that survived for three or more days in 0.3% Imidazolidinyl Urea solution were inoculated in the usual way into 0.5% Imidazolidinyl Urea solutions. As shown in Table V, two of the four pseudomonads were killed by 0.5% solution within 1 d, but the other two survived for 3 d. By 7 d, however, both of these two resistant "house" pseudomonad were also killed by 0.5% Imidazolidinyl Urea solution.

The combination preservative system 0.3% Imidazolidinyl Urea plus 0.2% methylparaben plus 0.1% propylparaben killed three of the four resistant "house" pseudomonads within 1 d and killed the exceptionally resistant pseudomonad number 37-3 within 2 d (Table VI). Reducing the level of Imidazolidinyl Urea from 0.3% to 0.2% gave a system which killed three of the four organisms within 1 d, but permitted the exceptional pseudomonad number 37-3 to survive for 3 d. Raising the level of Imidazolidinyl Urea from 0.3% to 0.5%, still in combination with 0.2% methylparaben plus 0.1% propylparaben, gave a system which killed all four pseudomonads, including 37-3, within 1 d.

These screening tests show that pseudomonads differ considerably in their vulnerability to a given antimicrobial, and that a so-called "adequate preservative system" against *Pseudomonas* depends in large part on the specific pseudomonad used in the test. Even the most resistant pseudomonad can apparently be killed by the right preservative system; but the test results warn that, even after full microbial testing, a mutated pseudomonad might later grow in an otherwise satisfactorily preserved product. Even after production is routine, good housekeeping is essential and constant watchfulness is prudent. After everything is worked out and running, the job of surveillance must continue.

Table III
Challenge of 0.3% Imidazolidinyl Urea Plus 0.2% Methylparaben Plus 0.1% Propylparaben Solutions with ATCC Pseudomonads(+ = Growth, − = No Growth)

Species	ATCC Number	Subculture After Incubation Times (Days) 1	2	3	7	14	21	28
P. putida	12633	−	−	−				
P. aureofaciens	13985	−	−	−				

Table IV
Challenge of 0.3% Imidazolidinyl Urea Solutions with "House" Pseudomonads
(+ = Growth, − = No Growth)

Pseudomonas	Subculture After Incubation Times (Days)						
Code Number	1	2	3	7	14	21	28
28	−	−	−				
34A	−	−	−				
37-1	−	−	−				
37-2	−	−	−				
37-3	+	+	+	+	+	+	+
37-4	−	−	−				
38-1	+	+	+	−	−	−	−
38-2	−	−	−				
41A	+	+	+	−	−	−	−
82A	+	−	−				
82B	−	−	−				
82C	−	−	−				
83A	+	+	+	−	−	−	−
83B	−	−	−				
87A	−	−	−				
87B	−	−	−				
99	−	−	−				

In order to test the effect of pH, a series of challenges were run on 0.3% Imidazolidinyl Urea solutions at pHs of 5.0, 6.0, 7.0, 8.0 and 9.0, using five different ATCC pseudomonad species (Table VII). In this series of challenges, *P. aeruginosa*, *P. putida* and *P. auerofaciens* were all completely killed at all pHs within 1 d. At pH 5.0, *P. cepacia* appeared to survive for 1 d, but not for 2 d. Interestingly, *P. fluorescens* survived for 3 d at pH 5.0 even though it was completely killed in 1 d at pH 7.0. At pH 5.0, *P. fluorescens* was killed within 7 d, but its increased survival at pH 5.0 suggests caution in assuming that optimum growth occurs for all pseudomonads at pH 6–8.

It is well documented that antimicrobials act faster (21) and better (22) at elevated temperatures, and that an increase in temperature has a potentiating effect on a preservative system (23). Our studies tended to confirm these generalizations. The five ATCC pseudomonads shown in Table VIII all were capable of surviving for long periods in dilute nutrient broth alone at 25, 35 or 45°C. In dilute nutrient broth also containing 0.3% Imidazolidinyl Urea, pseudomonads were killed at all temperatures, but survival times of the pseudomonads tended to be longer at 25°C and shorter at 45°C than at 35°C. The

Table V
Challenge of 0.5% Imidazolidinyl Urea Solutions with "House" Pseudomonads
(+ = Growth, − = No Growth)

Pseudomonas	Subculture After Incubation Times (Days)						
Code Number	1	2	3	7	14	21	28
37-3	+	+	+	−	−	−	−
38-1	+	+	+	−	−	−	−
41A	−	−	−				
83A	−	−	−				

Table VI
Challenge of 0.3% Imidazolidinyl Urea Plus 0.2% Methylparaben Plus 0.1% Propylparaben Solutions with "House" Pseudomonads (+ = Growth, − = No Growth)

Pseudomonas Code Number	Subculture After Incubation Times (Days) 1	2	3	7	14	21	28
37-3	+	−	−				
38-1	−	−	−				
41A	−	−	−				
83A	−	−	−				

one organism that survived 7 d at 25°C, *P. fluorescens*, was killed within 3 d at 25°C in 0.5% Imidazolidinyl Urea solution.

Screening the six most highly resistant "house" pseudomonads at incubation temperatures of 25, 35 and 45°C showed the same general behavior, although in this case survival times were approximately the same at incubation temperatures of 25 and 35°C (Table IX). Survival times at 45°C were shorter in all cases. The one organism that survived 7 d at 25 or 35°C not only was killed in screening tests by 0.5% Imidazolidinyl Urea solution, but also was surprisingly easy to kill with 0.3% Imidazolidinyl Urea solution in an actual cosmetic product. This latter observation reminds us once again that screening experiments may provide interesting general patterns of behavior, but they cannot substitute for the testing of actual cosmetic formulations.

APPLICATIONS TO COSMETICS

Using this same rigorous screening procedure, where anything other than complete kill is survival and therefore "growth" in the subculture, three cosmetic products now on the market from a major cosmetic company were challenged with five different pseudomonads. All three of these products as sold today are preserved with parabens alone. The results recorded in Table X represent subcultures after 2 d incubation. The cleansing cream with parabens alone showed survival of both types of ATCC *P. aeruginosa* and two of three "house" pseudomonads. When 0.3% Imidazolidinyl Urea was added, all five organisms were killed completely within 2 d. With the moisturizing lotion and the nutrient emulsion, three of the five pseudomonads survived after 2 d. Addition of 0.3% Imidazolidinyl Urea gave products which showed no growth on subculture, that is, complete kill of all five pseudomonads, within 2 days.

Table VII
Challenge of 0.3% Imidazolidinyl Urea Solutions with ATCC Pseudomonads at Different pHs (+ = Growth, − = No Growth)

		Subcultures of Different pH Incubations (Time in Days)															
		5.0				6.0			7.0			8.0			9.0		
Species	ATCC Number	1	2	3	7	1	2	3	1	2	3	1	2	3	1	2	3
P. aueruginosa	15442	−	−	−		−	−	−	−	−	−	−	−	−	−	−	−
P. cepacia	25416	+	−	−		−	−	−	−	−	−	−	−	−	−	−	−
P. putida	12633	−	−	−		−	−	−	−	−	−	−	−	−	−	−	−
P. fluorescens	13525	+	+	+	−	−	−	−	−	−	−	−	−	−	−	−	−
P. aureofaciens	13985	−	−	−		−	−	−	−	−	−	−	−	−	−	−	−

Table VIII
Challenge of 0.3% Imidazolidinyl Urea Solutions with ATCC Pseudomonads at Different Incubation Temperatures (+ = Growth, − = No Growth)

		Subcultures of Different Incubation Temperatures (Time in Days)											
		25°C				35°C				45°C			
Species	ATCC Number	1	2	3	7	1	2	3	7	1	2	3	7
P. aueruginosa	15442	+	+	−	−	−	−	−		−	−	−	
P. cepacia	25416	+	−	−	−	−	−	−		−	−	−	
P. putida	12633	+	+	−	−	+	+	+	−	−	−	−	
P. fluorescens	13525	+	+	+	+	−	−	−		−	−	−	
P. aureofaciens	13985	+	+	−	−	+	+	+	−	−	−	−	

This same test procedure was used on a model shampoo formulation (Table XI) recently proposed (24) by an ASTM Cosmetic Preservative Task Force for use in preservative testing. Challenges were carried out on the shampoo as described and also with added parabens or Imidazolidinyl Urea, or both. Each formulation was challenged not only with *P. aeruginosa*, but also with *Escherichia coli*, *Staphylococcus aureus*, *Candida albicans* and *Aspergillus niger*. The results are shown (Table XII) for subcultures after incubation for 2 d. The shampoo alone and the shampoo containing parabens were both able to kill *E. coli* and *S. aureus*, but not the other three organisms. When 0.3% Imidazolidinyl Urea was added to either one of those formulations, the product killed all five organisms. In this series, with these specific microorganisms, the Imidazolidinyl Urea alone was capable of killing the organisms that the unpreserved shampoo did not kill. However since variations exist even among different types of the same species and since the Imidazolidinyl Urea-paraben preservative system has wide-range effectiveness, it would be wise to include the parabens in the preservative system as added insurance.

Another example of the parabens' inability to withstand *Pseudomonas* challenge was reported recently (25) with a cosmetic lotion. The cosmetic lotion was developed by Amerchol (26) and used by the CTFA Preservation Subcommittee (11) for evaluation of methods of determining preservative efficacy. The Subcommittee tested (11) the formulation without any added preservative, and with 0.2% methylparaben plus 0.1% propylparaben. After inoculation of both lotions with approximately 10^6 organisms/ml of four different microorganisms, they did microbial counts over a 28-d period. The lotion without any added preservative killed *S. aureus* within 7 d, but failed to kill the other three

Table IX
Challenge of 0.3% Imidazolidinyl Urea Solutions with "House" Pseudomonads (+ = Growth, − = No Growth)

	Subcultures of Different Incubation Temperatures (Time in Days)											
Pseudomonas	25°C				35°C				45°C			
Code Number	1	2	3	7	1	2	3	7	1	2	3	7
34A	+	+	−	−	−	−	−		−	−	−	
37-3	+	+	+	+	+	+	+	+	+	+	+	−
38-1	+	+	−	−	+	+	+	−	−	−	−	
41A	+	+	−	−	+	+	+	−	−	−	−	
82A	+	+	+	−	+	−	−		−	−	−	
83A	+	+	+	−	+	+	+	−	−	−	−	

Table X
Challenge of Cosmetic Emulsions Containing Parabens
(+ = Growth, − = No Growth; After 2 Days)

	P. Aeruginosa ATCC 15442	P. Aeruginosa ATCC 13388	"House" Pseudomonad 34A	"House" Pseudomonad 37-3	"House" Pseudomonad 82C
Cleansing Cream	+	+	+	+	−
Cleansing Cream + 0.3% Imidazolidinyl Urea	−	−	−	−	−
Moisturizing Lotion	+	+	−	+	−
Moisturizing Lotion + 0.3% Imidazolidinyl Urea	−	−	−	−	−
Nutrient Emulsion	+	+	−	+	−
Nutrient Emulsion + 0.3% Imidazolidinyl Urea	−	−	−	−	−

organisms. The lotion with 0.2% methylparaben plus 0.1% propylparaben killed *S. aureus, E. coli* and *C. albicans,* but failed to kill *P. aeruginosa,* which remained at high levels throughout the 28-d study period. Addition of 0.3% Imidazolidinyl Urea to the lotion already containing the parabens gave a lotion which was capable of killing *P. aeruginosa* within 2 d and which showed no growth over the full 28-d test period (25). A second and third re-challenge of the lotion with *P. aeruginosa* also showed no survival within 2 d of the re-challenge, and the pseudomonad count stayed <10/ml throughout each 28-d test period.

SUMMARY

It has been shown in both screening experiments and in actual cosmetic products that Imidazolidinyl Urea is effective against a wide variety of pseudomonads. The few resistant strains that Imidazolidinyl Urea alone did not kill quickly enough at the indicated concentrations were killed quickly using an Imidazolidinyl Urea-paraben combination system. It is recommended that the combination preservative system of Imidazolidinyl Urea-methylparaben-propylparaben be used in proportions of 3:2:1, and it is strongly suggested that all microbial testing be carried out on the finished cosmetic product. Different pseudomonads vary in their susceptibility to antimicrobials, so good housekeep-

Table XI
Model Shampoo Formulation (Water Base Detergent System),
ASTM E-35. 15 Cosmetic Preservative Task Force

Ingredient	% By Weight	Grams/Kilo
Triethanolamine Lauryl Sulfate (40%)	25.00	250.0
Lauryl Diethanolamide	5.00	50.0
Amphoteric-2	5.00	50.0
Polyoxyethylene Lanolin (50%)	3.00	30.0
Phosphoric Acid	0.20	2.0
Demineralized Water	qs to 100%	

Detergent system containing an amphoteric (Miranol). Add all ingredients to mixing vessel and warm to 150°F. Sweep stir to 90°F.

Table XII
Challenge of Shampoo Formulation as Described in Table XI and with Added Preservatives
(+ = Growth, − = No Growth; After 2 Days)

	P. Aeruginosa 15442	E. Coli 10536	S. Aureus 6538	C. Albicans 10231	A. Niger 9642
Shampoo Alone	+	−	−	+	+
Shampoo + 0.2% Methyl-Paraben + 0.1% Propyl-Paraben	+	−	−	+	+
Shampoo + 0.3% Imidazolidinyl Urea	−	−	−	−	−
Shampoo + 0.3% Imidazolidinyl Urea + 0.2% Methylparaben + 0.1% Propylparaben	−	−	−	−	−

ing is essential to prevent contamination and even well established products must be checked frequently.

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(16) The concentrations referred to in the text and listed in the tables are "nominal" concentrations. In fact, after adding 1 part of inoculum to 9 parts of solution, the concentration of Imidazolidinyl Urea was reduced by 10%—that is, a "nominal" concentration of 0.3% was actually 0.27%.

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Eine Methode zur Bestimmung der Verteilungsgleichgewichte von Konservierungsmitteln in Emulsionen

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Synopsis — Partial phase separation of emulsions was achieved by ultrafiltration and ultracentrifugation without noticeable changes in partition coefficients and adsorption characteristics. The amount of preservatives in the continuous phase is then determined with the aid of HPLC. Data obtained by this technique are in good agreement with the results of microbiological challenge testing.

Problemstellung

Art und Menge eines in einer Emulsion vorliegenden Konservierungsmittels entscheiden erfahrungsgemäß nicht allein über den Schutz gegenüber mikrobiellem Befall und eventuell nachfolgendem Keimwachstum, sondern die Wirksamkeit der Konservierung hängt insbesondere auch von der jeweils vorliegenden Rezeptur ab. Dieser Rezeptureinfluß wird durch eine Reihe von Parametern bedingt, von denen die Verteilung zwischen den verschiedenen Kompartimenten der Emulsion (Wässrige Phase, Öl und Phasengrenze) und die Wiederherstellung dieser thermodynamisch bedingten Gleichgewichte nach Störung, z. B. durch Verbrauch der Substanzen im Fall mikrobieller Kontamination die wichtigsten sind. Die Konzentration der Bakteriostatika in der wässrigen Phase, in der Mikroorganismen ausschließlich wachsen, bestimmt das Ausmaß der Wechselwirkung mit den Mikroorganismen und somit auch die mikrobizide Wirkung. Zur Bestimmung der Verteilungsgleich-

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gewichte benötigt man Methoden, die eine zumindest teilweise Phasentrennung ohne Veränderung der in der Emulsion vorliegenden Lösungs- und Adsorptionsgleichgewichte gestatten. Derartige Methoden sind Ultrafiltration und Ultrazentrifugation; sie erlauben die Gewinnung der kontinuierlichen Phase — im Falle einer O/W-Emulsion also der wässrigen Phase — und bei Kenntnis der Gesamtmenge an eingesetzter Substanz die Ermittlung der Verteilungskoeffizienten. Zur Analyse der wässrigen Phase wurde fast ausschließlich die Hochdruckflüssigkeits-Chromatographie benutzt.

Experimentelles

Produkte

Die untersuchten Produkte waren Versuch-Cremes und -Lotionen, welche mit p-Hydroxybenzoesäureestern, Phenoxetol oder Formaldehyd konserviert waren. Produkt A enthielt 74,8% H_2O, 25% Öl + Emulgatoren, 0,13% Paraben A, 0,07% Paraben P, Produkt B war zusammengesetzt aus 89,8% H_2O, 10% Öl + Emulgator, 0,13 % Paraben A, 0,07% Paraben P, Produkt B' enthielt zusätzlich 2% p-Methoyzimtsäureester [5]) und 87,8% H_2O.

Ultrafiltration

Die Phasentrennung wurde, soweit es sich um fließfähige Produkte handelte, mit Ultrafiltrationszellen [1]) [2]) (∅ 46 nm) bei Drucken zwischen 1 — 2,5 bar unter Rühren durchgeführt. Die verwendeten Membranfilter waren im Fall der Trennung von Präparaten mit kontinuierlicher wässriger Phase vom Typ SM 12133 (∅ = 10 — 20 nm) oder SM 12136 (∅ = 5 — 10 nm) [3]). Bei W/O-Emulsionen können trockene Filter des Typs PSAC 04710 [2]) oder BM-500K [1]), welche die Ultrafiltration hydrophober Medien gestatten, zur Isolierung der Ölphase dienen. Das Filtrat wurde in Fraktionen von ca. 1 ml gesammelt und getrennt analysiert (1).

Ultrazentrifugation

Die Trennung viskoser Cremes wurde in einer Ultrazentrifuge [4]) unter Anwendung hoher Schwerefelder (10^5 bis $3 \cdot 10^5$ g) durchgeführt.

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[1]) Fa. Berghof/Tübingen, GFR.

[2]) Fa. Millipore/Neu-Isenburg, GFR.

[3]) Fa. Sartorius, Göttingen, GFR.

[4]) Beckman Instruments.

[5]) Neo-Heliopan, Haarmann und Reimer, Holzminden.

Um die kontinuierliche Phase problemlos zu gewinnen, wurden die Zentrifugenröhrchen aus Kunststoff in Abhängigkeit von der Höhe der einzelnen optisch erkennbaren Fraktionen zerschnitten.

Analyse

Bei wässrigen Medien wurde die Analyse (2) per HPLC [6]) unter Verwendung einer reversed-phase-Säule (ODS) durchgeführt. Als Elutionsmedium diente bei der Parabenanalyse ein Gradient 10% Äthanol/90% H_2O — 90% Äthanol/10% H_2O (sweeptime 32 min, Gradient 3, Detektorwellenlänge 254 nm, Integrator HP 3380 A, Flußrate 1,4 ml/min). Formaldehyd (= FA) wurde kolorimetrisch gemäß (3) gemessen. Im Fall hydrophober Medien wurden die Parabene UV-photometrisch als Summe analysiert.

Ergebnisse und Diskussion

Die Ultrafiltration ist mit zwei Phänomenen verknüpft, welche die Genauigkeit der Ergebnisse beeinflussen. Als erstes ist die Adsorption von polaren Substanzen am Filter zu nennen. Hierbei entstehende Fehler lassen sich dadurch vermeiden, daß man das anfangs anfallende Filtrat nicht zur analytischen Kennzeichnung des Präparates verwendet, sondern die bald eintretende Sättigung abwartet. Das zweite Phänomen ist die sogenannte Konzentrationspolarisation, unter der man die Verarmung des Filtrates an gelösten Substanzen versteht, die aus einer Penetrationsverlangsamung durch die oberhalb der Membran sich ausbildenden Gelschichten herrührt (1).

Die Konzentration an vorhandenem Konservierungsmittel wird über eine Reihe von Fraktionen hinweg messend verfolgt, um die störenden Einflüsse der beiden Phänomene zu erkennen, so daß die o. g. Fehler eliminiert werden können. In dem der Fig. 1 zugrunde liegenden Beispiel wird nur der Vorgang der Adsorption am Filtermaterial während der ersten Fraktionen dargestellt. Ein allmähliches Absinken der gefundenen Konzentrationen bei höheren Filtrat-Volumina infolge Konzentrationspolarisation, das bei nicht gerührtem Inhalt regelmäßig beobachtet wurde, war nicht meßbar. Auch die sich ergebenden kleinen Unterschiede bei Verwendung verschiedener Filter waren geringer als die Analysenfehler ($< \pm 5\%$).

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[6]) Spectra Physics 3500 bzw. Micromeritics Modell 7000.

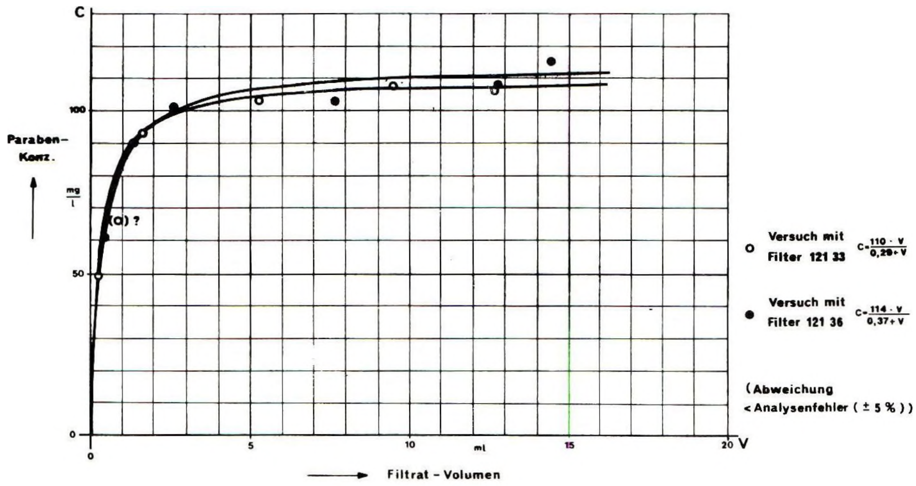

Fig. 1
Änderung der Konzentration an Paraben A mit zunehmendem Filtratvolumen.

Fig. 2 zeigt die Konzentration an Paraben P mit zunehmendem Filtratvolumen. Die Meßwerte sind aus Tabelle 1 zu ersehen. Gemäß der folgenden Berechnung werden die Verteilungskoeffizienten ermittelt, wobei man von den im Plateaubereich der Kurven vorliegenden Werten ausgeht.

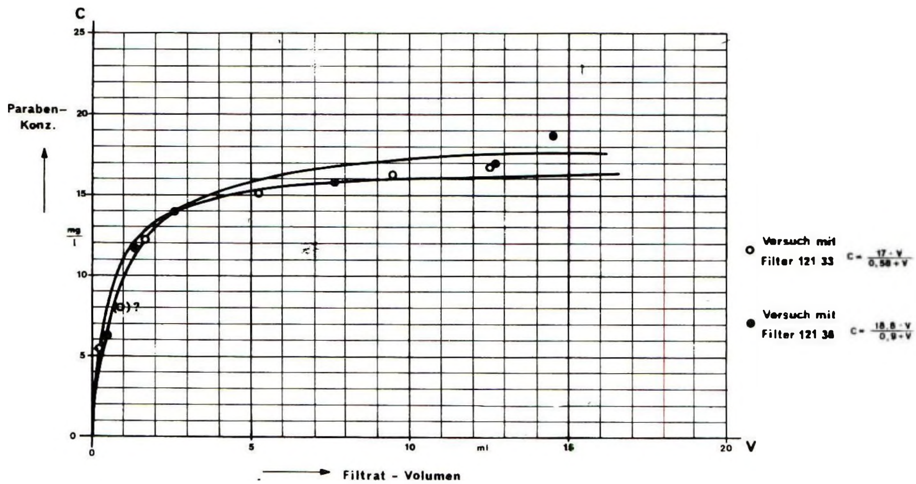

Fig. 2
Änderung der Konzentration an Paraben P mit zunehmendem Filtratvolumen.

Gefunden in der Wasserphase: Paraben A 110 mg/l (≙ 8,4 %)
Paraben P 17 mg/l (≙ 2,4 %)

→ Paraben A in der W. Ph. 81,4 mg
in der Ölph. 1218,6 mg ≙ 4687 ppm

→ Paraben P in der W. Ph. 12,6 mg
in der Ölph. 687,4 mg ≙ 2644 ppm

$$\text{Paraben A:} \quad \frac{C\ (H_2O)}{C\ (\text{Öl})} = \frac{110\ \text{ppm}}{4687\ \text{ppm}} = 0{,}0235$$

$$\text{Paraben P:} \quad \frac{C\ (H_2O)}{C\ (\text{Öl})} = \frac{17\ \text{ppm}}{2644\ \text{ppm}} = 0{,}0064$$

Tabelle 1

Ultrafiltrationsversuch an Produkt A; Filter SM 12133

Fraktion Nr.	Menge g	Paraben A mg/l	Paraben P mg/l
1	0,48	49	5,5
2	0,84	66	8,0
3	0,70	93	12,1
4	6,59	103	15,1
5	1,84	108	16,1
6	4,44	106	16,8
	14,89		

Ultrafiltrationsversuch an Produkt A; Filter SM 12136

Fraktion Nr.	Menge g	Paraben A mg/l	Paraben P mg/l
1	0,85	61	6,1
2	1,02	91	11,7
3	1,42	101	13,9
4	8,82	103	15,9
5	1,27	108	16,9
6	2,18	115	18,7
	15,56		

Fig. 3 gibt ein HPLC-Diagramm eines Produktes wieder, das zusätzlich Methylparaben und Phenoxyäthanol enthält.

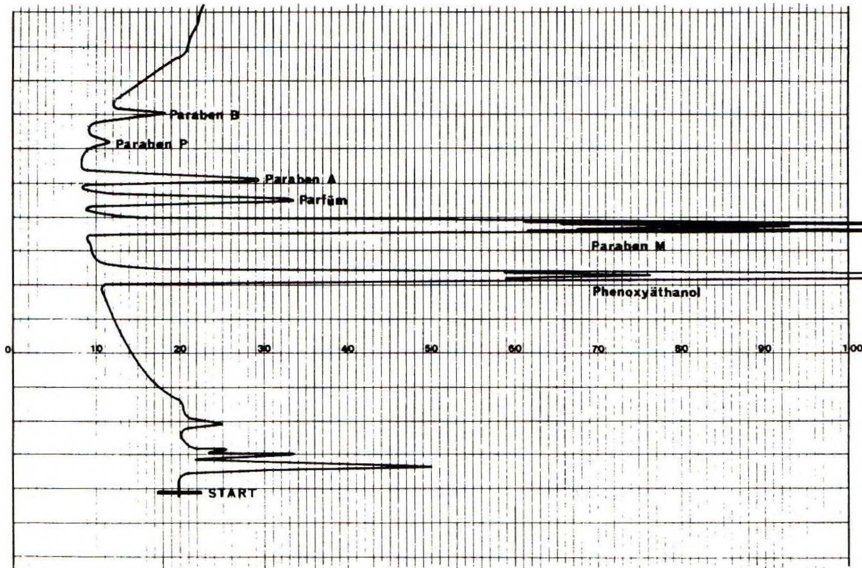

Fig. 3

HPCL-DIAGRAMM des Ultrafiltrats eines Produktes, das mit mehreren Konservierungsmitteln versehen ist.

Die Abhängigkeit der Verteilungskoeffizienten von der Zusammensetzung von Wasser- bzw. Ölphase liefert Hinweise auf die für den effektiven Einsatz nötigen Modifizierungen der Rezeptur. Erhöhte antimikrobielle Wirksamkeit erzielt man nicht nur durch bloße Konzentrationserhöhung, sondern auch durch gesteigertes chemisches Potential, welches durch die fraktionelle Sättigung ausgedrückt wird (4). Wie zu erwarten, verschiebt sich die Verteilung der Parabene zugunsten der Wasserphase bei Erhöhung der Löslichkeit der Parabene z. B. durch Aufnahme von Äthanol, Propanol etc. in die Wasserphase. Im Gegensatz dazu wird der in der Wasserphase enthaltene Anteil verringert, wenn das Mineralöl teilweise durch p-Methoxyzimtsäureester ersetzt wird.

Tabelle 2 zeigt die Verschiebung der Verteilungsgleichgewichte bei Aufnahme eines polaren Esters in die Ölphase. Der Anteil der in der wässrigen Phase gelösten Parabene sinkt beträchtlich, die mikrobielle Belastbarkeit ist dem parallel laufend viel geringer.

Tabelle 2

Verteilung der Parabene A und P und Produkt B bzw. B'

Probe	Paraben A (mg/ml)	Paraben P (mg/ml)
B'	0,062 = 6,2 % (d. Th.)	0,003 ≙ 0,15 % (d. Th.)
B	0,125 = 13,1 % (d. Th.)	0,014 = 2,8 % (d. Th.)

Tabelle 3

Belastungsfähigkeit der Produkte B bzw. B'

Keimart	Anzahl/ml Emulsion	B' negativ ab	B negativ ab
Staph. aureus	$9,5 \cdot 10^4$	4. Tag	1. Tag
E. coli	$7,7 \cdot 10^4$	4. Tag	1. Tag
Ps. aeroginosa	$11,5 \cdot 10^4$	4. Tag	1. Tag
Pen. glaucum	$2 \cdot 10^4$	2. Woche	1. Tag

Ersetzt man dagegen die Parabene durch ihre Na-Salze, so nimmt die Konzentration der Parabene in der Wasserphase zu.

Ob die Parabene in der Wasser- oder in der Fettphase vor der Emulsionsbereitung dispergiert werden, hat keinen Einfluß auf die resultierende Verteilung.

Die Bestimmung der Oberflächenspannung der Ultrafiltrate führte bei allen untersuchten Präparaten zu Werten > 40 dyn/cm, d. h. die Emulgatorkonzentration liegt unter der kritischen Micellbildungskonzentration. Damit entfällt im Normalfall die Möglichkeit der Solubilisierung von Konservierungsmitteln in Emulsionen.

Bei verkeimten Mustern taucht immer ein Reihe von Substanzpeaks im Chromatogramm auf, deren Identität unbekannt ist; ihre Herkunft aus Parabenen scheint deshalb naheliegend, da ihre Konzentration sich invers zur Paraben-Konzentration verhält (Fig. 4a und 4b).

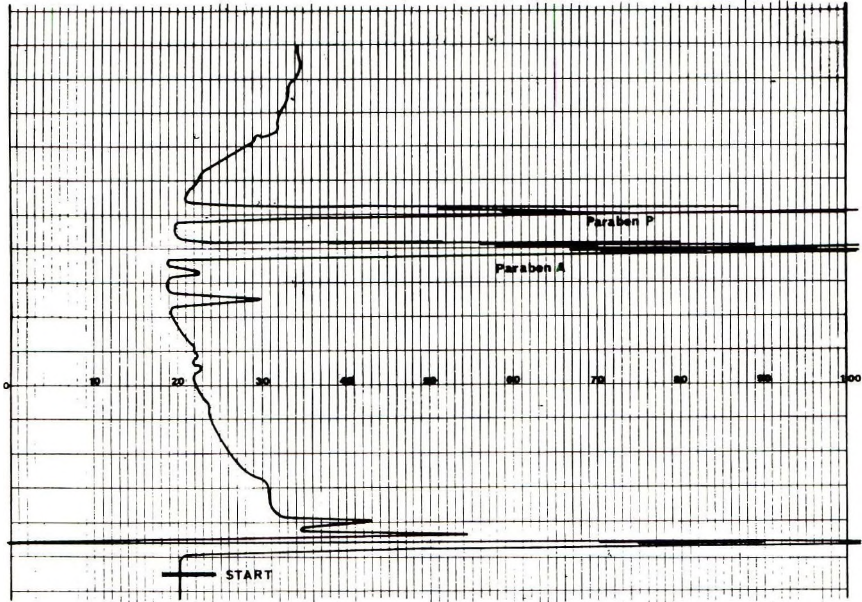

Fig. 4a
HPLC-DIAGRAMM des Ultrafiltrats eines keimfreien Produktes.

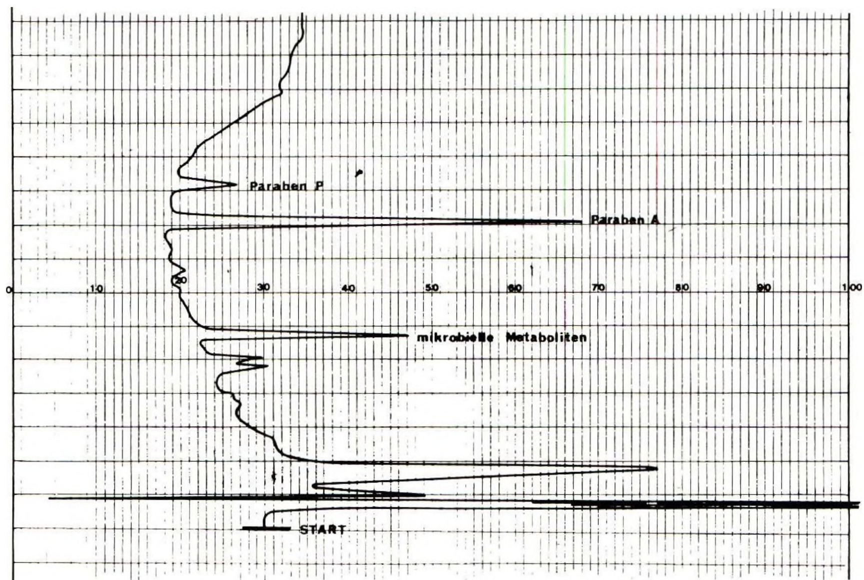

Fig. 4b
HPCL-Diagramm des gleichen Produktes bei Verkeimung mit Cephalosporium.

Nach unseren Erfahrungen ändern sich erwartungsgemäß die Verteilungsgleichgewichte in Abhängigkeit von der Lagerdauer der Präparate und der Tröpfchengrößenverteilung nicht. Beim Vorliegen kleinerer Teilchen erfolgt der Anstieg in Abhängigkeit vom Filtratvolumen verzögert, erreicht aber ebenfalls die gleiche Plateauhöhe. Das demonstriert Fig. 5, der ebenfalls Produkt A — feiner verteilt — zugrunde liegt.

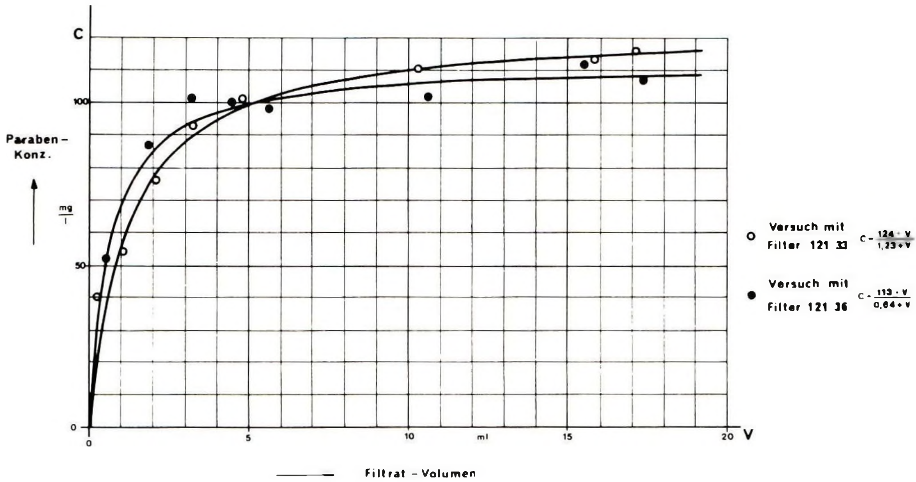

Fig. 5

Änderung der Konzentration an Paraben A mit zunehmendem Filtratvolumen.

Solange sich die Verteilungsgleichgewichte und absoluten Konzentrationen im Laufe der Zeit nicht ändern, kann man erwarten, daß die Ergebnisse des mit einer derartigen Bestimmung parallel einhergehenden mikrobiologischen Belastungstest reproduzierbar sind; der Schutz des Präparates gegenüber mikrobieller Kontamination bleibt also über einen längeren Zeitraum voll erhalten. Der aufwendige wiederholte Belastungstest kann somit auf eine schneller durchzuführende chemisch-analytische Bestimmung reduziert werden.

Gänzlich andere Verhältnisse werden bei langsam ablaufenden chemischen Reaktionen, z. B. des Konservierungsmittels mit Produktinhaltsstoffen, erreicht. FA regiert beispielsweise mit Amiden, Aminen und anderen Stoffen. Im Ultrafiltrat harnstoffhaltiger Produkte ist FA nach (3) nicht nachweisbar: Diesen Befunden entsprechen die mikrobiologischen Ergebnisse. Bei konventioneller Probenaufbereitung mit Wasserdampfdestillation dagegen läßt sich FA quantitativ wiederfinden.

Derartige Veränderungen laufen nicht innerhalb von Minuten oder Stunden nach der Herstellung ab, sondern nehmen Wochen in Anspruch. Der unmittelbar nach der Herstellung gegebene gute Schutz gegen mikrobielle Kontamination ist zum Zeitpunkt des Verbrauchs dann nicht mehr gegeben. Bei Verkeimung setzt ungehemmtes Keimwachstum ein.

Die oben angedeutete Abhängigkeit der Verteilung der teilweise wasserlöslichen Parabene von der Rezeptur und ihren Variationen und damit synchron zu beobachtende Änderungen der mikrobiellen Belastbarkeit belegen die Bedeutung derartiger Untersuchungen. Sie gestatten, den Grund für Unterschiede in der Belastbarkeit zu finden und die Optimierung der Konservierung eines Präparates zu steuern. Bei fehlender zeitlicher Veränderungen von Konservierungsmittelgehalt und -verteilung kann man über die mikrobielle Belastbarkeit die Aussage treffen, daß die Ergebnisse der einmalig durchgeführten, mikrobiologischen Untersuchung weiterhin gültig sind.

Zusammenfassung

Durch Ultrafiltration und Ultrazentrifugation ist aus Emulsionen die kontinuierliche Phase ohne merkliche Änderungen hinsichtlich Verteilungsgleichgewichten und Adsorptionscharakteristik zu gewinnen. Die Analyse per HPLC ermöglicht die quantitative Bestimmung der in der kontinuierlichen Phase enthaltenen Konservierungsmittel. Diese Daten korrelieren gut mit den Resultaten des mikrobiologischen Belastungstests.

Herrn Düsing danken wir für die Durchführung einiger Untersuchungen, Fr. Dr. Kran für die mikrobiologischen Daten.

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Effects of harvesting techniques on hydration dynamics: gravimetric studies of stratum corneum

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Synopsis

STRATUM CORNEUM specimens HARVESTED by several methods commonly used for in vitro studies have been compared by HYGROSCOPICITY. Cantharidin blisters give superior data when compared to heat, trypsin and ammonia fume separated specimens. As the cantharidin data represent skin from a different age group and body site, a comparison to animal and human data in the literature is made.

INTRODUCTION

Many experiments have been done using isolated sheets of stratum corneum. In 1963, Kligman and Christophers reported three techniques for preparation of these sheets: 1) exposure to ammonia fumes for 30 min in a closed vessel over concentrated ammonium hydroxide, 2) heat separation by immersing skin sandwiched between metal plates in a water bath at 56°C for 2 min followed by the removal of the epidermis, which is floated overnight in a buffered 0.0001% solution of trypsin and 3) intraepidermal blistering with a 0.2% cantharidin solution occluded for 8–10 hr (1). They claimed that stratum corneum prepared by canthardin and heat methods was the same with respect to water diffusion and stress-strain characteristics, but no data was presented. Onken and Moyer obtained stratum corneum by digesting skin samples for 24 hr in 3% trypsin (2). Proteolytic enzymes other than trypsin have been used. Kligman and Christophers used papain, pepsin, ficin and elastase, but found them less effective than trypsin. Pronase is also useful (3). Middleton used 2*M* urea with 0.5% trypsin to obtain guinea pig foot-pad corneum which he compared with foot pad removed at 60°C for 30 min (4). He found no difference in the water binding ability of guinea pig foot pad prepared in these two ways. Another popular technique requires immersing skin in 2*M* sodium bromide for 2 hr at 37°C (5). Stretching has also been employed (6). Marzulli uses tape stripping of stratum corneum eluted with organic solvents (7).

Each technique has its limitations. Some can only be utilized on excised skin. Several methods employ prolonged water immersion. The effects of enzymes on epidermal

protein cannot be ignored (8-9). Singer and Vinson showed a decrease in water binding capacity in corneum of neonatal rats after 17 hr of water immersion (10). At 93% relative humidity they found water immersed corneum had a 47% increase over its dry weight as compared to 60% for the control. However Scheuplein reported that in comparing heat-separated stratum corneum to cantharidin blister tops, "neither technique measurably damages the membrane. Some protein denaturation and dissolution must indeed occur; but if this changed permeability, the differences are within the average experimental error, viz 20%, and cannot be isolated from the variations between different samples." (11)

Polano et al. studied the effect of heat plus trypsin preparation of stratum corneum by measuring in vitro penetration of methyl nicotinate. They found no significant difference in penetration utilizing 1–16 min immersions in 60°C water and concentrations of trypsin up to 0.1% (12).

It seems reasonable that the stratum corneum is damaged by the separation procedures; but due to difficulty in quantifying the denaturation, many investigators have chosen to ignore it. A sensitive, accurate and highly reproducible gravimetric method for studying the uptake of water vapor by stratum corneum samples from atmospheres of controlled humidity and temperature has recently been developed (13). By studying hygroscopicity with this device, stratum corneum specimens prepared by several of the more commonly used methods described above have been compared. Anderson, Cassidy, Hansen et al. have recently shown that in vivo evaluation of dry skin correlates with in vitro hygroscopicity (14). Thus hygroscopicity seems a reasonable way to assess one factor in the normalcy of stratum corneum samples.

MATERIALS AND METHODS

Abdominal skin from autopsy cases was collected. The specimens were refrigerated, but never frozen, until separation procedures could be effected. Each piece of skin was divided into four pieces. The first piece was separated by heat at 56°C for 3 min in a water bath. The second was separated as the first, then placed in 3% trypsin for 24 hr. The third was floated, epidermis down, in a 3% trypsin solution for 24 hr. (The 3% concentration was chosen to maximize any trypsin related effect.) And the fourth was removed by the ammonia fume method. All specimens were stored over a disiccant until ready for use. A 4-mm punch was taken from each specimen for comparison. The weight of each 4-mm piece was taken in a special chamber containing a Cahn electrobalance, controlled temperature and relative humidity regulated by saturated salt solutions (13). The per cent increased over dry weight of each sample was noted at 62 and 90% relative humidity and 20°C.

Cantharidin blisters were prepared by placing a 2-cm by 2-cm piece of moistened filter paper containing 0.5 mL of a 0.2% cantharidin solution on the backs of volunteers for 3–4 hr. The filter paper was covered sequentially with Saran wrap®, Mystic tape®, Reston foam® and finally Micropore tape®. After removing the occlusive dressing, the blister was allowed to fill overnight before removal of the top. Blister tops were stored over a desiccant and measurements were taken as above.

RESULTS

Separation techniques proved to be more variable than reported. Ammonia fumes did not always give good separation of dermis from epidermis. Trypsin was somewhat

Table I
Percent Increase Over Dry Weight at 62% Relative Humidity

Subject	Separation Method				Average for subject
	Heat	Heat + Trypsin	Trypsin	Ammonia	
1	5.4	5.4	4.8	4.95	5.1
2	4.6	2.3	4.2	6.9	4.5
3	8.1	10.0	8.1	8.3	8.6
Average for separation method	6.0	5.9	5.7	6.7	

more consistent. Heat alone or combined with trypsin always worked smoothly as did cantharidin. Results reported here represent only those specimens in which all techniques were successful (three of five).

The data for the hygroscopicity of samples is presented in Tables 1, 2 and 3. Two-way analysis of varience fails to show a significant difference attributable to harvest methods at either 62 or 90% relative humidity (R.H.). However at 62% R.H. a significant difference was detected among individuals not found at 90% R.H. ($F = 10.06$; p 0.05).

DISCUSSION

The data for cantharidin appears clearly superior to that for any other method. At 62% R.H., cantharidin averaged 12% increase over dry weight; with ammonia, the next highest was 6.7%. At 90% R.H., the figures are 35.5% for cantharidin and next highest was heat plus trypsin at 28.5%. However these figures are not directly comparable. The skin of the autopsy cases came from the abdomens of a 70-year-old female, a 72-year-old male, and another 70-year-old female. The cantharidin blisters were made on the backs of healthy males in their 20's. The differences could be attributged to 1) age, 2) cantharidin or 3) back/abdominal differences.

Table II
Percent Increase Over Dry Weight at 90% Relative Humidity

Subject	Separation Method				Average for subject
	Heat	Heat + Trypsin	Trypsin	Ammonia	
1	17.5	21.9	18.0	27.3	21.2
2	21.9	28.5	26.0	25.2	25.4
3	18.0	35.0	23.6	20.4	24.2
Average for separation method	19.1	28.5	22.5	24.3	

Table III
Percent Increase Over Dry Weight at 62% and 90% Relative Humidity by Cantharidin Blister Tops

Subject	62%	90%
1	12.8	36.3
2	10.9	41.3
3	11.5	36.2
4	12.1	31.3
5	12.5	32.6
Average	12.0	35.5

Middleton worked with guinea pig foot pads and found a 28.2% increase over dry weight at 90% R.H. at room temperature (4). Fox et al. worked with pulverized, unwashed human callus and found an 11% increase over dry weight at 60% R.H. and 34% increase at 90% R.H. at 23°C (15).

These values are similar to our cantharidin data. For comparison, Table 4 presents values that other workers have reported with various methods. One might draw the conclusion that callus and stratum corneum obtained from the back by cantharidin are analogous in their physical chemical properties; however, Blank and Shappirio pointed out that water extraction of callus for 2 hr did not alter water holding capacity, but did decrease flexibility (16).

Different stratum corneum harvest methods may lead to differences in hygroscopicity, but the differences cannot be demonstrated to be statistically significant. Yet, a

Table IV
Comparison of Stratum Corneum Hygroscopicity

Investigator	Source of Corneum	Temp. °C	% Relative Humidity	% Increase Over Dry Weight
Singer & Vinson (10)	Neonatal rat	25	57	11
			71	16
			81	23
			93	60
Middleton & Allen (17)	Guinea pig footpad	4	86	26.1
		22	81	20.6
Middleton (4)	Guinea pig footpad	21–24	90	38.2
Flesch & Esoda (18)	Pulverized, ether defatted, nonhydrolysed callus &	N.R.[a]	100	28
	Psoriasis & erythroderma scales	N.R.[a]	100	19
Blank & Shappirio (16)	Callus	23	71	20
			88	38
			97	60
Laden & Spitzer (19)	Callus	N.R.[a]	37	9.2
			70	16.8
			95	78–123
Fox, Tassoff,	Callus	23	45	5
Rieger et al. (15)	Pulverized callus	23	45	6.9
			57	11.2
			90	34.0
		37	40	4.1
Anderson et al. (14)	Human calf (control)	30	95 (24 hr)	65
	(occluded)	30	95 (24 hr)	61
	(control)	30	95 (14 days)	309
	(occluded)	30	95 (14 days)	384
Buettner (20)	Tape stripped corneum	N.R.[a]	76	30
			95	110
Scheuplein (11)	Human stratum corneum	25	100	50
Present study	Human stratum corneum			
	Abdomen (heat, trypsin, ammonia)	20	62	6.1
			90	23.6
	Back (cantharidin)	20	62	12.0
			90	35.5

[a] N.R.-Not recorded.

significant variability among individuals from whom samples are taken was seen at 62% R.H. Cantharidin seems to be a superior method, but experimental design does not allow direct comparison of the data due to the different age groups and body sites. Anderson et al. found that the most hygroscopic stratum corneum samples contained the greatest amount of water soluble components (14). It seems reasonable that cantharidin would contain more of these components than other methods; the stratum corneum spends less time in water when prepared by the cantharidin technique, except for the ammonia method. These water soluble materials are also a logical mechanism for the greater hygroscopicity that the cantharidin specimens demonstrated here and make cantharidin a most attractive method for in vitro studies.

ACKNOWLEDGMENTS

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Prediction of hair assembly characteristics from single fiber properties

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Synopsis

An hypothesis is developed with relationships that PREDICT how changes in the behavior of HAIR ASSEMBLIES (tresses or heads) depend on changes in SINGLE FIBER PROPERTIES that are measurable, i.e., how changes in combing ease, flyaway, body, managability and style retention of hair assemblies relate to changes in fiber friction, stiffness, static charge, curvature, weight and diameter. From these relationships desired changes in assembly characteristics may be approached through changes in the fiber properties.

INTRODUCTION

A recent stimulating publication by Hough, Huey and Tolgyesi (1) reminded us of an internal report that we had written several years ago defining an approach for predicting characteristics of hair assemblies (tresses or hair on heads), using single fiber properties. Although the paper by Hough et al. is concerned only with hair body, our general approaches, essential definitions and conclusions are consistent. When these authors suggested "further discussions by individuals from different areas of the fiber and cosmetic fields," we decided to respond by putting our report into a publishable format.

Types of testing used to evaluate the hair effects of cosmetic products are: measurement of physical properties of single fibers; evaluation of fiber assemblies on tresses or on heads; and perceptions of how products perform in blind, identified and sales testing.

This manuscript is concerned only with the first two types of assessments, i.e., with relating measurable changes in single-fiber properties to the evaluation of changes in characteristics of fiber assemblies, where psychological perceptions from advertising, labelling and product properties such as fragrance, lather feel, viscosities, etc., are eliminated.

DISCUSSION

The consumer's vocabulary for describing the behavior of hair includes a number of assessment terms, such as combing ease, style retention, flyaway, body, raspiness,

Table I
Factors Which Can Influence Primary Single Fiber Properties

	Other Single Fiber Properties										Treatment Effects					
Primary Single Fiber Property	Hygroscopicity	Conductivity	Polarity	Shape (cross-sectional)	Tensile elastic yield and break	Oiliness	Sorptivity	Cohesion	Surface Roughness	Water set	Surfactants	Dye	Bleach	Permanent Wave	Hair Dressings	Hair Sprays
Static Friction (F_s)	x				x (2)	x	x	x (3)	x		x	x	x	x	x	x
Kinetic Friction (F_k)	x				x (2)	x	x		x		x	x	x	x	x	x
Stiffness (S)	x			x	x							x (1)	x (1)	x (1)		
Static Charge (E)	x	x	x			x	x				x	x	x	x	x	
Hair Fiber Curvature (C)				x	x					x				x		
Weight (W)	x											x	x	x	x	x
Diameter (4) (D)	x											x	x	x		
Luster (I)						x	x		x		x	x	x	x	x	x
Color (H)												x	x			

(1) Only excessive treatment, and wet properties affected first.
(2) Elastic and yield properties influence friction. (16)
(3) Cohesion is considered as one part of F_s in this report.
(4) Cross-sectional areas.

manageability, luster and feel or handle. Most of these terms are extremely complex to analyze since generally each depends of several physical properties of a fiber assembly which are interrelated. For example, stiffness on a fiber assembly includes contributions from the number of entanglements, and from kinetic and static friction as well as from the actual stiffness of the fibers. This principle holds for most properties of fiber assemblies. By contrast, certain "primary" properties of single fibers are isolatable, i.e., can be measured independently of each other, and they directly influence the properties of an assembly of hair.

Thus it occurred to us that most of the complex terms that are used by consumers for describing hair behavior might be approximated by algebraic expressions combining single fiber properties, such as: static friction (F_s), kinetic friction (F_k), stiffness (S), static charge (E), curvature (C), weight (W) and diameter or cross-sectional area (D).

Certain nonchemical treatments that are applied to the fiber surface nonuniformly, e.g., hair sprays, will produce small changes in the fiber weight but not in the fiber diameter, thus we include fiber weight as a separate term. Table I lists other fiber properties or characteristics and several cosmetic treatment effects. The primary fiber properties relate to these other characteristics and to the prior history including treatments to the fibers.

In this manuscript, five assessments of a consumer's hair (combing ease, style retention,

Table II
Effect of Changes in Primary Single Fiber Properties on Changes in Assembly Properties

Increase in Subjective Hair Property	ΔF_k	ΔF_s	ΔE	ΔW	ΔD	ΔS	ΔC
+ combing	−N	−N	−N	0	+n	+n	−N
+ style retention	+n	+N	0	−n	0	−n	±N
+ flyaway	0	−n	+N	−n	0	−n	−n
± body limpness	+n	+N	0	−n	+N	+N	+N
+ manageability	−N	+N	−N	−n	0	−n	±N

F_k = Kinetic Friction
F_s = Static Friction
E = Static Charge
W = Weight of Fiber
D = Diameter (cross-sectional area)
S = Stiffness
C = Hair Fiber Curvature
± = Effect may improve or detract (see text)
N = Very important to subjective property
0 = No influence

flyaway, body and manageability) are described in terms of the primary single fiber properties (F_k, F_s, S, E, C, W and D) which could "change" in a specific treatment, Table II. Considering changes in place of absolute values permits us to neglect properties that undergo no change during treatment, e.g., density of hair population on the scalp and fiber length. We also assume no change in hair style. Furthermore, values of the primary properties are representative of the "average hair fiber" of the assembly under consideration.

For simplification, it is assumed that cohesion is a part of static friction. If this simplification proves unsatisfactory, then separate terms will have to be used. Hair fiber curvature is proportional to the number of crooks and bends of the fiber and may be estimated by the decrease in length and it may be neglected when considering changes produced by products other than permanent waves, straighteners or setting aids. For example, an initial state of the assembly (K) is described in (eq 1) as a function of the appropriate primary properties which may undergo change in a specific treatment on a specific head.

$$K \simeq f(F_s, F_k, S, E) \quad (1)$$

A change in the assembly property is defined in (eq 2) as a linear combination of changes in the single fiber properties. Changes in single fiber properties are considered as "after treatment" minus "before treatment," e.g., $\Delta S = S_{\text{After Treatment}} - S_{\text{Before Treatment}}$, which results in a positive change in the assembly property as indicating an increase by treatment.

$$+\Delta K \simeq N_1 \Delta F_k + n_1 \Delta F_s - N_2 \Delta S - n_2 \Delta E \quad (2)$$

Multiplying constants that are capitalized (N) indicate that specific primary property to be of greater importance, in our judgment, than those preceded by small letter multiplying factors (n).

Initial uses of the system are anticipated to be directional, providing approaches for

maximizing certain assembly properties while minimizing losses in others. The system at present is more useful in planning stages of products. With adequate test methods for measuring the required primary fiber properties and refinement of equations, the analysis should become more quantitative and useful for development of hair products.

COMBING EASE

Combing ease may be defined as the ease of aligning fibers of an assembly with a comb so they are essentially parallel. This process is considered in our beauty salon evaluations in two stages: the first is snag (entanglement) removal and the second is comb slip.

Equation 3 states that six primary single fiber properties, measured under the conditions for combing, describe the ease of combing for any assembly of hair. Many of the factors which control or influence these six primary single fiber properties and their interrelations are described in Table I.

$$\text{Combing ease} = f(F_s, F_k, S, E, C, D) \quad (3)$$

If one considers any particular consumer's hair which has been described by K combing ease, then the change in combing ease produced by treatment of this hair is represented by (eq 4.)

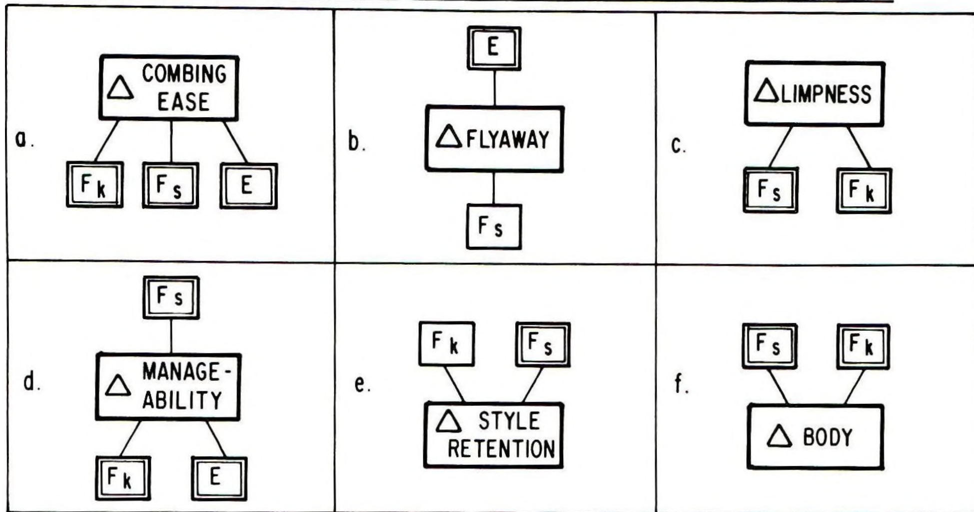


Figure 1.

$$\Delta \text{Combing ease} = -N_1\Delta F_k - N_2\Delta F_s - N_3\Delta E + n_1\Delta S + n_2\Delta D - N_4\Delta C \quad (4)$$

Thus Δ combing ease describes the most recent treatment effects on the hair and may be defined in terms of changes in the primary single fiber properties. For most shampoos and creme rinses, in which the active ingredients do not affect the fiber curvature, diameter, or stiffness and the styling is not changed as a part of the treatment, Δ combing ease is defined by (eq 5), illustrated in schematic form in Figure 1.

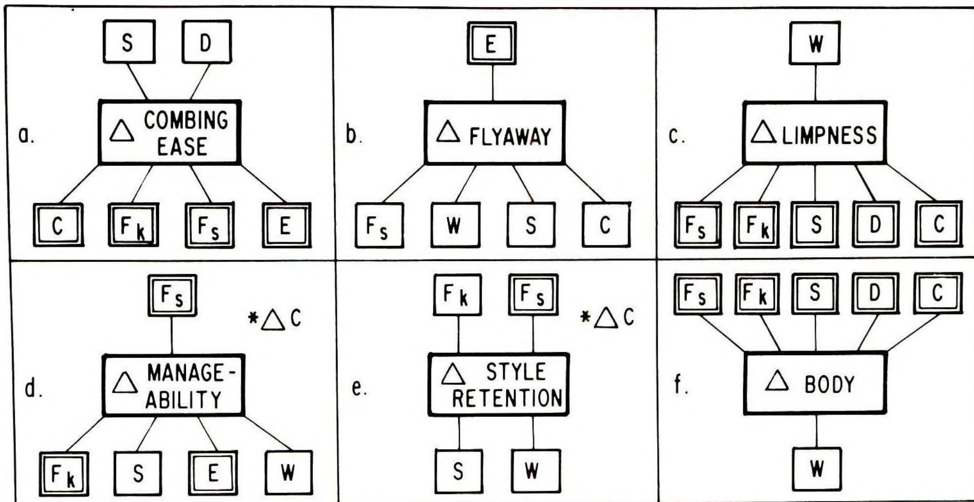
$$\Delta \text{Combing ease} = -N_1\Delta F_k - N_2\Delta F_s - N_3\Delta E \quad (5)$$

Equation 5 suggests that combing ease is influenced largely by changes in three primary properties F_k, F_s and E. Furthermore, increases in either F_k, F_s or E lead to poorer combing (note negative signs).

More complex hair treatments such as permanent waves can produce changes in hair fiber curvature and stiffness, while polymer depositions or graftings can produce changes in hair fiber mass and/or diameter. Equation 4 considers the effects of changes in these primary properties on combing ease, see the schematic in Figure 2a.

An increase in fiber curvature will increase the number of possible entanglements in the hair and have a relatively large effect on combing ease. On the other hand, increases in stiffness and diameter make hair easier to comb. The frictional forces produced by combing an assembly of hair should be independent of fiber diameter yet proportional to

CHANGES PRODUCED BY OTHER HAIR PRODUCTS



1. [box] = n = SMALL MULTIPLYING FACTOR OR OF LESSER IMPORTANCE TO SUBJECTIVE PROPERTY.
2. [double box] = N = LARGE MULTIPLYING FACTOR.
3. WHERE PRIMARY PROPERTIES ARE DRAWN ABOVE SUBJECTIVE PROPERTIES, AN INCREASE IN THE PRIMARY PROPERTY INCREASES THAT SUBJECTIVE PROPERTY, WHILE THE REVERSE IS TRUE FOR THOSE PRIMARY PROPERTIES DRAWN BELOW THE SUBJECTIVE PROPERTIES.

*ΔC EFFECT IS NOT STRAIGHTFORWARD (SEE TEXT).

Figure 2.

the number of fibers in contact with the comb. If one assumes the same total volume of fiber assembly is combed in each stroke, then the larger the individual fiber's volume, the fewer fibers in contact with the comb surface. Therefore an increase in fiber diameter should produce a small positive increase in combing ease.

Measurement of primary properties at or near the same humidity for combing evaluations is necessary. With this in mind, (eq 3–5) hold for both wet and dry combing.

Wet combing is a relatively well defined condition, while dry combing can involve any condition from zero to near 100% R.H. For wet combing, E and ΔE approach zero and can be neglected in (eq 4 and 5).

This suggests that for wet combing, where there is no change in the hair fiber curvature, F_k and F_s are the important factors, since S and D play less important roles in both wet and dry combing.

At low humidities, static charge becomes relatively important to combing ease. As the humidity is increased, the moisture content of hair also increases and its ability to acquire an electric charge decreases, i.e., ΔE decreases, rapidly becoming of less importance. At high RH, swelling of the fibers becomes greater, (ΔD increases) the fibers become less stiff and friction increases. Although ΔE approaches zero at high humidities and ΔD increases, making hair easier to comb, the other three factors F_s, F_k and S predominate making hair more difficult to comb.

FLYAWAY

Flyaway is the condition, during combing, in which hair fibers of an assembly separate due to repulsive forces of electric charge. We can consider two steps in flyaway: those factors which lead to charge build-up on the hair and the flyaway state itself.

Those factors which lead to charge build-up during combing are those that increase the work of combing, which are described in the previous section by the combing ease equation 4, and the inherent ability of the fibers to acquire a charge.

The flyaway state itself is influenced by five primary single fiber properties, see eq 6 and Table II.

$$\text{Flyaway} = f(E, F_s, S, C, W) \tag{6}$$

For hair products such as shampoos, hair sprays, creme rinses, groomers, etc., in which the active ingredients do not affect the hair fiber curvature and the hair styling is not changed as a part of the treatment, Δflyaway may be defined by (eq 7), illustrated schematically in Figure 2b.

$$\Delta\text{Flyaway} = + N_1\Delta E - n_1\Delta F_s - n_2\Delta W - n_3\Delta S \tag{7}$$

As suggested earlier, measurement of these primary properties is necessary at or near the humidity at which the flyaway is observed.

Where there is no change in the hair fiber curvature, and at low humidities, flyaway is influenced principally by changes in E, (eq 7), and the greater the charge build-up on the fibers, the greater the flyaway. Opposing the extent to which the fibers may separate (flyaway) under a given electric charge, are S, W and F_s. This latter term is considered to include the cohesive forces that exist between the fibers.

As ambient conditions change and the humidity increases, the moisture content and ΔW increase and the fibers' ability to acquire an electric charge decreases, i.e., ΔE and flyaway decrease rapidly.

The greater the fiber curvature, the greater the chance for fiber entanglements as the fibers separate due to electrical forces. Therefore the greater the fiber curvature, the less flyaway. Hair fiber curvature also plays a key role in the preflyaway condition, while the fibers are becoming charged. The greater the fiber curvature, the greater is the work of combing—one key factor which promotes the electric charge build-up on the fibers. Since the charging effect is opposite to that which exists at the charged state, it may "appear" as if hair fiber curvature enhances flyaway. Similarly, kinetic friction involved in combing or brushing of the fiber assembly influences the work of combing which in turn influences the amount of charge build-up and ultimately the extent of flyaway.

BODY

Body is defined in the textile trade as the compact, soft or firm feel of textile stock or fabric (2), a tactile property. In our beauty salon body is evaluated as apparent thickness or volume of the assembly, involving sight and touch for assessment. Both descriptions are consistent with the quality of liveliness or springiness (3) that is often associated with body. Our beauty salon generally evaluates body immediately after setting and drying, but evaluation over longer time periods is also relevant.

An "increase in body" is not necessarily an improvement in hair behavior: hair with too much body may be unmanageable; the stylist should be able to make the hair appear thick only where she wants it to appear thick. Also hair can have body and yet have poor style retention, e.g. the hair may not hold a style well, yet will appear thick in the relaxed state.

Equation 8 states that six primary single fiber properties measured under the conditions for evaluation of body, describe this parameter for an assembly of hair.

$$\text{Body} \simeq f(F_s, F_k, S, C, D, W) \tag{8}$$

For those shampoos and creme rinses which do not generally affect the fiber curvature, body may be described by (eq 9). This equation appears in schematic form in Figure 2.

$$\Delta\text{Body} = + N_1\Delta F_s + N_2\Delta F_k + N_3\Delta S + N_4\Delta D - n_1\Delta W \tag{9}$$

An assumption inherent in this equation is that the hair styling is unchanged to the extent that fiber contacts are unchanged. In fact, for maximum body, fiber contact should be at a minimum.

In effect, (eq 9) suggests that an increase in body will result by simply making the total hair assembly stiffer. This suggests that limpness is not enough body produced by making the entire fiber assembly less stiff, i.e., $+\Delta\text{Body} = -\Delta\text{Limpness}$.

An increase in body according to (eq 9) is not time dependent and is consistent with our beauty salon evaluation. For treatments such as permanent waves, whose active ingredients affect the hair fiber curvature, increasing fiber curvature should increase body.

MANAGEABILITY

In cosmetic terms, manageability is concerned with the ease of arranging hair in place and its temporary ability to stay in place. This property is evaluated in our beauty salon immediately after setting and drying and is not concerned with longer term effects on the hair fiber assembly.

Equation 10 states that six primary single fiber properties measured under the conditions for manageability evaluation determine this parameter (K manageability) for an assembly of hair, see Table I. For hair products

$$\text{Manageability} \simeq f(F_s, F_k, S, E, C, W) \tag{10}$$

such as those shampoos, hair sprays and creme rinses whose active ingredients do not affect the hair fiber curvature and for unaltered styling, change in manageability may be defined by (eq 11). This equation is illustrated in schematic form in Figure 2d.

$$\Delta\text{Manageability} = -N_1\Delta F_k + N_2\Delta F_s - n_1\Delta S - N_3\Delta E - n_2\Delta W \tag{11}$$

Equation 11 suggests that manageability will be increased by decreasing F_k, E, S and W. Stiffer fibers are less flexible and are therefore less manageable. However fiber stiffness is less important than E and F_k. The effects of humidity on static charge and flyaway have already been described and an increase in W may decrease the "staying in place" part of manageability to a small extent.

The opposite signs in the two friction terms suggest that maximizing the force required to initiate movement between surfaces (to transiently hold style in place) and minimizing the force to maintain movement of surfaces past one another will increase manageability. A certain minimum amount of F_k helps to maintain a harmonious assembly pattern in styling movements. If F_k is high before treatment, then our friction analysis in manageability is almost certainly correct; as F_k approaches the minimum required value, however, modification to (eq 11) may be required.

If the fiber curvature is altered in any way such as a change in set or the ability of the fibers to accept a water set, it will be reflected in the manageability of the hair, i.e., if the treatment changes the fiber curvature so that it is either too straight or too kinky, for the particular style desired, it will be less manageable.

STYLE RETENTION

Style retention may be defined as the capability of hair to stay in place after styling. Since style retention is time dependent, evaluation should involve a time period of several hours to a few days as compared to evaluation immediately after setting. Style retention includes curl or wave retention.

Equation 12 states that five primary single fiber properties measured under the conditions for style retention evaluation, describe style retention for any assembly of hair fibers, see Table II.

$$\text{Style retention} = f(F_s, F_k, S, C, W) \tag{12}$$

For hair products such as those shampoos, hair sprays, creme rinses, groomers, etc. in which the active ingredients do not affect the hair fiber curvature and the hair styling is not changed as a part of the treatment, style retention may be defined as in (eq 13). See the

schematic illustration of this equation in Figure 2e. Equation 13 suggests that style retention may be increased by increasing F_s and F_k, with F_s being the more important factor.

$$\Delta \text{Style retention} = + N_1 \Delta F_s + n_1 \Delta F_k - n_2 \Delta S - n_3 \Delta W \qquad (13)$$

Increases in S and W can decrease style retention. The role of W is rather straightforward, while that of S is more subtle. Since style retention is time dependent as contrasted to the other assembly properties defined in this report, the natural changes that occur to hair fiber curvature—from immediately after water setting and styling until equilibrium is reached—are of extreme importance. The fibers on a head are generally water set to produce a desired contour. Changes in humidity promote a deterioration of the water set and a change in the hair fiber curvature. Therefore hair fibers that have been water set, styled and exposed to ambient conditions do not have the same curvature stresses as after setting and styling. Frictional forces primarily tend to hold the assembly in the "set" style. Thus these natural curvature changes induced by water vapor absorption produce transient stresses within each fiber assembly. These stresses, which are dependent on the amount of curvature change and fiber stiffness, tend to decrease the style retention.

In the hypothetical situation that assumes no changes in fiber curvature due to humidity, the maximum fiber curvature consistent with the desired hair styling will produce the maximum number of possible entanglements and therefore the maximum style retention.

For treatments such as permanent waves in which the active ingredients produce changes in the "relaxed" hair fiber curvature or for polymer depositions or graftings, which may also change the hygroscopicity of the hair, the rate and extent of change in hair fiber curvature in response to humidity is altered.

METHODS FOR EVALUATING PHYSICAL PROPERTIES

Both single fiber and fiber assembly methods are available for most of the properties described in this report. Single fiber methods have been described for friction (5), stiffness (6,7), static charge (8), and fiber diameter (3, 9, 10). Fiber curvature may be estimated from the fibers relaxed length and its taut length. Fiber assembly methods have been described for combing ease (11, 12), flyaway (13, 14, 15), body (3) and percentage of set retention (3). Manageability is the most complex of these assembly properties and a quantitative method for this property has not been described.

CONCLUSIONS

Changes in the behavior of hair assemblies can be usefully represented in algebraic form as combinations of changes in single fiber properties. Considering directional changes instead of absolute values leads to useful simplifications. A summary of how the assembly behaves as a function of changes in the single fiber properties follows.

1. **Combing** changes depend primarily on frictional effects (including cohesive forces), static charge and fiber curvature. For most products other than permanent waves and straighteners, changes in fiber curvature are negligible and for wet combing, static charge is not relevant.

2. For **flyaway**, static charge is the determining factor.
3. **Body** depends on static friction, fiber diameter, fiber stiffness and curvature, while limpness is too little body. For most products, except permanent waves and straighteners, changes in fiber curvature are not relevant and changes in fiber diameter and stiffness are negligible.
4. **Manageability** depends on frictional effects, static charge and fiber curvature. For most products except permanent waves and straighteners, fiber curvature is unchanged, leaving static friction, kinetic friction and static charge as the determining factors. Increasing static friction makes hair more manageable, while increasing kinetic friction makes it less manageable.
5. For **style retention**, static friction (including cohesive forces) and fiber curvature are the determining factors and for most products, except permanent waves and straighteners, static friction is the determining factor.

Equations as depicted in this manuscript can provide guidance for developing and documenting different hair products. Improvement in single fiber methods should permit the equations to approach more quantitative forms.

With Hough et al. (1), we recommend further discussions and definitions of important cosmetic terms.

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Book Reviews

RECENT ADVANCES IN DERMATO-PHARMACOLOGY, Edited by Phillip Frost, M.D., Edward C. Gomez, M.D., Ph.D., and Nardo Zaias, M.D., Spectrum Publications, Inc., New York, 1978, 259 pages. Price $17.50.

The book compiled presentations that were made at a meeting held at Mt. Sinai Medical Center of Greater Miami in August of 1975. As such, some of the information is not "recent," nor are some of the chapters. However the book will provide a review for the practicing dermatologist about some recent breakthroughs in the areas of dermatology therapeutics; the researcher will have known about these advances some time ago.

Among the materials presented that are of recent nature are: the immunological transfer factor involved in candidrasis and other infectious diseases; the management of pemphigus with gold compound, as well as the use of thalidomide for polymorphous light-like eruptions in American Indians.

Chapters on evaluating new potent topical steroids, as well as the adrenal effect of the same, should round out and update the practicing dermatologist in the area of steroid potency. Discussion on topical nonsteroidal antiinflammatory agents is quite limited as dictated by the limited knowledge in this field of the usefulness of these agents in diseased conditions. Chapters on mycophenolic acid, puva therapy, coal tar gel therapy and management of hyperkeratosis with alphahydroxy acid and salicylic acid may be new to the practicing dermatologist who has not kept up with the Journals, but does provide a repository of information which has appeared in Journals. The information provided in these subjects cannot be considered of recent vintage, but is informative in updating the practicing practicioner.

The chapters on Delivery System, bioavailability of griseofulvin in the recent ultra microsize form, are somewhat outdated and could have been enhanced by the review of topical griseofulvin therapy and its potential. Similarly information on new antifungal agents of the imadazole variety are limited in value since they do not provide comparisons with other known antifungal agents which have been on the market for a longer period of time.

The chapters on cortical steroid/antibiotic combination pros and cons make for interesting reading and the problem is still being debated by the FDA and Antimicrobial II panel.

The discussion of pharmacdynamics of silver sulfurdiazine is quite old. It first appeared in the literature in the 60's and truly does not fit into the category of recent advances, although we may better understand the mechanism of action of these agents now than we did a decade ago.

The last chapter on topical insect repellents has very limited application to a dermatologist, but may be of research interest.

The book is simple to read but does not get into basic science research questions involved in mechanistic approach to some of the recent drugs, such as nonsteroidal antiinflammatory agents, mycophenolic acid. It, does, however, provide the reader with an update and a valuable index that he can utilize in his office practice. Literature references are somewhat limited and, indeed, the most pertinent literature references have been omitted knowingly or unknowingly. If they were included, it would make the volume more valuable for purposes of archivalization. The book may be useful for the practicing physician, pharmacist and related paramedic personnel, but of limited usefulness to researchers and students.—O. J. LORENZETTI, Ph.D.—Alcon Laboratories, Inc., Fort Worth, Texas.

POLLUTION EVALUATION, Environmental Science and Technology Series, Volume 2, William F. Pickering, Marcel Dekker, Inc., New York, 1977, 199 pages. Price $16.50

The author has covered an often emotional issue with the hand of a scientist and his publication departs from the recent offering in the pollution and environmental science fields in several areas.

It is written for the general scientific community rather than a specific discipline. The book is arranged for a dual purpose. The odd chapters discuss modes of evaluating some typical forms of pollution (atmospheric, water, soil, food), along with a generic approach, rather than a discussion of specific methodologies for individual pollutants. The even chapters cover the analytical methods currently used in the field of environmental analysis. The coverage of the principles of gravimetry, titrimetry, etc., are brief by design; nevertheless, the reader can understand the problems facing the analytical chemist trying to measure PPB of a pollutant.

The chapter on water quality should prove useful to the cosmetic chemist, as it outlines some of the criteria used in determining water quality. Specific analytical methods are discussed for some of the more important pollutants found in industrial waste water.

Ample literature references appear throughout the book; most of them are three to five years old, which is unfortunate, since much has been published in this field.

This volume will serve both the occasional reader and those actively involved in the field and is recommended for both individual and company libraries.—GERELD S. ROYE—Chesebrough-Pond's, Inc.

Journal of the Society of Cosmetic Chemists

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   (1) L. E. Gaul and G. B. Underwood, Relation of dew point and barometric pressure to chapping of normal skin, *J. Invest. Dermatol.*, **19**, 9-19 (July 1952).

   References to books are handled similarly and should include pertinent page numbers:

   (2) S. Rothman, "Physiology and Biochemistry of the Skin," The University of Chicago Press: Chicago, Ill., 1954; pp 494-560.

   References to books containing contributions from authors appear as follows:

   (4) S. D. Gershon, M. A. Goldberg and M. M. Rieger, Permanent waving, in "Cosmetics, Science and Technology," 2nd ed., M. S. Balsam and E. Sagarin, Eds., Wiley-Interscience: New York, 1972; Vol. 2, pp 167-250.
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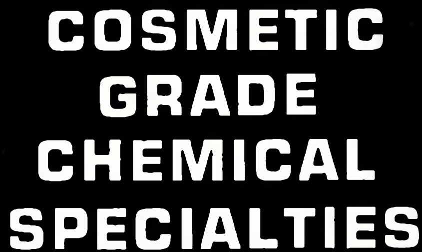

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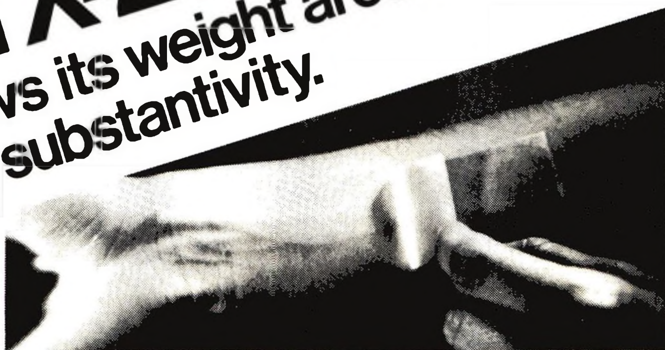

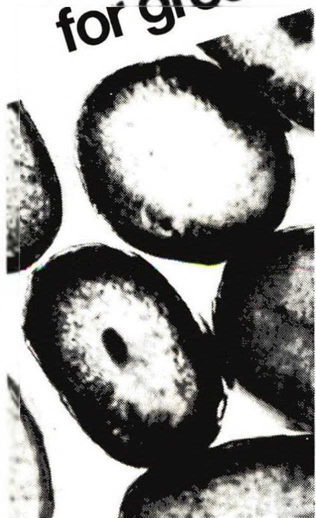

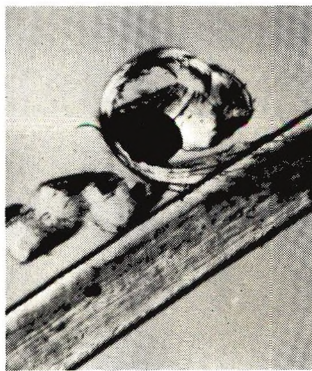

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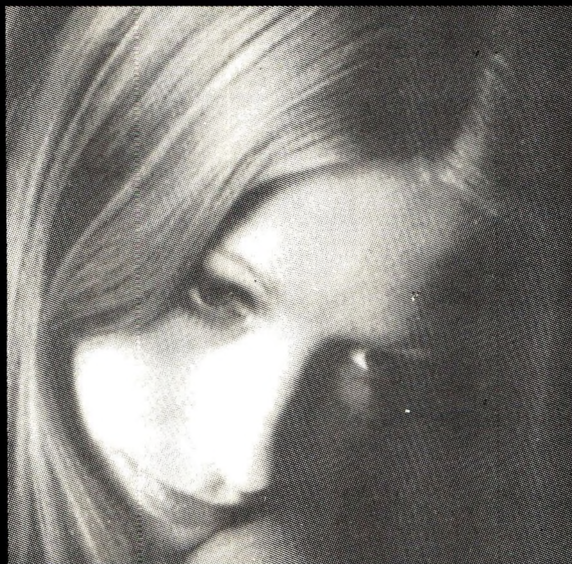

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