Vol. 26 No. 3 March 1975

Journal of the Society of Cosmetic Chemists

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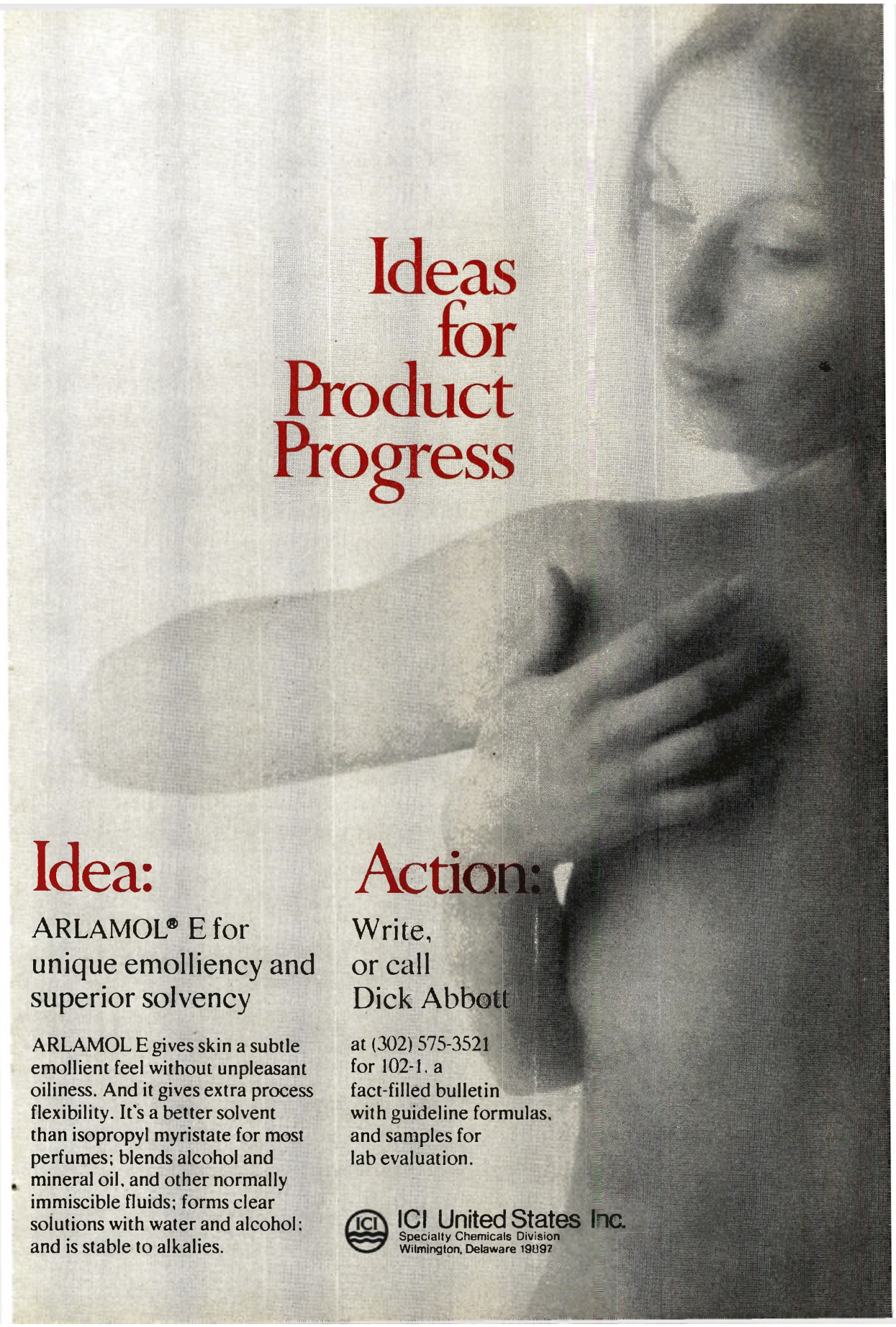

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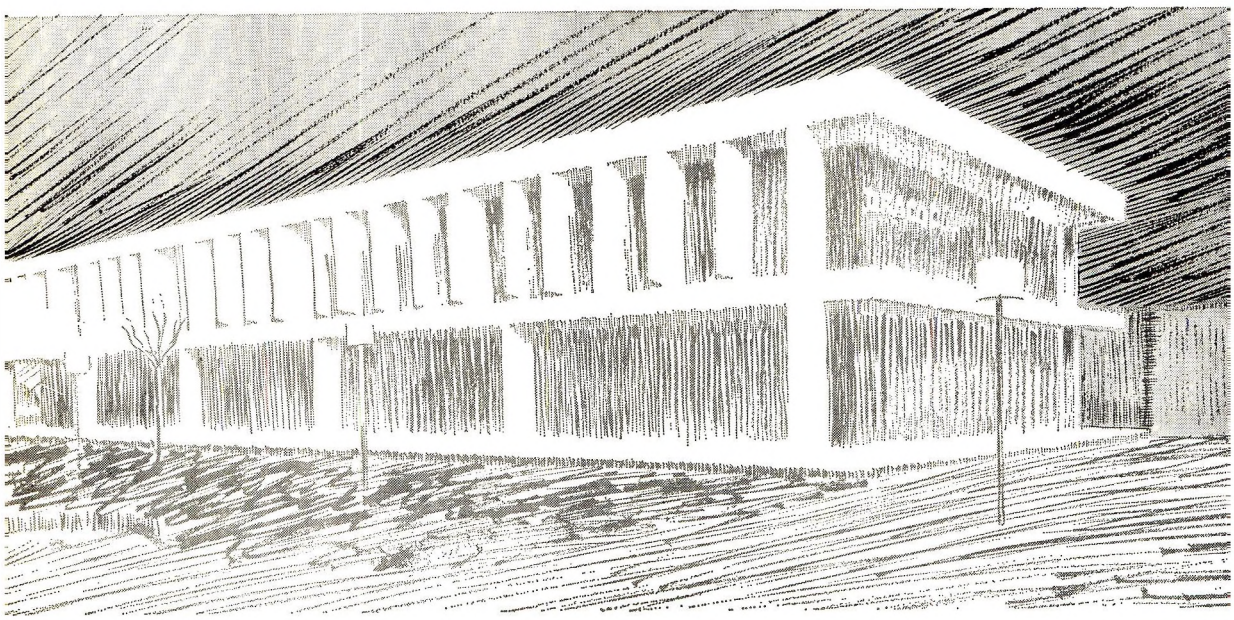

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SYNOPSES FOR CARD INDEXES

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

Effects of phase inversion and surfactant location on the formation of O/W emulsions: T. J. Lin, Haruki Kurihara, and Hideaki Ohta. *Journal of the Society of Cosmetic Chemists* **26**, 121 (March 1975).

Synopsis—Placing of the surfactant in the oil phase prior to emulsification generally produces a finer O/W emulsion than the emulsion, with the same composition, prepared by initially placing the surfactant in the aqueous phase. To investigate the basic mechanisms responsible for the difference, a series of emulsions stabilized with various nonionic surfactants and oils were studied by using various preparative methods. Microscopic examination of the emulsion droplets and surfactant migration studies support a theory that when a hydrophilic surfactant is initially placed in the oil phase, a portion of the aqueous phase added during the emulsification process is solubilized or emulsified into the oil phase to form a W/O primary emulsion. This primary emulsion eventually inverts to form an O/W final emulsion as the surfactant migrates to the aqueous phase. A short-lived double emulsion of (W/O)/W type may be formed in the process of the phase inversion.

Methods for the determination of sebaceous matter of the skin in regard to acne: Hagen Tronnier. *Journal of the Society of Cosmetic Chemists* **26**, 141 (March 1975)

Synopsis—Problems in the determination of skin lipids are caused by its non-uniform composition, the different locale used for collection, and the questionable completeness of lipid collection. Advantages and disadvantages of the different methods—in part based on the author's experiments—are described. The three disorders of the skin lipid systems which are of concern for seborrhoea and related diseases include the amount, the composition, and the physical behavior of the lipid film on the skin's surface. Of these, the last appears to be most important. This is based on numerous investigations of acne and of seborrhoea cases. The importance of the ratio of quantity of lipid to its spreading characteristics in the formation of acne comedones is pointed out and is illustrated with investigations of skin lipids by different methods during glucocorticoid therapy. The disturbance of this ratio in acne is further illustrated with the aid of additional experimental results.

Utility of amine oxides in oil/water cosmetic systems: Burt Like, Ralph Sorrentino, and Alfonso Petrocci. *Journal of the Society of Cosmetic Chemists* **26**, 155 (March 1975)

Synopsis—The use of amine oxides as emulsifiers for oil-in-water cosmetic systems is investigated. Various typical cosmetic systems are described in which amine oxides are used in place of conventional nonionic and anionic emulsifiers. The amine oxide emulsified systems were successfully preserved from microbiological contamination by using readily available, inexpensive quaternary ammonium compounds. These preserved systems were then tested for skin and eye irritation and found to be nonirritating.

Occurrence of Pseudomonas aeruginosa and Staphylococcus aureus in the orbital area: John F. McConville and David W. Anderson, Jr. *Journal of the Society of Cosmetic Chemists* **26**, 169 (March 1975)

Synopsis—*Pseudomanas aeruginosa* and *Staphylococcus aureus* were recovered in large numbers from the orbital area of two female subjects. Further investigation of these two subjects revealed that the source of these microorganisms appeared to be an infection of of the external auditory canal. Medical diagnosis confirmed the existence of chronic ear infection in both subjects caused by *P. aeruginosa* in one case and S. *aureus* in the other.

J. Soc. Cosmet. Chem., 26, 121–139 (March 1975)

Effects of Phase Inversion and Surfactant Location on the Formation of O/W Emulsions

T. J. LIN, Ph.D.,* HARUKI KURIHARA, B. S.,† and HIDEAKI OHTA, B.S.†

Presented August 26–30, 1974, 8th IFSCC Congress, London

Synopsis—Placing of the SURFACTANT in the oil phase prior to emulsification generally produces a finer O/W EMULSION than the emulsion, with the same composition, prepared by initially placing the surfactant in the aqueous phase. To investigate the basic mechanisms responsible for the difference, a series of emulsions stabilized with various nonionic surfactants and oils were studied by using various preparative methods. Microscopic examination of the emulsion DROPLETS and surfactant MIGRATION STUDIES support a theory that when a hydrophilic surfactant is initially placed in the oil phase, a portion of the aqueous phase added during the emulsification process is solubilized or emulsified into the oil phase to form a W/O primary emulsion. This primary emulsion eventually inverts to form an O/W final emulsion as the surfactant migrates to the aqueous phase. A short-lived double emulsion of (W/O)/W type may be formed in the process of the PHASE INVERSION.

Introduction

One of the major problems constantly faced by cosmetic chemists who are in charge of manufacturing emulsion products is the maintenance of batch-to-batch consistency. Often, without apparent reasons, a batch or two may turn too thick, too thin, or unstable. Checking of the weighing errors and raw material qualities may yield no clue as to the real cause. Frequently, the source of the problem lies in the unintentionally introduced process variables which are often very difficult to pinpoint.

Although it has been known that variables such as mixing speed, temperature, method of adding one phase to the other, etc., can cause marked effects on the physical characteristics of the finished emulsions, the precise manners by which these variables influence the emulsions are not well understood.

*Consultant, 628 Enchanted Way, Pacific Palisades, Calif. 90272.
†Takasago Perfumery Co., Ltd., Tokyo Central P.O. Box 1033, Tokyo, Japan.

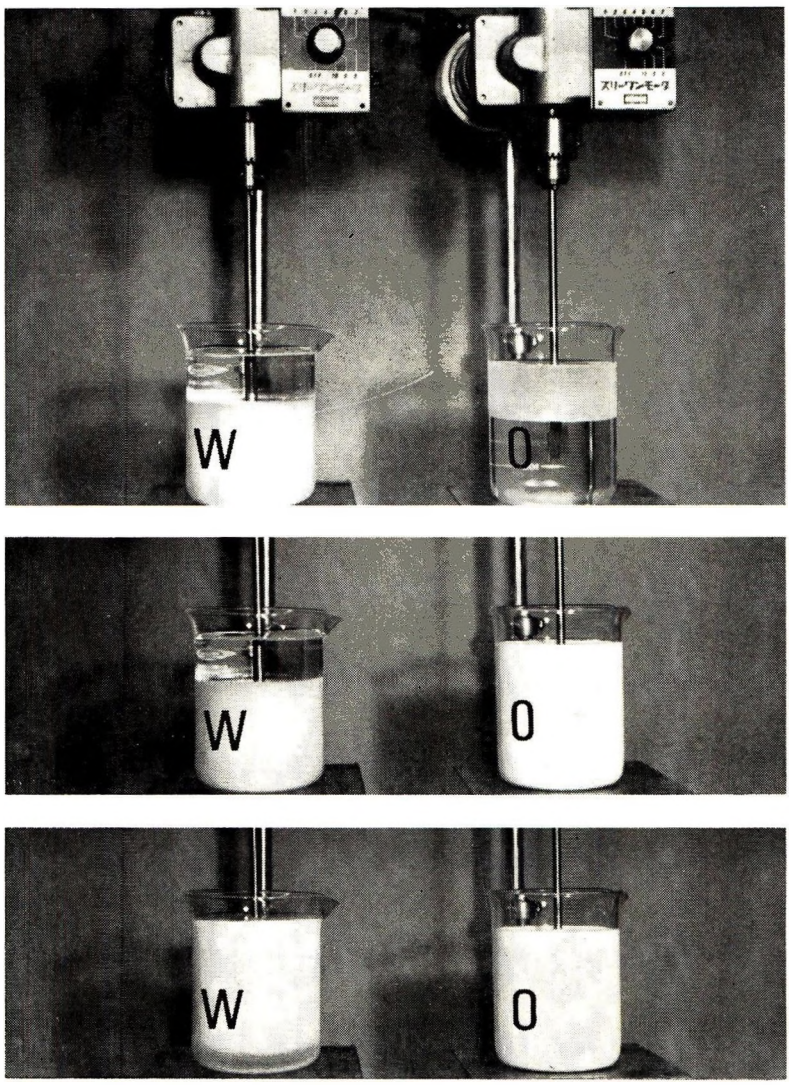

Figure 1. Emulsification with different initial surfactant location. Emulsion O (right), surfactant initially in oil phase; Emulsion W (left), surfactant initially in aqueous phase
Top. Before emulsification
Center. After 60-sec mixing
Bottom. After 2-hour mixing and ½-hour standing

From our investigation, it appears that many process variables can promote or retard the migration of the surfactant from one phase to the other during the emulsification process and consequently influence the quality of the final emulsion. In our previous works, it was reported that the initial surfactant lo-

cation could affect many emulsion properties (1–3). By using a successive centrifuge technique, we have measured the migration of the surfactant between the dispersed phase and the continuous phase, and found it to be an important factor affecting emulsification.

In particular, we have been interested in finding the reasons for the marked difference in the quality of emulsions produced by varying the initial surfactant location. To illustrate the degree of difference obtainable by simply changing the surfactant location, the following O/W emulsion was prepared with two identical beakers and mixers operated at 150 rpm.

	% by Wt.
Light mineral oil	30
Polyoxyethylene (6) oleyl ether	5
Deionized water	65
	100

In the emulsion identified as "W," surfactant was initially dispersed in the aqueous phase; whereas in the one identified as "O," the polyoxyethylene oleyl ether was initially placed in the oil phase. In Fig. 1, the first photograph was taken immediately before emulsification. The aqueous phase of the "W" shows a slight turbidity because at the concentration employed, the surfactant was not completely soluble in water. The second photograph was taken 60 sec after the emulsification and the third photograph was taken after 2 hours of continuous mixing and ½ hour standing. The emulsion prepared by initially placing the surfactant in the oil phase yielded a very good emulsion with quite uniform droplet size distribution. On the other hand, the same formulation made by initially placing the surfactant in the aqueous phase produced a very unstable emulsion with coarse droplets.

In this investigation, we were primarily concerned with the formation of O/W final emulsions. By assuming that there are two separate emulsification mechanisms in operation, we have attempted to explain this difference in terms of aqueous solubilization and phase inversion.

Theoretical Considerations

During the investigation of the effects of surfactant location, it was discovered that, in some instances, double emulsions such as the one shown in Fig. 2 would form when the surfactant was placed in the oil phase. Multiple emulsions are known to exist in many systems (4). In our investigation, however, double emulsion droplets were not observed when the surfactant was relatively hydrophilic and was placed in the aqueous phase to start. It was speculated that when the surfactant was first placed in the oil phase, the formation of the double emulsion might in some way be related to the mechanism of emulsification.

As illustrated in Fig. 3, when water is mixed with an oil phase containing a relatively hydrophilic surfactant, a portion of the water can be solubilized by

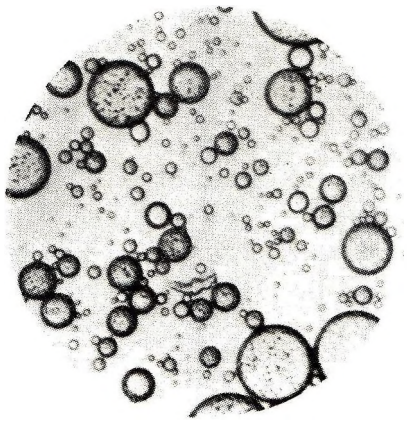

Figure 2. Microphotograph of a (W/O)/W type double emulsion

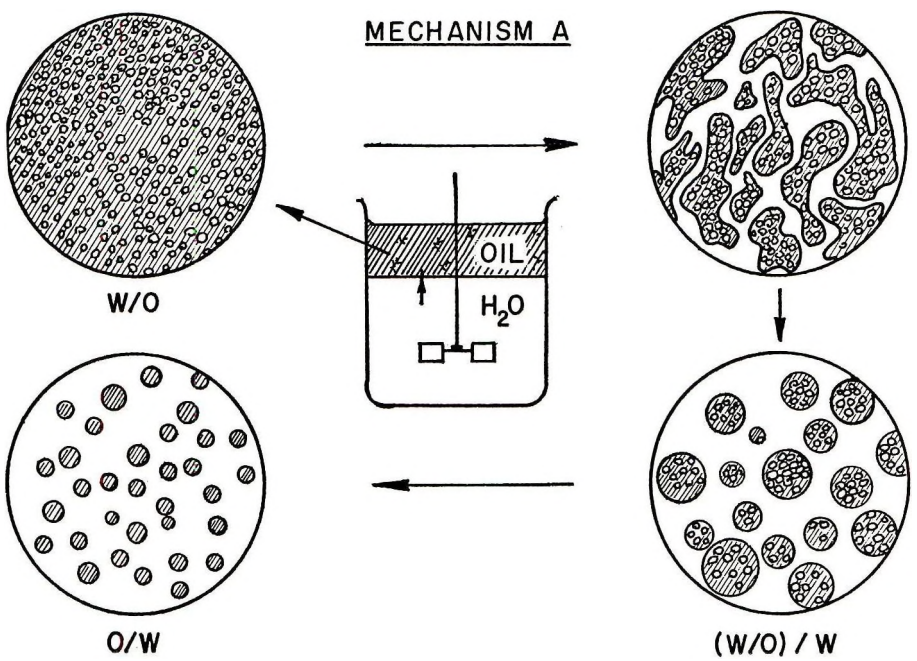

Figure 3. Illustration of Mechanism A with surfactant initially in the oil phase

the surfactant micelles or emulsified by the oil phase to form a W/O primary emulsion. If the formulation is such to favor an O/W final emulsion, the initial W/O emulsion will not be stable. With continued mixing, this primary W/O emulsion is mixed into the excess water and may form a (W/O)/W type double emulsion as illustrated. As the surfactant migrates to the outer, aqueous phase, the unstable, larger globules are readily broken into small ones and the final emulsion may be a simple O/W emulsion. For the sake of discussion, the proposed mechanism will be referred to as Mechanism A.

When the surfactant is initially placed in the aqueous phase, Mechanism A is inoperative. Instead, as illustrated in Fig. 4, if a sufficient mixing action is provided, the oil is mixed into the aqueous phase. The globules become progressively smaller as the mixing continues and the final size is very much dependent on the intensity of the mixing. This mechanism is referred to as Mechanism B.

Therefore, the major difference in these two mechanisms is that in Mechanism A, there is an inversion from W/O to O/W which may involve a temporary formation of the (W/O)/W type double emulsion. The inversion may involve the entire emulsion at once or may be a localized process at a given instant. The double emulsion is not always noticeable since the transi-

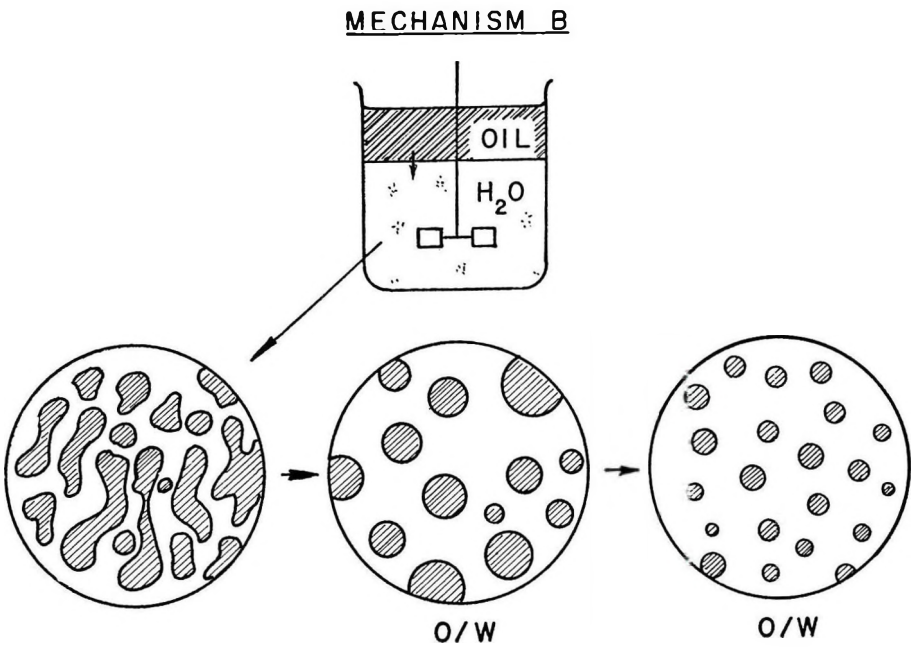

Figure 4. Illustration of Mechanism B with surfactant initially in the aqueous phase

tion may be extremely quick; also, in some instances, this primary W/O emulsion may be a microemulsion not observable under an ordinary optical microscope. The important point is that as a result of the phase inversion, the droplets are broken easily with minimum mechanical agitation. On the other hand, the breaking of the droplet in Mechanism B is entirely dependent on mechanical shear. Unless a high mixing speed is employed or a larger quantity of the emulsifier is used, the droplets of this final O/W emulsion will not generally be very small.

Based on the postulated Mechanism A, it would follow that the following three conditions are required for this mechanism to be operative:

1. The surfactant must be soluble in the oil phase and initially placed in the oil phase.
2. The surfactant in the oil phase must solubilize or emulsify a part of the aqueous phase.
3. A phase inversion must take place to form an O/W final emulsion.

By carefully selecting different surfactants, oils, and process conditions, investigations were carried out to determine whether or not one could produce a finer emulsion by meeting these conditions, than by not meeting them. In all the experimental work, the mixing speed was deliberately kept low to minimize the effect of Mechanism B.

Experimental

For emulsification, experiments were carried out in glass beakers using a 2 x 6-cm flat paddle mixer set 1 mm above the bottom of the beaker. The aqueous phase was first placed in the beaker and the oil phase was carefully placed on the top of it before the mixer was turned on to start emulsification. The mixer speeds were carefully calibrated with a tachometer to ensure correct speed setting before each experiment. For emulsification, the speed was set at 150 rpm. The measurements of surfactant migration were carried out by using the method previously described (3). A colorimetric method was used to analyze the surfactant concentration (5). For the measurement of phase inversion temperature, we used both a conductivity meter* and a torque meter.† A photograph of the torque meter attached to a mixer shaft is shown in Fig. 5. For solubilizing water into the oil phase prior to emulsification, a magnetic stirrer or a paddle mixer was used. The temperature was kept at 21°C±0.5°C. The polyoxyethylene oleyl ethers, sorbitan monooleate, and polyoxyethylene sorbitan monooleate used in the experiments were commercial grade materials. The Griffin's HLB values for these surfactants are given in Table I.

*Model CM-2A Conduct Meter by Toa Electronics Ltd., Tokyo, Japan.
†Model SS-IR Rotary Torque Meter by Yamasake Seiki Kenkyuzyo, Kyoto, Japan.

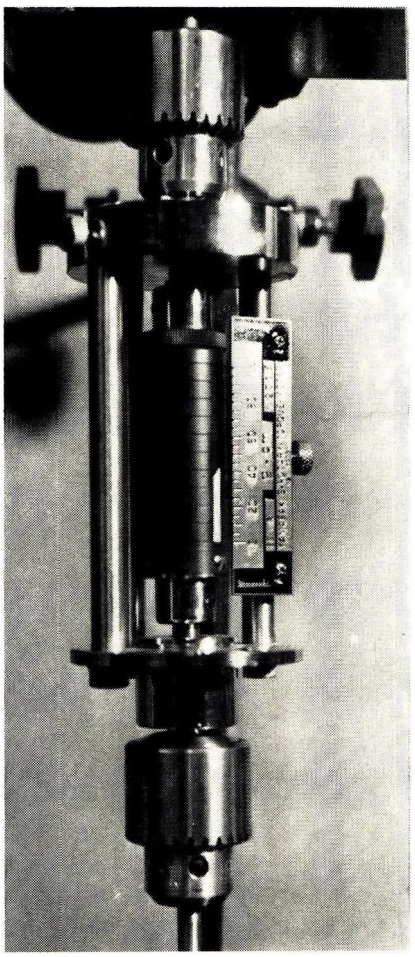

Figure 5. Photograph of a torque meter

Table I

HLB Values of the Surfactants Used in Experiments

Surfactants	Trade Name	Griffin's HLB
POE (2) oleyl ether	Nikkol BO-2[a]	4.9
POE (6) oleyl ether	Nikkol BO-6[a]	9.9
POE (7) oleyl ether	Nikkol BO-7[a]	10.7
POE (10) oleyl ether	Nikkol BO-10[a]	12.4
POE (20) oleyl ether	Nikkol BO-20[a]	15.3
POE (20) sorbitan monooleate	Tween 80[b]	15.0
Sorbitan monooleate	Arlacel 80[b]	4.3

[a] Nikko Chemical Co., Tokyo, Japan
[b] Kao-Atlas Co., Tokyo, Japan

Results and Discussion

Effect of Surfactants

From the three requirements for Mechanism A listed, one could ask what sort of surfactant properties are required to make this mechanism function. First of all, it is clear that the surfactant must not be too hydrophilic or, more accurately, not too lipophobic; otherwise it will not dissolve in the oil phase. Secondly, it also cannot be too hydrophobic or it will not form the desired final O/W emulsion. Furthermore, the second condition for Mechanism A calls for a solubilization or emulsification of water into the oil phase. It was reasoned that if aqueous solubilization was a prerequisite, a system which would not solubilize much water would not allow Mechanism A to function well. It would then appear that there might be a certain optimum HLB requirement for the surfactant to fulfill these conditions. It would be extremely interesting to determine if the required value would coincide with Griffin's required HLB to emulsify the oil.

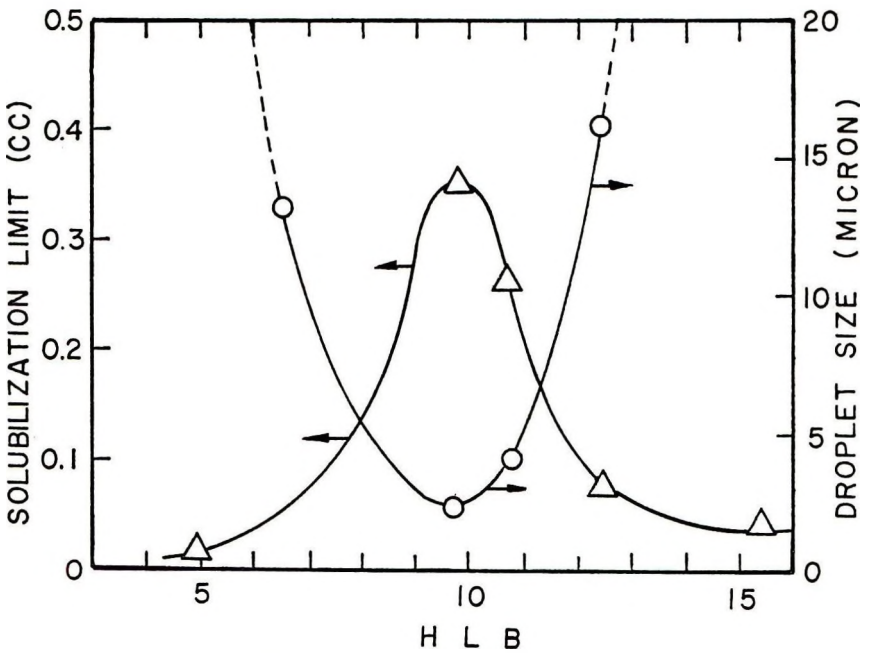

Figure 6. Effect of surfactant HLB on solubilization limit and average droplet size of the final emulsion (emulsions contain 36% mineral oil, 60% deionized water, and 4% polyoxyethylene oleyl ethers with varying ethylene oxide chain lengths corresponding to the indicated HLB)

Δ = solubilization limit

O = droplet size

To carry out the investigation, a series of polyoxyethylene oleyl ethers with ethylene oxide ranging from 2 moles to 20 moles were selected. All emulsions consisted of 30% light mineral oil, 4% surfactant, and 66% deionized water. Emulsions were prepared with the surfactant initially dispersed in the oil phase and the droplet size distribution was measured from photographs taken through an optical microscope immediately after 5 minutes of mixing at 150 rpm. To determine the amount of solubilization, water was dispersed, drop by drop, into the oil containing the same amount of the test surfactant as used in the emulsification experiments. The first sign of turbidity was taken as a sign of the solubilization limit. In some systems, however, the surfactant-oil mixture was not clear at first, but became clear with the addition of water. In such case we continued to add the water until it became turbid again.

The results of these two experiments were plotted in Fig. 6. In this series, the best emulsion was obtained with a surfactant having an HLB value corresponding to about 10. The solubilization limit was expressed in terms of the amount of water solubilized per 100 g of the final emulsion. It is interesting to

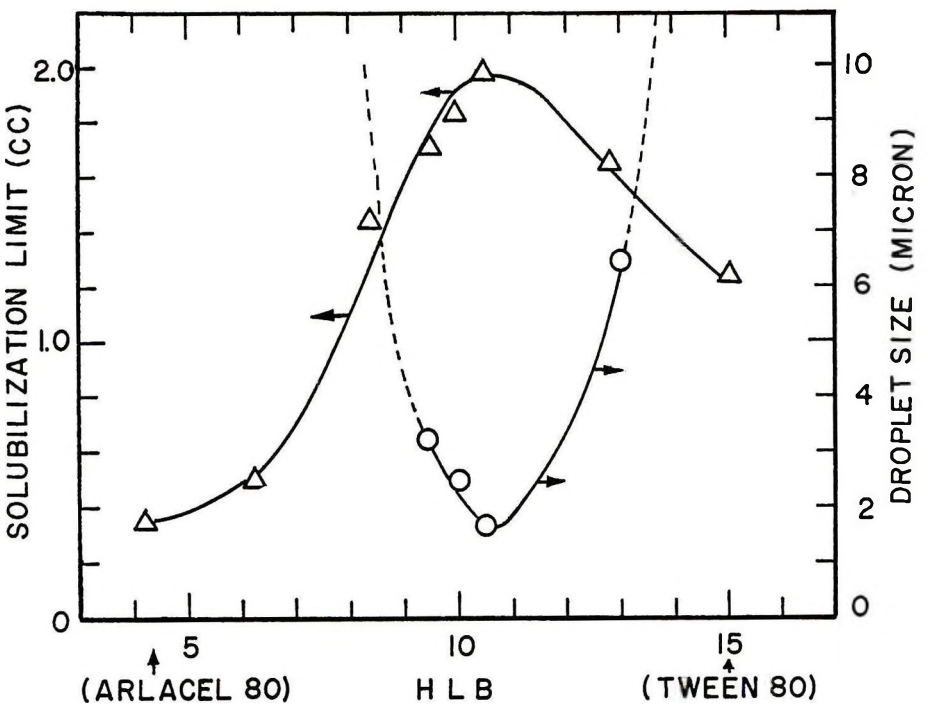

Figure 7. Effect of surfactant HLB on solubilization limit and average droplet size of the final emulsion (emulsions contain 30% mineral oil, 65% deionized water, and 5% mixtures of Tween 80–Arlacel 80 corresponding to the indicated HLB)

Δ = solubilization limit

O = droplet size

note that a maximum water solubilization was also obtained at the same HLB.

In the next series of experiments, a mixture of surfactants was used instead of a single surfactant. The experimental procedure was the same as before but Tween 80 and Arlacel 80 mixtures were used. As shown in Fig. 7, the maximum solubilization for this system also appears to correspond to the emulsion with smallest droplet size at about HLB 10.5. The required HLB value for the paraffinic mineral oil to form an O/W emulsion given in literature is about 10 (6).

From these experimental results, it would appear that in the systems studied, the surfactants or the surfactant combinations which are soluble in oil and also capable of solubilizing appreciable amounts of water can produce fine emulsions.

Effect of Oils

From the standpoint of oils, the first of the three conditions requires that the oil phase must dissolve the surfactant to make Mechanism A operative. If the hydrophilic surfactant were not soluble in the oil, even if it were initially placed in the oil phase, it would soon migrate to the aqueous phase without producing a phase inversion; consequently, Mechanism A would not be the controlling mechanism.

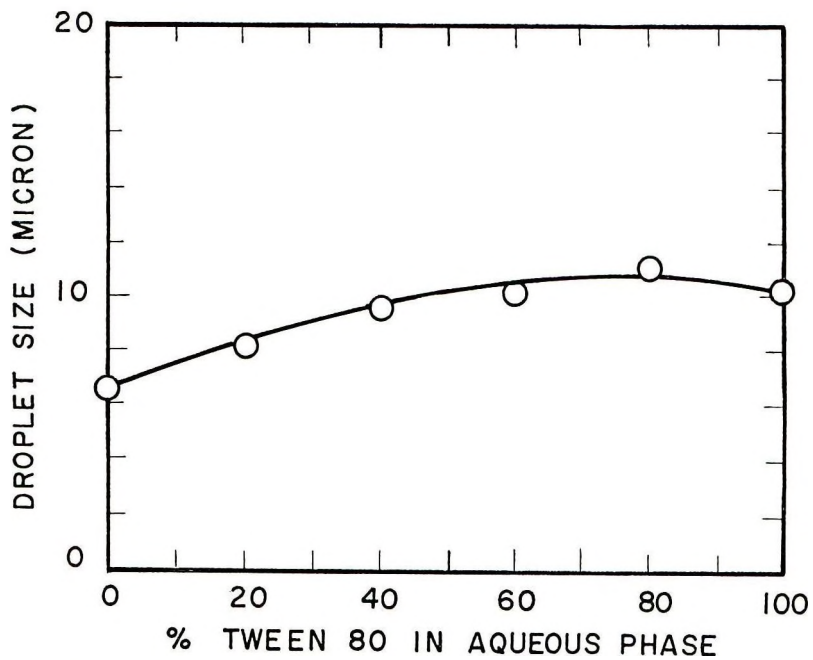

Figure 8. Effect of initial surfactant location on the droplet size of mineral oil emulsions (30% mineral oil emulsified with 8% Arlacel 80–Tween 80 mixture at HLB 11)

The two oils selected for this series of experiments were: mineral oil, which has relatively poor solubility for Tween 80; and oleyl alcohol in which the surfactant has good solubility. Emulsions were prepared with varying initial surfactant locations and the droplet size distributions were measured from microphotographs obtained for each emulsion after 5 min of mixing.

The result showing the effect of initial surfactant location on the average droplet size for mineral oil system is shown in Fig. 8. The abscissa indicates the percentage of the total Tween 80 initially placed in the aqueous phase. The nearly flat curve indicates that the effect of the initial surfactant location on the droplet size of the final O/W emulsion is a minor one. Because of the poor oil solubility of Tween 80, the effect of Mechanism A is not pronounced.

In the oleyl alcohol system, the surfactant is completely soluble and, as represented by Fig. 9, the emulsion had the smallest average droplet size when all the surfactant was placed in the oil phase. It appears that Mechanism A was controlling the emulsification near the left end of the curve; the droplets were quite small. On the right, where Mechanism A was not allowed to function, the droplets were large and the finished emulsions were unstable.

If there are indeed two separate mechanisms, it should be possible to find a point somewhere between the two extremes where both mechanisms

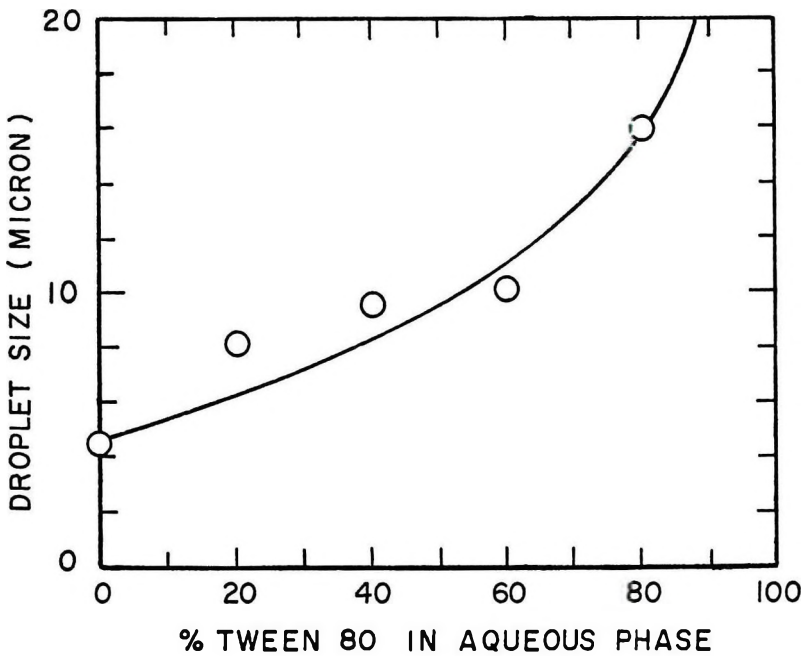

Figure 9. Effect of initial surfactant location on the droplet size of oleyl alcohol emulsions (30% oleyl alcohol emulsified with 5% Tween 80)

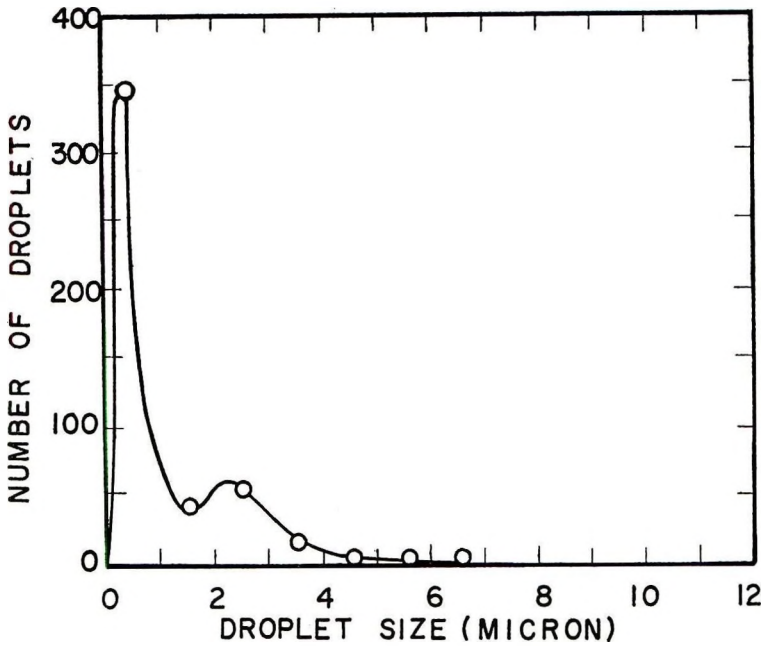

Figure 10. Droplet size distribution of an emulsion prepared with ½ surfactant initially in oil and ½ in aqueous phase (emulsion contains 30% mineral oil, 5% polyoxyethylene (6) oleyl ether, and 65% deionized water)

are functioning. A droplet size determination of such an emulsion would also reveal the operation of these two mechanisms.

An example of a droplet distribution curve for an emulsion initially prepared with half surfactant in mineral oil and half in water is shown in Fig. 10. The two peaks in the distribution curve appear to indicate that droplets were derived from two separate origins.

Effects of Preparative Methods

In addition to studying the effects of changing surfactants and oils, the effects of changing the manufacturing methods, while keeping the formula constant, were also investigated. It was reasoned that any process variables introduced in preparing the emulsion which adversely affected any of the three conditions would also cause the formation of a coarser emulsion. Conversely, variables which favorably affected any of the conditions for Mechanism A would help to produce a finer emulsion.

One of the three conditions is that the hydrophilic surfactant must be initially dissolved in the oil phase. When a mixture of 5% Tween 80–Arlacel 80 (4:1) was dispersed into the 30% oil phase, a turbid solution was obtained. However, it was discovered that if a small amount of water was mixed into this dispersion, a clear solution could be obtained.

Table II

Dissolution of the Surfactant in the Oil Phase and the Droplet Size of the Final O/W Emulsion

Per Cent Water Initially Added	Oil Phase	Average Emulsion[a] Droplet Size (μ)
0	Turbid	50
0.4	Turbid	50
0.6	Turbid	50
0.8	Clear	2
0.9	Clear	2
1.0	Clear	2

[a] Final emulsions contain 30% light mineral oil, 5% Tween 80–Arlacel 80 (4:1) mixture, and 65% deionized water. The percentage of water initially added was substrated from the remaining water to form the final emulsion.

By initially adding small portions of the total water in the formulation into the oil phase containing the surfactant mixture, experiments were carried out to determine if the enhancement of the surfactant solubility in the oil phase, without introducing any change in the over-all formulation, would also result in an improvement of the final emulsion.

The data in Table II indicate that when 0.8% of the total water was initially added to the oil-surfactant mixture to make an emulsion, the oil phase became clear. Droplet size measurements of emulsions prepared with the corresponding oil phases indicated that at the same point where the Tween 80 became soluble in the oil phase, there was a great reduction in the average droplet size of the emulsion. Therefore, it appears that the promotion of the initial dissolution of the surfactant in the oil phase does help Mechanism A to function better.

The second condition for Mechanism A requires that a portion of the aqueous phase must be quickly solubilized or emulsified into the oil phase before the hydrophilic surfactant migrates to the aqueous phase. It was postulated that an oil-surfactant mixture containing some solubilized water might emulsify more readily than the same mixture without the solubilized water. To check the validity of this argument, a series of mineral oil emulsions were prepared with polyoxyethylene (6) oleyl ether dissolved in the oil phase. Various amounts of water were mixed into this phase before carrying out emulsification using the remainder of the water to form O/W emulsions.

Figure 11 presents the results of this series of experiments. Even though all the emulsions represented here have an identical composition, the initial aqueous solubilization produces remarkable effects. In this system, an O/W emulsion with the finest droplets was produced when the emulsion was prepared by initially dissolving about 1.5% of water in the oil phase. This point,

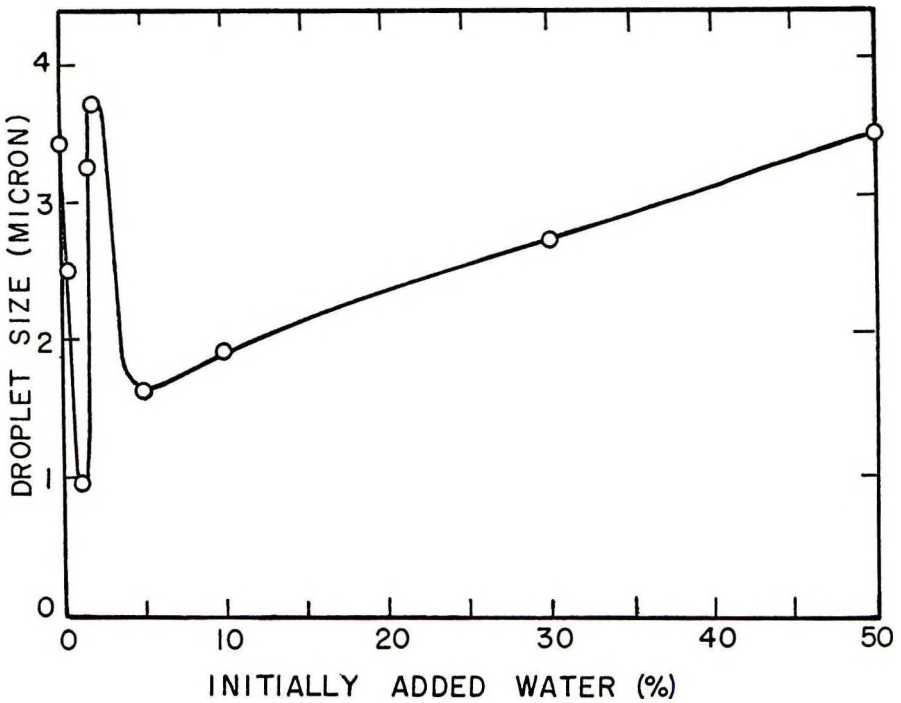

Figure 11. Effect of initial water solubilization on droplet size of final emulsion (30% mineral oil emulsified with 5% polyoxyethylene (6) oleyl ether with the surfactant initially in oil)

indicated by a letter A in the expanded curve in Fig. 12 corresponds to the point of maximum aqueous solubilization.

What was particularly surprising was the remarkable effect of the very small amount of water initially placed in the oil-surfactant mixture. When only an additional 0.3% of water was added beyond point A, the emulsion produced became very poor and had large droplets, as indicated by point B. The microphotographs of emulsions at point A and B are shown in Fig. 13.

Further investigation revealed that the probable reason for the adverse effect was that when the initial amount of water added exceeded the solubilization limit at point A, the primary W/O emulsion became quite unstable and released water from the system. The chemical analysis of the separated aqueous phase indicated that the relatively hydrophilic surfactant used soon migrated to the aqueous phase and deprived the oil phase of the surfactant needed to make Mechanism A operative. The remarkable effect on an emulsion produced by such a minor variation in the preparative method demonstrates the difficulty in trying to control or pinpoint some manufacturing variables in a large-scale plant operation.

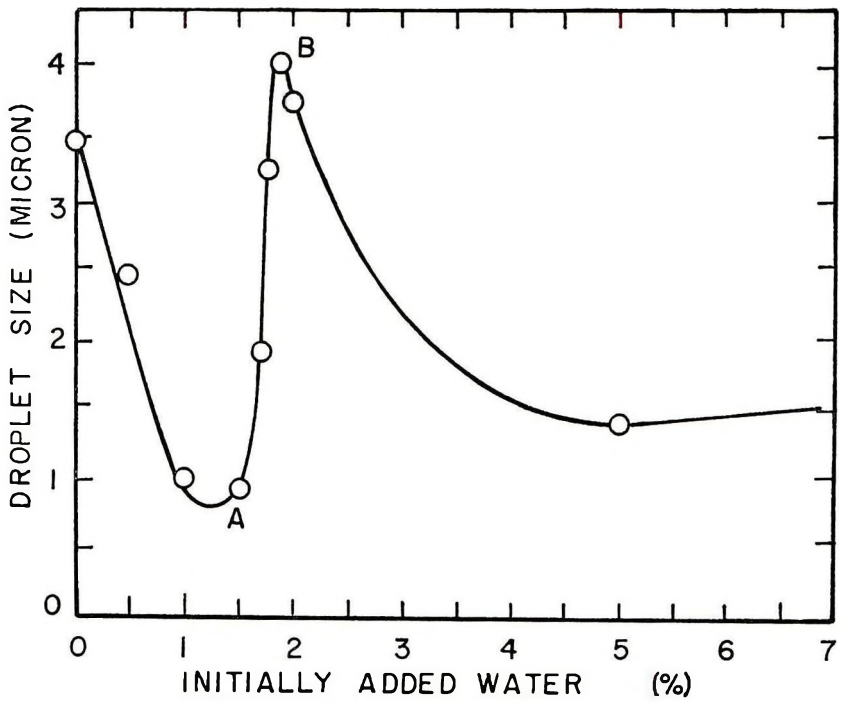

Figure 12. Left end of Fig. 11 expanded (point A corresponds to solubilization limit; point B corresponds to a point where the primary emulsion became unstable)

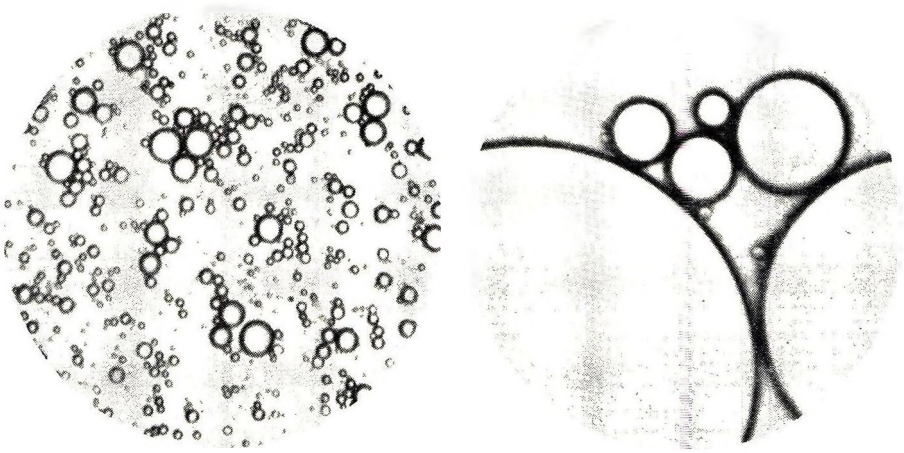

Figure 13. Microphotographs of O/W emulsions prepared at points A (left) and B (right) in Fig. 12

The third condition which is the most important condition for Mechanism A is that a phase inversion must take place. This inversion may involve the entire emulsion or may be localized. When Mechanism A is operative, an intensive mixing is not usually required to produce a fine emulsion. However, sufficient mixing must be provided to effect a phase inversion before the surfactant migrates to the aqueous phase.

One important factor affecting phase inversion during the manufacture of emulsion is the temperature. It is known that O/W emulsions stabilized by nonionic surfactant can undergo a phase inversion to form W/O emulsions when the temperature is raised to a certain point. This limiting temperature is called the "phase inversion temperature" or simply PIT. Shinoda and other investigators have measured the PIT's of many nonionic systems and reported their relationships with other factors (7,8).

Since a hydrophilic nonionic surfactant becomes more lipophilic at a temperature above the PIT, it is expected that the surfactant in an O/W emulsion prepared at room temperature would migrate to the oil phase to form a W/O emulsion. Upon cooling, this W/O emulsion reinvert to form an O/W emulsion and the surfactant would again migrate from the oil phase to the aqueous phase. This process is quite similar to Mechanism A. Hence, by

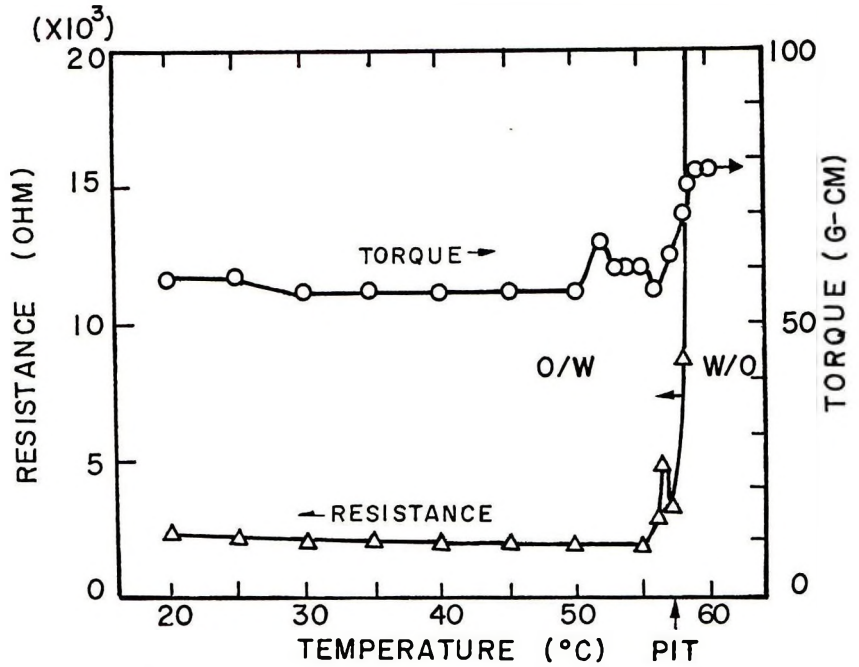

Figure 14. Calibration of torque meter readings against electrical resistance measurements (emulsion contains 30% mineral oil, 5% polyoxyethylene (6) oleyl ether, and 65% deionized water)

first heating an O/W emulsion to a temperature above the PIT and subsequently cooling the emulsion rapidly, one can expect formation of a fine emulsion.

There are several different ways of determining the PIT of an emulsion. One may measure the electrical conductivity or use differential thermal analysis (9,10). For this investigation, a torque meter, shown in Fig. 5, was used. This is a relatively inexpensive device which can be directly attached to the mixer shaft to continuously monitor the torque on the mixer while the emulsification is in progress. The emulsification system is not disturbed by the presence of other probes.

Normally, at the point of phase inversion, there is a sharp change in the viscosity of the emulsion which is reflected in the torque reading. As shown in Fig. 14, this method was first checked against the measurements obtained by a conductivity meter. As indicated, there was a sharp increase of both the electrical resistance and the torque measured by the torque meter at about 57°C, which was the PIT of this system.

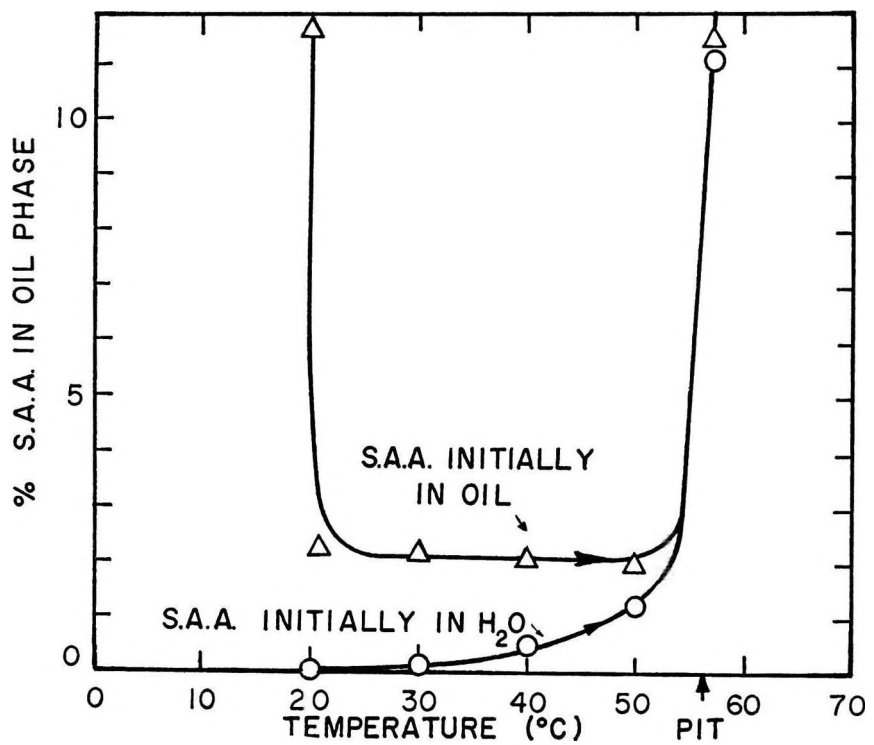

Figure 15. Migration of the surfactant in emulsified systems (emulsions contain 30% light mineral oil, 5% polyoxyethylene (6) oleyl ether, and 65% deionized water)

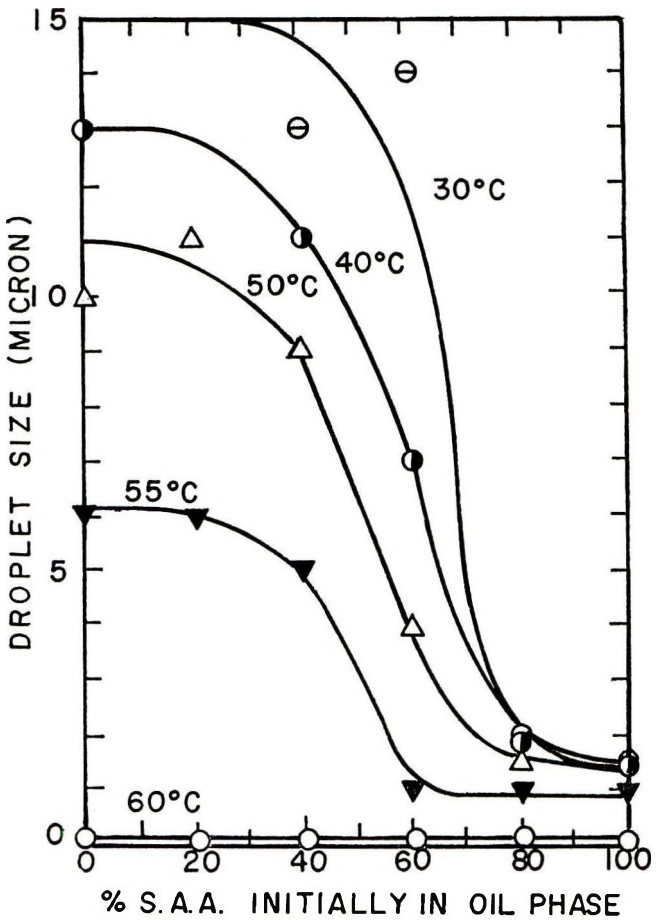

Figure 16. Effects of surfactant location and emulsification temperature on droplet size (emulsions contain 30% mineral oil, 5% polyoxyethylene (6) oleyl ether, and 65% deionized water)

Measurements on the migration of the surfactant were also made to make certain there was a significant migration near the PIT. As indicated in Fig. 15, whether the surfactant was first placed in the oil phase or the aqueous phase, the relatively hydrophilic surfactant polyoxyethylene (6) oleyl ether would rapidly migrate from the aqueous phase to the oil phase at the PIT.

After it was certain that a rapid surfactant migration would take place near the PIT, a series of emulsions with different surfactant locations was prepared, and samples were taken at various temperatures to determine the droplet size distribution by microphotography.

As shown in Fig. 16, at lower temperatures, the effect of the surfactant location was more significant than the temperature. For example, by increasing the emulsification temperature from 30°C to 55°C, the average drop-

let size was reduced to ⅓. However, even at 30°C, the droplet size could be reduced to about 1/10 by placing the entire surfactant in the oil instead of the aqueous phase. However, above the PIT (57°C), the situation was quite different as all emulsions prepared at 60°C produced extremely fine droplets regardless of the initial surfactant location. Therefore, as pointed out by Shinoda, a good way to make emulsions stabilized by nonionic surfactants is to heat the emulsion to a temperature above the PIT and then rapidly cool it to a lower temperature (**11, 12**).

Conclusions

In summary, it has been demonstrated that by directly or indirectly varying the surfactant location, remarkable effects can be produced on the physical characteristics of O/W emulsions.

It is believed that this is caused by the basic difference between the emulsification mechanisms involved. By investigating the effects of varying surfactants, oils, and manufacturing conditions, it has been shown that the effects produced were consistent with the postulated mechanisms.

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Methodisches zum Nachweis des Hauttalges unter besonderer Berücksichtigung der Akne

HAGEN TRONNIER*

Vortrag anläßlich der Vortrags- und Diskussionstagung der Gesellschaft Deutscher Kosmetik-Chemiker e. V., Baden-Baden, Bundesrepublik Deutschland, 14.—16. März 1974

Synopsis—Methods for the determination of sebaceous matter of the skin in regard to acne.—Problems in the DETERMINATION OF SKIN LIPIDS are caused by its non-uniform composition, the different locale used for collection, and the questionable completeness of lipid collection. Advantages and disadvantages of the different methods—in part based on the author's experiments—are described. The three disorders of the skin lipid systems which are of concern for SEBORRHOEA and related diseases include the amount, the composition, and the physical behavior of the LIPID FILM ON THE SKIN'S SURFACE. Of these, the last appears to be most important. This is based on numerous investigations of ACNE and of seborrhoea cases. The importance of the ratio of quantity of lipid to its SPREADING CHARACTERISTICS in the formation of ACNE COMEDONES is pointed out and is illustrated with investigations of skin lipids by different methods during GLUCOCORTICOID THERAPY. The disturbance of this ratio in acne is further illustrated with the aid of additional experimental results.

Der Lipidgehalt der Haut und ihrer Oberfläche hat bekanntlich wichtige Funktionen innerhalb der verschiedenen Schutz- und Abwehrmechanismen der Haut. Er beeinflußt positiv und negativ die Penetration chemischer Substanzen und spielt eine wichtige Rolle für das Funktionieren der Hornschicht, z. B. ihres Wasser-Lipid-Mantels [Schneider (1)]. Ebenso wie fehlende oder zu geringe Lipidproduktion in der Haut zu Krankheitsbildern führen kann (z. B. dem État craquelé), gehört die vermehrte Talgsekretion als Seborrhoe zu den häufigsten kosmetischen Störungen und stellt als solche eine Mitursache auch dermatologischer Krankheitsbilder dar (z. B. der Acne vulgaris). Im Rahmen allgemeiner Anpassung an die sie umgebende Umwelt unterliegt naturgemäß auch der Lipidgehalt der Haut Schwankungen und wird zudem durch externe Maßnahmen beeinflußt. So ist es verständlich, daß seit langem mit unterschiedlichen

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Methoden versucht wurde, den Lipidgehalt der Haut zu bestimmen und seine Beeinflussung durch therapeutische oder kosmetische Maßnahmen zu erfassen.

Mehr noch als bei anderen hautphysiologischen Untersuchungen ergeben sich hierbei aber Probleme, vor allem aus folgenden Gründen:

1. Das Hautfett stellt keine einheitliche Substanz dar,
2. es entsteht an unterschiedlichen Stellen der Haut (Talgdrüsen und Hornschicht) und unterliegt noch sekundären Veränderungen bei der Sekretion,
3. es ist unterschiedlich in und auf der Haut lokalisiert (Oberfläche, Hornschicht, Talgdrüsen und deren Ausführungsgänge).

Die unterschiedliche chemische Zusammensetzung der Hautlipide führt dazu, daß mit einigen Nachweismethoden die Fraktionen nicht vollständig erfaßt werden. Die unterschiedliche Lokalisation und Bildung in der Haut hat zur Folge, daß Menge und Zusammensetzung von der jeweiligen Methode der Fettgewinnung von der Haut abhängig sind. Man kann dabei bezüglich der Abnahmemechanismen zwischen Abdruckmethoden und Extraktionsmethoden unterscheiden.

Durch die Abdruckmethoden wird nur das Hautoberflächenfett, also der casual level, erfaßt, wobei natürlich auch wieder Unterschiede in Abhängigkeit

Versuchsperson	Alter (Jahre)	Extrahierte Fläche (cm^2)	Lipidmenge (mg)	Lipidmenge auf 1000 cm^2 (mg)
A) Hautgesunde				
1	28	950	13,3	14,0
2	25	950	12,8	13,5
3	27	900	10,9	12,1
4	31	1100	14,5	13,2
5	27	1100	14,2	12,9
Mittelwerte	27,6	1000	13,14	13,14
B) Acnekranke				
1	30	950	14,5	15,2
2	18	1000	16,4	16,4
3	17	900	13,8	15,3
4	18	850	13,1	15,4
5	17	850	11,5	14,3
Mittelwerte	20,0	910	13,86	15,32 (+ 16,6 %)

Tabelle 1

Unterschiede in den Hautoberflächenlipiden bei gesunden Probanden im Vergleich zu Patienten (n = 5) mit Acne. Methode: Abreiben einer Fläche von 500—1100 cm^2 auf Stirn und Rücken mit äthergetränktem Wattebausch, Extraktion der Lipide mit Äther und Abdampfen des Lösungsmittels im Vakuum, bei Raumtemperatur und Wägung des Rückstandes (5).

vom Abnahmematerial (wie Filter- oder Zigarettenpapier, Stoff, Leder, Glas) bestehen.

Bei den Extraktionsmethoden bestimmen die verwendeten Lösungsmittel und deren Einwirkungszeit die Menge der extrahierbaren Lipide. Zu Nachweis und Bestimmung der Lipide der Haut wurden chemische und physikalische Verfahren [wie mittels Osmiumsäure, Sudanfarbstoffen, Oxidation mittels Dichromat, Gas-Chromatographie, IR-Spektroskopie, Extraktions- und Abdruckverfahren (gravimetrisch und optisch), Nephelometrie, Spreitung] empfohlen. Es sei auf die zusammenfassende Literatur verwiesen (2) (3) (4). Die Auswahl der Methodik hängt in erster Linie stets von der Fragestellung ab.

So läßt sich zwar bei Patienten mit einer Acne eine vermehrte Talgmenge z. B. gravimetrisch oder colorimetrisch nachweisen *(Tab. 1)*, wenn es jedoch

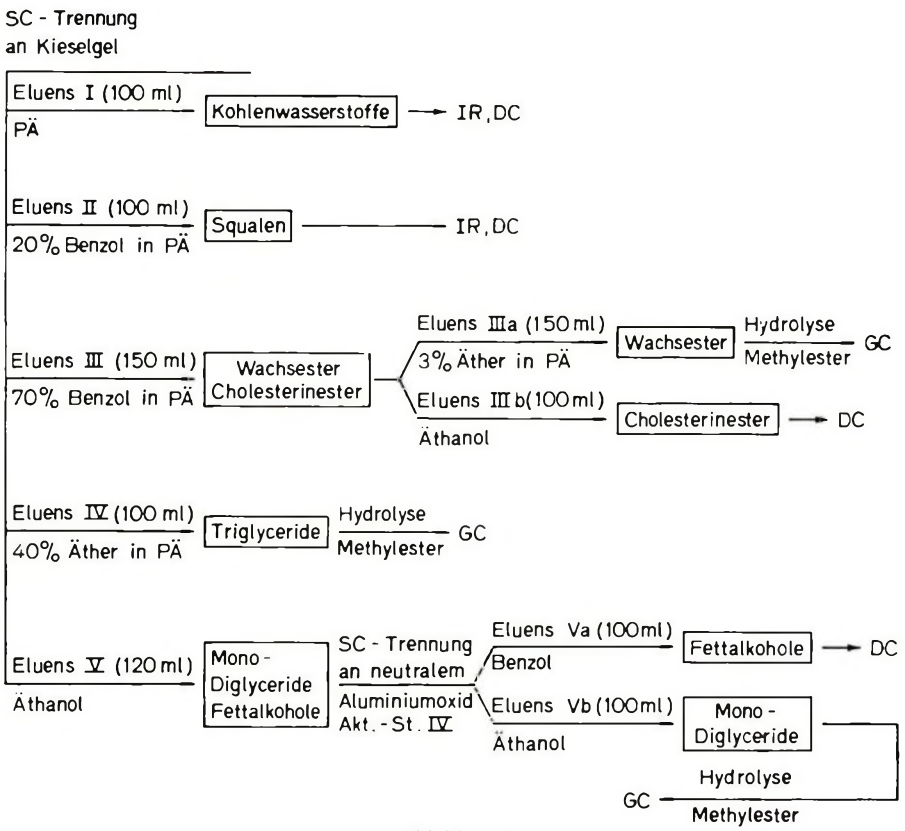

Abbildung 1

Auftrennung der Hautoberflächenlipide mittels Säulenchromatographie (Sc). PÄ = Petroläther, IR = IR-Spektroskopie, DC = Dünnschicht-Chromatographie, GC = Gas-Chromatographie (5).

darum geht, eventuelle unterschiedliche Talgzusammensetzungen im Hinblick auf die Pathogenese diese Erkrankung zu untersuchen, ist eine ausführliche Analyse notwendig, wie sie als Möglichkeit im folgenden Schema *(Abb. 1)* dargestellt ist. Mit dem letzteren Verfahren lassen sich, wie hier für die Hautoberflächenlipide durchgeführt, quantitative Unterschiede in den einzelnen Fraktionen ebenso nachweisen wie z. B. eine relative Vermehrung kurzkettiger Fettsäuren, mit ihrer bekannten Eigenschaft stark zu irritieren, bei Acne-Kranken *(Abb. 2 u. 3)*.

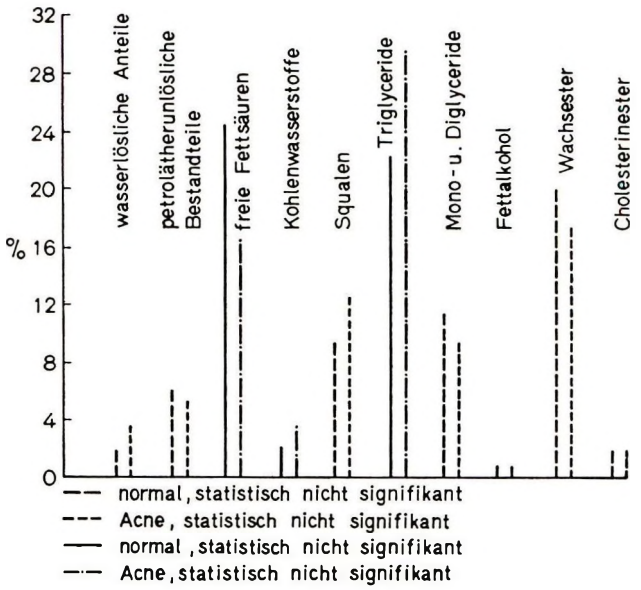

Abbildung 2

Prozentuale Verteilung einzelner Fraktionen des Hautoberflächentalgs bei gesunden Probanden und Patienten mit Acne (6).

Wenn auch die physikalischen Methoden primär eine geringere Abhängigkeit der Meßwerte von der chemischen Zusammensetzung des Hautfettes erwarten lassen, so trifft dies, wie die Praxis zeigt, keineswegs generell zu. In einer Studie (7) über den Vergleich zwischen gravimetrischen und colorimetrischen Verfahren einerseits und dem Spreittest (Sebotest) nach Röth (8) andererseits ließ sich zeigen, daß insbesondere der Osmium-Test von Brun (9) besser geeignet ist, eine quantitative Aussage über die Talgmenge zuzulassen.

In einer Untersuchungsreihe wurden die drei erwähnten Methoden zur Bestimmung der eventuell unterschiedlichen Wirkung verschiedener Corticosteroide auf die Talgsekretion verwendet. Für die Versuche wurden jeweils 12 Probanden herangezogen. Sie erhielten über 7 Tage je 20 mg Prednisonäqui-

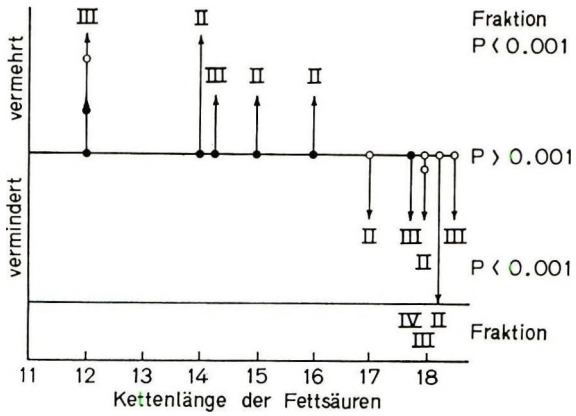

Abbildung 3

Unterschiede in dem Anteil Fettsäuren unterschiedlicher Kettenlängen im Acnetalg in den einzelnen Fraktionen (s. *Abb. 1)*, gegenüber dem hautgesunden Probanden (6).

valent, nämlich: 16 mg Triamcinolon, 3 mg Betamethason, 20 mg Fluocortolon oder 20 mg Prednison.

Die Abnahme des Talges erfolgte am 1., 3., 5., 6. und 7. Versuchstag. Jeweils 16 Std. vor der Abnahme waren die Meßstellen mit Hilfe eines mit Aceton getränkten Wattebausches abgerieben und danach so abgedeckt worden, daß kein Abrieb erfolgte. Die Abnahme fand ebenfalls mit einem solchen Wattebausch statt. Abnahmeort war die Haut der Fossa supraspinata beider Schulterblätter. Bezüglich weiterer methodischer Einzelheiten zu diesen Versuchen sei auf eine zusammenfassende Darstellung verwiesen (10).

Berücksichtigt man in diesem Versuch zunächst einmal die Mittelwerte aller Messungen unabhängig vom verwendeten Steroid und unterteilt diese dann noch entsprechend der Talgsekretion in einer starke, mittlere oder geringere anhand der Ausgangswerte, so erhält man mit den drei Methoden die in der *Abb. 4* dargestellten Kurvenverläufe. Deren Ähnlichkeit, vor allem der Anstieg der H- und Mittelwertkurven, sowie die Ähnlichkeit der beiden M- und Mittelwertkurven für das Verfahren mittels Dichromatoxidation und den Osmiumtest sind auffallend, obwohl es sich einmal um ein Extraktionsverfahren, zum anderen um eine Abdruckmethode handelt. Der Knick im 6-Tage-Wert beim Osmiumtest ist auf äußere klimatische Einflüsse zurückzuführen, die sich an den Oberflächenlipiden natürlich stärker bemerkbar machen als bei der Extraktionsmethode. Das gleiche gilt wahrscheinlich auch für die größeren Unterschiede in der N-Kurve, die im übrigen in beiden Tests den Anstieg durch die Corticoid-Medikation vermissen läßt.

Keine Parallelität zu den Werten des Osmiumtests zeigt dagegen der Sebotest. Hier findet man auch keine Zunahme bei mittleren und geringen Talg-

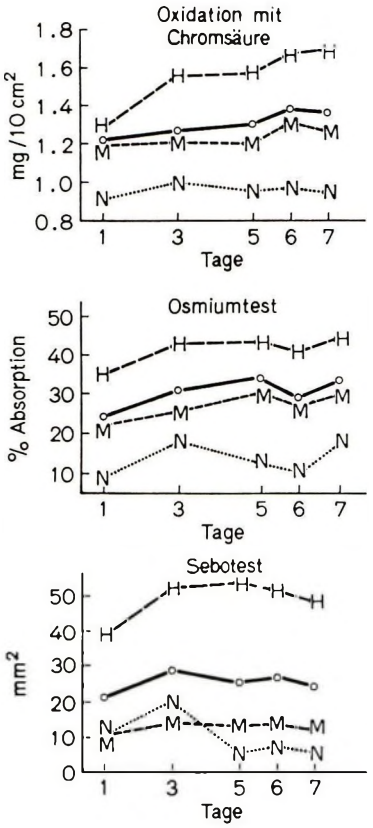

Abbildung 4

Zeitlicher Verlauf der Talgmeßwerte nach den drei Methoden: Oxidation mit Osmiumtest und Sebotest unter Corticosteroidmedikation, bestimmt (n = 48 bzw. 16); Probanden mit verschiedenen Ausgangstalgspiegeln: H: hohe Ausgangswerte, M: mittlere Ausgangswerte, N: niedrige Ausgangswerte, ausgezogene Kurve: Mittelwerte.

spiegeln, während bei hohen nur im ersten Meßzeitbereich eine solche Zunahme zu verzeichnen ist. Unterstellt man den verwendeten Methoden Zuverlässigkeit bezüglich der Parameter der Bewertung, so würde dies einer Zunahme der Talgmenge gemäß den ersten beiden Tests bei fehlender oder nur geringer Spreitungszunahme entsprechen. Dadurch würde aber sich eine Situation ergeben, wie sie bei der Acne vorliegt, in der die Talgmenge zwar ebenfalls zunimmt *(Tab. 2)*, aber die Spreitung sich eher verschlechtert.

Die geringere Spreitung der Lipide bei Acne ist aus *Tab. 2* ersichtlich, in der die prozentuale Änderung gegenüber hautgesunden Kontrollpersonen dargestellt ist (11). Während die Wasserspreitung bei der Acne relativ stärker ist, liegt der Wert der Lipidspreitung trotz größerer Talgmenge unter derjenigen

Test Einwirkungszeit: 30″	wäßrige Methylenblau-Lösung 0,3 %	öilge Sudanrot-Lösung 1 %
Kontrolle	136	84
Wasser	200	95
Seifenlösung 1 %	100	106
Natriumlaurylsulfat 1 %	163	93
Quart-Ammonium-Verbindung 1 %	276	98
wäßrig-alkoholische Lösung (25 % Alkohol)	204	57
absoluter Alkohol	122	60
Aceton	97	85
Umkehr-Emulsion	76	82

Tabelle 2

Prozentuale Änderung der Spreitung wäßriger und öliger Lösungen nach Vorbehandlung der Haut bei Acnekranken gegenüber hautgesunden Kontrollpersonen (n = 12, Meßort: Rücken).

der Kontrollwerte. Die Lipidspreitung wird dabei, dies sei nebenbei vermerkt, durch Alkohol und alkoholisch-wäßrige Lösungen im Gegensatz zu oberflächenaktiven Stoffen relativ noch stärker vermindert. Dies stimmt mit Alkalineutralisationsmessungen an der Haut von Seborrhoikern und Acnekranken (12) überein. Auch bei der Acne wurden niedrigere Alkalineutralisationswerte, d. h. längere Zeiten als bei Seborrhoikern gefunden.

Aufgrund der Untersuchungsergebnisse kann man hinsichtlich der Verwertbarkeit der verwendeten Tests folgern, daß das gravimetrische Extraktionsverfahren und der Osmiumtest zur quantitativen Messung der Hautoberflächenlipide geeignet sind, während der Spreittest vorzugsweise als qualitatives Verfahren, und zwar zur Beurteilung der „Spreitungsfähigkeit", brauchbar ist. Bei der vorhin beschriebenen Vergleichsuntersuchung mit Zunahme und relativer Verringerung der Spreitungsfähigkeit des Talges müssen aber, wie die Vergleichsuntersuchungen an Acnekranken gezeigt haben, acnefördernde Bedingungen entstehen. Dies entspricht auch der klinischen Nebenwirkung der Glucocorticoide, bei der die Steroidacne gegenüber der — allerdings auch vorkommenden — Seborrhoe eindeutig klinisch im Vordergrund steht.

Differenziert man nun die Werte nach den einzelnen Glucocorticoiden für die drei Methoden *(Abb. 5)*, so fällt zunächst, besonders deutlich im Osmiumtest, die Zunahme der Talgmenge auf. Bei der Extraktionsmethode erscheinen die Werte für die einzelnen Corticoide differenzierter, besonders hoch nach Prednison, am niedrigsten nach der Gabe von Betamethason. Im Sebotest nehmen die Spreitwerte sehr viel weniger zu oder, wie bei Betamethason, gar nicht.

Vergleicht man die prozentuale Zunahme der Werte unter dem Versuch (Anfangs- zum Endwert) zwischen der Extraktionsmethode und dem Sebotest, unter Bildung der Differenz zwischen beiden Werten *(Tab. 3)*, so zeigt sich,

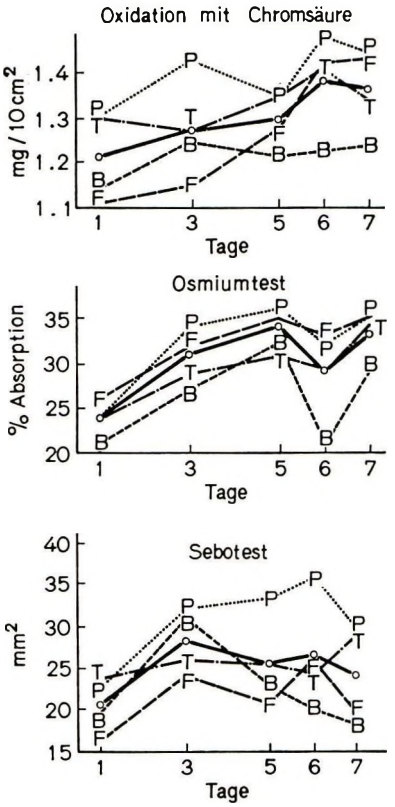

Abbildung 5

Zeitlicher Verlauf der Talgmeßwerte nach den drei Methoden: Oxidation mit Chromsäure, Osmiumtest und Sebotest, unter Corticoidmedikation bestimmt (n = 48 bzw. 12); Probanden mit verschiedenen Ausgangstalgspiegeln für die Präpaealegruppen: P: Prednison, T: Triamcinolon, F: Fluocortolon, B: Betamethason. — Ausgezogene Kurve: Mittelwerte.

Präparat	Osmiumtest in %	Sebotest in %	Differenz in %
Prednison	+ 46	+ 35	+ 9
Triamcinolon	+ 41	+ 17	+ 24
Fluocortolon	+ 35	+ 25	+ 10
Betamethason	+ 33	— 10	+ 43

Tabelle 3

Prozentuale Änderung der Menge der Hautoberflächenlipide (Osmiumtest) zur Spreitungsänderung (Sebotest) während des Versuches (n = 48 bzw. 12).

daß für Betamethason und Triamcinolon die Differenz am größten ist, mithin ihre acneigenen Eigenschaften ausgeprägter sein müssen, als die des Prednisons und — was etwas überrascht — auch des Fluocortolons. Wenn also die klinische Beobachtung der starken acneogenen Eigenschaft doppelt-fluorierter Steroide auch durch diesen Versuch grundsätzlich zu bestätigen war, die klinisch besonders eindrucksvoll bei der perioralen Dermatitis gefunden wird, scheint dies doch nicht für alle diese Verbindungen gleicherweise zu gelten. Daß andererseits auch nicht fluorierte Glucocorticoide, wie das Prednison, so wirksam sind, geht aus dem Vergleich der nach Osmium- und Sebotest erhaltenen veränderten Werte hervor. Die Differenz zeigt eine größere Steigerungsrate zugunsten des Osmiumtests an.

Bei gravimetrischer Bestimmung der Hautoberflächenfette, wie z. B. von Strauss et al. (13) angegeben, wird die Auswertung dadurch erschwert, daß Schweiß abgetrennt werden muß, was im Falle einer Emulsion an der Oberfläche nicht ganz leicht ist; außerdem ist die abgenommene Talgmenge im Vergleich zum Träger Papier immer noch relativ gering. Die Werte variieren sehr infolge Veränderungen durch Hantieren oder andere Einflüsse, wie wir (z. B. bei gravimetrischen Schweißuntersuchungen) feststellen konnten.

Eine diese Fehler weitgehend vermeidende physikalische Methode wurde kürzlich von Schaefer und Kuhn-Bussius (14) angegeben. Sie beruht auf der Erhöhung der optischen Transmission einer Mattglasscheibe bzw. eines Mattglasblocks in Abhängigkeit von der aufgetragenen Fettmenge. Die Brauchbarkeit des Verfahrens wurde in verschiedenen Publikationen belegt (15) (16).

Abbildung 6
Hautlipidmeßgerät

Das Verfahren setzt aber das Vorhandensein eines entsprechenden Photometers voraus und erfordert im Umgang mit diesen Geräten geschultes Personal. Während die Methode für wissenschaftliche Laboratoriumsuntersuchungen sehr geeignet ist, würde sich ein einfacher zu bedienendes Gerät für die klinische Routine und auch im Hinblick auf Aufgaben der Kosmetik leichter einsetzen lassen.

Ein auf dem prinzipiell gleichen Meßprinzip aufgebautes handliches Gerät (Hersteller Fa. Creachem, Holzminden), bei dem anstelle der geeichten Mattglasscheiben ein etwa 0,1 mm starkes mattiertes Kunststoffband auf eine Rolle gewickelt zur Lipidabnahme benutzt wird, dürfte für den genannten Zweck brauchbar sein *(Abb. 6)*.

Das Gerät ist mit zwei 1,5-V-Batterien ausgerüstet und wird vor der Messung geeicht. Die ca. 1 cm² große Meßfolie wird vor der Messung durch eine einfache Drehung an der Aufwicklungsrolle eingestellt, anschließend der eigentliche Meßansatz mit einem Druck von 1 kp auf die Haut aufgedrückt. Danach wird durch Einstecken dieses Meßansatzes in das Grundgehäuse die durch einen hinter der Meßfolie angebrachten Spiegel verstärkte 45°-Reflexion der Meßfolie mit einem Photoelement gemessen und der Wert an einem Mikroampère-

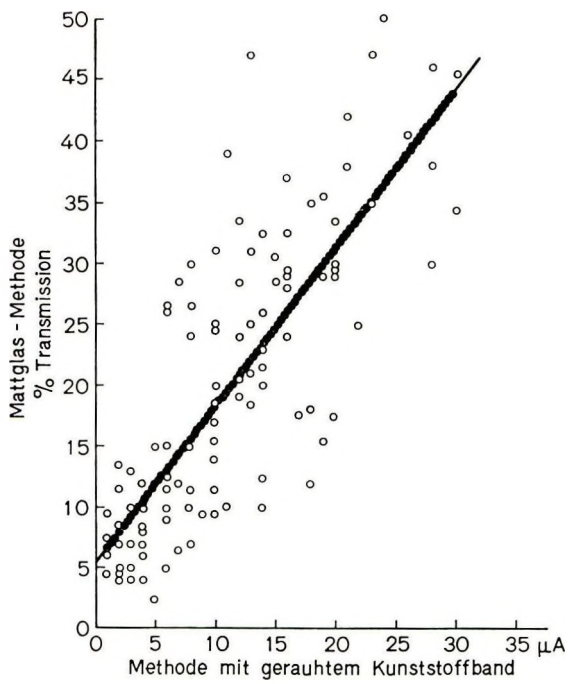

Abbildung 7

Korrelation der Werte nach der Mattglas- mit den Werten nach der Kunststoffband-Methode.

meter abgelesen. Anschließend kann, nach Weiterdrehen des Folienbandes, das für ungefähr 500 Messungen ausreicht, sofort die nächste Bestimmung durchgeführt werden. Bei Auswertung ist die Leerreflexion von den Meßwerten abzuziehen, eine Absoluteichung auf Lipidmenge ist auch mit diesem Verfahren möglich.

Zur Überprüfung der Brauchbarkeit dieses Gerätes wurde eine Vergleichsuntersuchung zu dem Originalverfahren von Schaefer und Kuhn-Bussius durchgeführt. Im Rahmen dieser Untersuchung sollte dann gleichzeitig der Einfluß einer „antiseborrhoischen" Substanz überprüft werden, über deren Effekt von Eberhardt (17) berichtet wurde. Dieser Wirkstoff schien wegen der beschriebenen ausgeprägten Hemmung der Rückfettung der Haut als Modellsubstanz besonders geeignet. Über diese Untersuchungen soll jedoch gesondert berichtet werden. Es sei hier nur das Ergebnis einer Vergleichsmessung mit beiden Methoden dargestellt *(Abb. 7)*.

Generell fällt hinsichtlich des Effekts extern zugeführter Stoffe auf den Talgspiegel, wie der Literatur zu entnehmen ist, auf, daß lediglich bei interner Medikation Hormone die Talgsekretion beeinflussen. Dies konnten wir auch bezüglich der Einnahme von Ovulationshemmern (18) finden, weiterhin wurde es für bestimmte Antibiotika und Psychopharmaka (2) (3) bestätigt.

Externe Maßnahmen, wie die Anwendung von Lösungs- oder Emulsionswaschmittel, führen zwar zu einer Entfettung der Hautoberfläche, die Rückfettung wird aber offenbar lediglich entsprechend dem unterschiedlichen Maß der Entfettung beeinflußt. Für übliche antiseborrhoische Wirkstoffe konnten kürzlich Gloor et al. (19) keine seboregulierende Wirkung feststellen. Wir selbst konnten dagegen kürzlich mit einem ein Cystein-Derivat enthaltenden Shampoo gegenüber einem wirkstofffreien mit Hilfe des Osmiumtests auch eine Abnahme des Fettes der Kopfhaut nachweisen (20) *(Tab. 4)*.

Kopfwäsche	Shampoo	
	mit Wirkstoff	ohne Wirkstoff
vorher	60,1	58,5
nach		
4.	67,6	41,4
8.	33,6	34,8
12.	22,4	38,3
16.	17,4	53,0
20. Waschen	15,7	69,1
1 Woche nach letztem Waschen	27,7	77,9

Tabelle 4

Oberflächenfett der Kopfhaut bei Seborrhoikern in Abhängigkeit von der Zahl der Kopfwäschen (n = 10, Osmiumtest, Werte der Lichtabsorption in %).

Während ein kontinuierlicher Abfall der Menge des Hautoberflächenfettes nach Anwendung des wirkstoffhaltigen Shampoos und nur ein geringer erneuter Anstieg nach 1 Woche Pause gegenüber dem letzten Wert festzustellen war, stieg der Talgspiegel der Hautoberfläche bei der Kontrolle nach anfänglicher Verminderung gegen Ende des Versuches sogar noch über den Anfangswert.

ZUSAMMENFASSUNG

Die Problematik der Hautfettbestimmung wird beschrieben, die in der uneinheitlichen Zusammensetzung, in der unterschiedlichen Lokalisation und in der verschieden vollständigen Gewinnung des Hauttalgs begründet ist. Vor- und Nachteile der verschiedenen Methoden werden, teilweise unter Heranziehung eigener Versuche, dargestellt. Von den drei für die Seborrhoe und die Erkrankungen des seborrhoischen Formenkreises in Frage kommenden Störungen im Hauttalgsystem, nämlich in der Menge, in der Zusammensetzung und im physikalischen Verhalten des Talgfilms auf der Hautoberfläche, scheint letzterem die wesentlichste Rolle zuzukommen. Dies konnte aus zahlreichen Untersuchungen einerseits bei der Acne und andererseits bei der Seborrhoe abgeleitet werden. Auf die Bedeutung des Verhältnisses von Talgmenge zur Spreitungsfähigkeit für die Ausbildung von Comedonen bei Acne wird anhand vergleichender Talguntersuchungen mit verschiedenen Methoden und unter Glucocorticoid-Medikation hingewiesen. Die bei Acne vorliegende Störung in dieser Relation wird an weiteren experimentellen Befunden erörtert.

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Utility of Amine Oxides in Oil/Water Cosmetic Systems

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Presented May 9, 1974, Seminar, Chicago, Illinois

SYNOPSIS—The use of AMINE OXIDES as EMULSIFIERS for OIL-IN-WATER COSMETIC SYSTEMS is investigated. Various typical cosmetic systems are described in which amine oxides are used in place of conventional nonionic and anionic emulsifiers. The amine oxide emulsified systems were successfully PRESERVED from microbiological contamination by using readily available, inexpensive quaternary ammonium compounds. These preserved systems were then tested for SKIN and EYE IRRITATION and found to be nonirritating.

Introduction

The cosmetic industry today uses many emulsifiers in the production of creams, lotions, and specialty products. These emulsifiers cover just about every class of surfactant. There are nonionics, examples of which would be glycerol or glycol fatty acid esters, and ethylene oxide condensates of alcohols or fatty acids; anionics such as amine soaps of fatty acids or fatty alcohol sulfates; and, to a lesser extent, cationics of the quaternary ammonium halide type, and the amphoterics. It is the object of this paper to show that another class of surfactants, amine oxides, have utility and inherent advantages in their use.

Amine oxides are prepared generally by the reaction of a tertiary amine with hydrogen peroxide under controlled conditions. Figure 1 shows the general reaction with three typically used tertiary amines. Here alkyl dimethyl amine, alkyl amido propyl dimethyl amine, and alkyl bis hydroexthyl amine are illustrated as examples of amines reacted with hydrogen peroxide to give the oxide. Other amines (alkyl morpholine, for example) are also converted to the oxide and sold commercially.

*Onyx Chemical Co., Div. of Millmaster Onyx Corp., 190 Warren St., Jersey City, N.J. 07302.

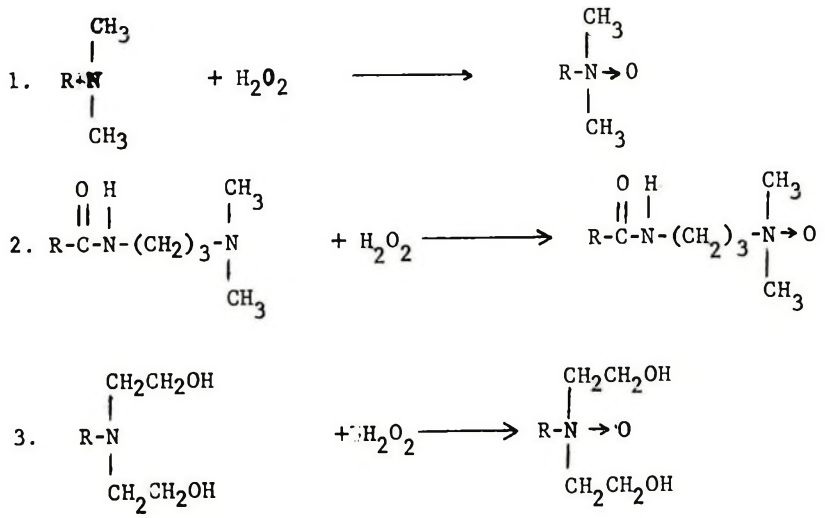

Figure 1. Typical amine oxides

The amine oxides have been found to be useful in a variety of products and areas of application. Dishwashing compounds, shampoos, hair conditioning agents, detergents, shaving creams, electroplating baths, paints, fuel additives, polymerization (1), and laser technology (2) are some examples of their application. In fact, the utility of amine oxides was recognized before World War II. A patent was issued in 1939 disclosing the use of amine oxides for their surfactant properties, either alone or in combination with soap or alkyl sulfates (3).

The amine oxides have been described as having many characteristics which make them attractive to the formulator of cosmetic products. They are reported to be mild to the skin (4), and some recent references disclose synergistic mildness when they are blended with more irritating surface active agents (5). Additionally, the amines oxides are known to impart lubricity and emolliency to the skin. They have antistatic properties, are wetting and foaming agents, have foam-building and stabilizing properties, viscosity-building effects, are resistant to precipitation by hard water salts in solution, and have lime soap dispersing properties (1). Naturally, all properties are not present in every amine oxide structure, but are dependent on alkyl chain length and amine type.

Over the past few years the cosmetic chemist's problems have grown considerably. Government restrictions on the use of certain antimicrobials both as preservatives and as active agents, rigorous toxicity testing, proof of product efficacy claims, need for product composition disclosure, etc., have all

added their burden. To try to ease this burden, we have investigated the use of amine oxides as the primary emulsifier in cosmetic products. Also, we have looked at the quaternary ammonium compounds as preservatives in conjunction with an amine oxide emulsifier in a finished cosmetic product.

Experimental and Results

Formulation Studies

In an attempt to cover a range of products, cosmetic systems made with nonionic and anionic emulsifiers were taken from available literature and prepared. These systems were then made, replacing the nonionic or anionic with amine oxides. Initially, model systems were prepared in order to give some indication as to optimum alkyl chain length for emulsification. We found that the C_{18} alkyl dimethyl amine oxide appeared to give the best results from an appearance and stability viewpoint when compared to the controls.

Stearyl dimethyl amine oxide (C_{18} DMAO),* as commercially supplied, is a 25% active material in aqueous solution with minimal amounts of free amine and peroxide. It is a white paste at room temperature, melting to a viscous, almost water white fluid at 50°C.

Several model systems were prepared to determine whether or not amine oxides could be substituted for conventional emulsifiers which are currently used in practice (Tables I and II). In Table I, the controls 1 and 3 were made up using sorbitan and polysorbate 60 to emulsify stearic acid and cetyl alcohol, respectively. These emulsifiers were replaced by stearyl dimethyl amine oxide at the levels shown, namely 2.5 and 1.0% on an active basis. The resultant emulsions were equivalent to the control systems in appearance and stability.

*Ammonyx SO, Onyx Chemical Co., Div. of Millmaster Onyx Corp., Jersey City, N.J.

Table I
Model Systems Used in Formulation Studies

Ingredient	1	2	3	4
	Per Cent			
Stearic acid	8.0	8.0	...	...
Cetyl alcohol	...	...	5.0	5.0
Sorbitan stearate	2.0	...	2.0	...
Polysorbate 60	3.0	...	3.0	...
C_{18} DMAO	...	2.5	...	1.0
Water	87.0	89.5	90.0	94.0
Total	100.0	100.0	100.0	100.0

Table II
Model Systems Used in Formulation Studies

	5	6	7	8	9	10
Ingredient	Per Cent					
Petrolatum	10	10	...	...	...	...
Isopropyl myristate	...	...	10	10	...	...
Beeswax	...	...	...	...	10	10
Sorbitan stearate	3	...	1.5	...	1.5	...
Polysorbate 60	2	...	3.5	...	3.5	...
C_{18} DMAO	...	1	...	1	...	1
Water	85	89	85	89	85	89
Total	100.0	100.0	100.0	100.0	100.0	100.0

Table III
Formulation for Cleansing Cream

	A	B
Ingredient	Per Cent	
Mineral oil	50.0	50.0
Beeswax	7.0	7.0
Polysorbate 40	2.0	...
PEG 20 sorbitan beeswax	8.0	...
C_{18} DMAO	...	2.5
Water	33.0	40.5
Total	100.0	100.0

Table IV
Formulation for Skin Lotion

	C	D
Ingredient	Per Cent	
Mineral oil	35.0	35.0
Cetyl alcohol	1.0	1.0
Lanolin	1.0	1.0
Polysorbate 80	5.4	...
Sorbitan oleate	2.6	...
C_{18} DMAO	...	2.5
Water	55.0	60.5
Total	100.0	100.0

Table V
Formulation for TEA Stearate Lotion

	E	F
Ingredient	Per Cent	
Lanolin alcohol	5.0	5.0
Acetylated lanolin alcohol	2.0	2.0
Glycerol monostearate	1.0	1.0
Propylene glycol	1.0	1.0
Stearic acid	5.0	...
Triethanolamine	1.0	...
C_{18} DMAO	...	2.5
Water	85.0	88.5
Total	100.0	100.0

Table VI
Formulation for Cleansing Cream
(Anionic)

	G	H
Ingredient	Per Cent	
Mineral oil	52.0	52.0
Beeswax	5.6	5.6
Paraffin wax	5.0	5.0
Cetyl alcohol	3.0	3.0
Petrolatum	8.4	8.4
Sodium lauryl sulfate (30%)	10.0	...
C_{18} DMAO	...	2.5
Water	16.0	23.5
Total	100.0	100.0

In model systems 5–10 (Table II), petrolatum, isopropyl myristate, and beeswax, respectively, were emulsified using 1% active C_{18} DMAO (systems 6, 8, and 10) in place of a combination of sorbitan stearate and polysorbate 60 at a total 5% active level, as shown in systems 5, 7, and 9.

Since the results of this work looked encouraging, we then investigated more complex systems which had combinations of ingredients. Again C_{18} DMAO was used as the sole emulsifier.

In the simple cleansing cream shown in Table III, made from mineral oil and beeswax, C_{18} DMAO (formula B) replaced the polysorbate 40 and polyethlene glycol 20 sorbitan beeswax emulsifiers of formula A. The finished product retained its consistency and stability when compared to the control, and 2.5% active amine oxide was used as compared to 10% active emulsifier level in the control.

A skin lotion (Table IV) containing mineral oil, cetyl alcohol, and lanolin had 8.0% total active of polysorbate 80 and sorbitan oleate in formula C replaced with 2.5% active C_{18} DMAO, as shown in formula D. In this system we also found that the active level of amine oxide could be lowered to 1% while still retaining stability.

In Table V, formula E is a TEA stearate lotion containing lanolin alcohol, acetylated lanolin alcohol, and glycerol monostearate. As shown in formula F we again used 2.5% active C_{18} DMAO in place of the soap.

Formula G in Table VI is an anionic cleansing cream in which 3% active sodium lauryl sulfate was used to emulsify mineral oil, beeswax, paraffin wax, cetyl alcohol, and petrolatum. Again, 2.5% active C_{18} DMAO was substituted and an excellent emulsion resulted as listed in formula H.

In preparing the control systems, conventional manufacturing procedures were followed. We blended the oil phase ingredients, heating until all solid constituents were melted, and maintained the temperature at 65°C. The water phase was heated to 70°C and added to the oil phase. Nonionic emulsifiers were included in the oil phase. Where soap prepared *in situ* was the surfactant, the stearic acid was included in the oil phase and triethanolamine in the water phase. Sodium lauryl sulfate was included in the water phase in the one system in which it was used. In working with the amine oxides we added the amine oxide to the aqueous phase, and heated it to 65°C to melt and solubilize the amine oxide. The water phase was then added to the oil phase. The pH was adjusted to 7.0 in all systems except the soap-based controls.

Our work has shown that optimum results are achieved with stearyl dimethyl amine oxide, although there undoubtedly are systems where other alkyl chain lengths or structural types may be best.

It was found that cetyl dimethyl amine oxide had utility, and that blends of lower amine oxides (C_{12}, for example) could be used with C_{18} DMAO to modify properties such as viscosity. These amine oxides can be used in

place of conventional emulsifiers, although some formula modification, for example, viscosity or ingredient level adjustment, may have to be made to bring physical characteristics to match those of the control. When starting from a new product concept however, the use of amine oxides presents new areas of investigation.

Preservation Studies

It is well known that most cosmetic preparations are subject to microbiological attack. It is also documented that oil-in-water emulsions are particularly susceptible to contamination; and those prepared using nonionic emulsifiers are exceptionally difficult to preserve. Even well-accepted preservative compounds are occasionally rendered ineffective when sudden massive contamination occurs (6). When this happens in a finished product, recall is necessitated, company and individual reputations are tarnished, and thousands of dollars are lost.

In the past, the use of quaternaries as preservatives for cosmetic products was severely limited. These compounds are generally incompatible with anionics and the amount of quat to nonionic in nonionic systems is so small that the quaternary activity is inhibited through nonionic interference. Experience has shown us that the amine oxides do not markedly inhibit quaternary performance and two patents were issued on the basis of this work (7, 8).

The skin lotion shown in Table IV was known from previous work to be extremely susceptible to microbiological attack. This lotion was prepared consisting of mineral oil, cetyl alcohol, and lanolin. Polysorbate 80 and sorbitan oleate, or C_{18} DMAO were used as the emulsifiers. Both systems were prepared with 0 and 2000 ppm of various quaternaries and tested for preservation capabilities. Testing was carried out using the following procedure.

A microbial background count was made on each formulation immediately after its preparation and prior to preservation efficacy testing. TGE Agar pour plates were prepared with 1-ml aliquots of a $\frac{1}{10}$ and $\frac{1}{100}$ dilution of formulation. Background pour plates were incubated at 37°C for 72 hours before colony counts were made. Formulations with background counts of 1000 organisms/ml or less were adjudged adequate for preservation efficacy testing, which was carried out as follows:

A 100-gram aliquot of each formulation was aseptically added to a sterile 8-ounce wide-mouth jar; three replicates were so prepared from each formulation. Each jar was then inoculated with 5 ml of pooled $\frac{1}{10}$ sterile nutrient broth cultures of *Staphylococcus aureus* (ATCC #6538), *Escherichia coli* (ATCC #11229), *Pseudomonas aeruginosa* (ATCC#10145), *Enterobacter aerogenes* (ATCC #13048), *Proteus mirabilis* (ATCC #9921), and *Bacillus cereus* (ATCC #6462). In this matter, an initial inoculum of 10^6–10^7 bacteria/ml of inoculated jar content was obtained. The inoculated jars were stored at 25°C.

At weekly intervals following inoculation, each jar was plated *via* TGE Agar pour plates to determine the numbers of viable bacteria present. Initial weekly platings were made at 10^{-1} and tenfold serial dilutions therefrom to 10^{-6}. At subsequent weekly intervals, jars of untreated control formulations were plated only at higher serial dilution levels of 10^{-4}, 10^{-5}, and 10^{-6}. Jars of experimental treated formulation demonstrating preservation efficacy were plated at lower dilution levels of 10^{-1}, 10^{-2}, and 10^{-3}. All the plates were incubated at 37°C for 72 hours prior to colony counts for viable bacteria present in the jars.

At the four intervals following inoculation, those jars of treated samples which showed zero counts at the 10^{-1} dilution were reinoculated, incubated, and plated at weekly intervals. All jars were incubated and examined weekly for a total of 8 weeks following the intial inoculation. Treated formulations which demonstrated the absence of viable bacteria at the 10^{-1} dilution level over an 8-week incubation period and including the reinoculation challenge were adjudged to contain efficacious preservation systems.

Various structural types of commercially available quaternaries were tested for suitability in this application. The first (Quaternary I), shown in Fig. 2, is a 50:50 mixture of *n*-alkyl dimethyl benzyl ammonium chloride and *n*-alkyl dimethyl ethylbenzyl ammonium chloride.* Here the alkyl distribution on the benzyl quat is 5% C_{12}, 60% C_{14}, 30% C_{16}, and 5% C_{18}; on the ethyl benzyl quat it is 68% C_{12}, 32% C_{14}.

Figure 3 shows the structural formula for diisobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride (Quaternary II).† The next quat (Fig.

*BTC 2125M, Onyx Chemical Co., Div. of Millmaster Onyx Corp., Jersey City, N.J.
†Hyamine 1622, Rohm & Haas Co., Philadelphia, Pa.

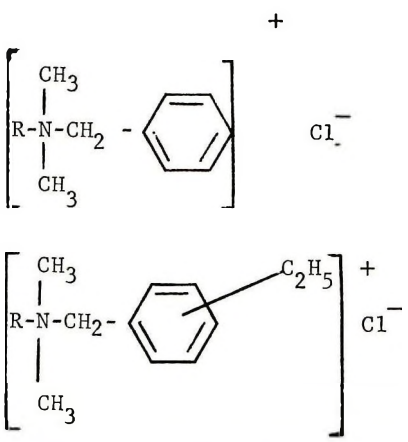

Figure 2. Structural formula for Quaternary I

4) is an 80:20 mixture of methyldodecylbenzyl trimethyl ammonium chloride and methyldodecylxylylene bis (trimethyl ammonium chloride) (Quaternary III).*

The last quat tested (Quaternary IV) is a dialkyl structure, octyldodecyl dimethyl ammonium chloriden,† and is shown in Fig. 5.

*Hyamine 2389, Rohm & Haas Co., Philadelphia, Pa.

†BTC 812, Onyx Chemical Co., Div. of Millmaster Onyx Corp., Jersey City, N.J.

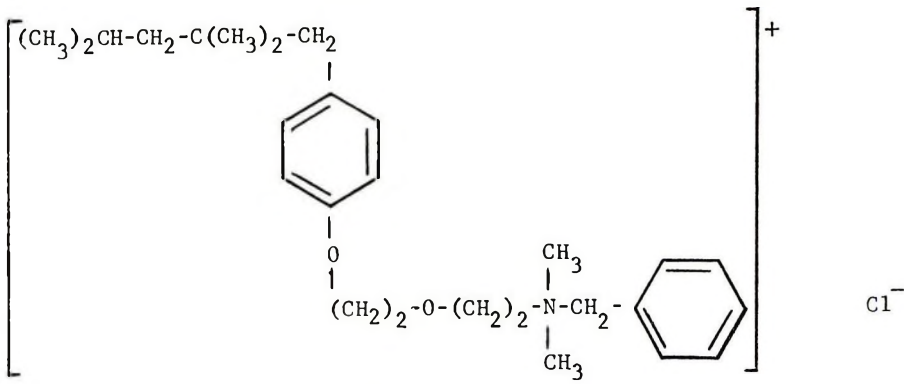

Figure 3. Structural formula for Quaternary II

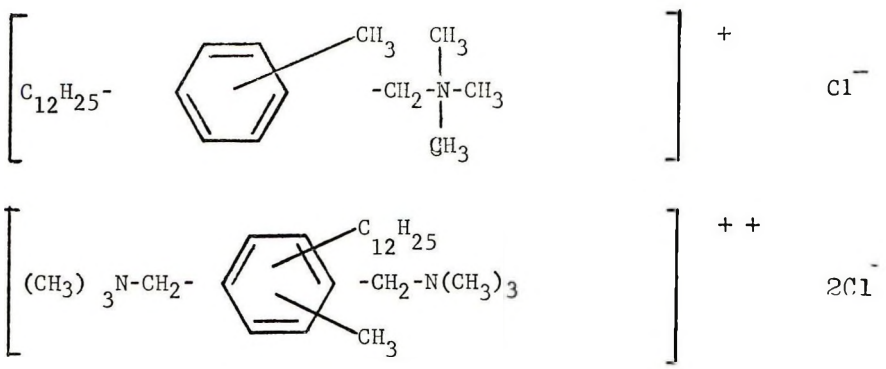

Figure 4. Structural formula for Quaternary III

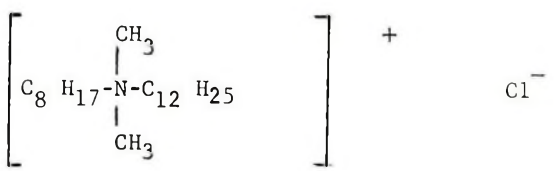

Figure 5. Structural formula for Quaternary IV

Table VII
Preservation Results Obtained with the Four Quarternaries Tested

Sample	ppm Quat	No. of Bacteria ($\times 10^6$)/ml			
		1 Week	4 Weeks	5 Weeks	8 Weeks
		Quaternary I			
Nonionic	0	18	106	109	121
Nonionic	2000	9	80	26	62
C_{18} DMAO	0	74	104	89	82
C_{18} DMAO[a]	2000	(0)[b]	(0)[b]	(0)[b]	(0)[b]
		Quaternary II			
Nonionic	0	22	94	106	94
Nonionic	2000	15	92	56	43
C_{18} DMAO	0	24	108	89	124
C_{18} DMAO[a]	2000	(0)[b]	(0)[b]	(0)[b]	(0)[b]
		Quaternary III			
Nonionic	0	20	92	79	76
Nonionic	2000	12	40	29	20
C_{18} DMAO	0	16	102	116	90
C_{18} DMAO[a]	2000	(0)[b]	(0)[b]	(0)[b]	(0)[b]
		Quaternary IV			
Nonionic	0	16	142	167	94
Nonionic	2000	4	61	67	54
C_{18} DMAO	0	18	92	114	102
C_{18} DMAO[a]	2000	(0)[b]	(0)[b]	(0)[b]	(0)[b]

[a] Sample reinoculated after 4 weeks.
[b] Determined from 10^{-1} dilution.

Table VII illustrates the preservation results obtained with the four quarternaries tested. With the 50:50 blend of alkyl dimethyl benzyl and alkyl dimethyl ethylbenzyl ammonium chlorides (Quaternary I), both nonionic samples, without and with quaternary show heavy growth as does the control C_{18} DMAO sample. The C_{18} DMAO sample with 2000 ppm quaternary shows no growth, even when reinoculated after four weeks. It should be pointed out that testing was done on a weekly basis but for the sake of legibility only the 1st, 4th, 5th, and 8th weeks are shown.

The results with diisobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride (Quaternary II) are similar to the previous test. Heavy growth is evident in the two nonionic systems and the control amine oxide system. The other amine oxide system with 2000 ppm quat shows no growth, even after reinoculation.

With Quaternary III, an 80:20 blend of methyldodecylbenzyl trimethyl ammonium chloride and methyldodecylxylylene bis(trimethyl ammonium chlo-

ride), the results follow a familiar pattern. The two nonionic samples, without and with quat, and the control C_{18} DMAO system have growth. The quat-treated C_{18} DMAO sample shows an absence of microorganisms before and after reinoculation.

With octyldodecyl dimethyl ammonium chloride (Quaternary IV) results are the same. Three systems, both nonionic and untreated amine oxide, show growth. The amine oxide formula with the quaternary has no growth after reinoculation.

Although not shown, more recent work in this laboratory has indicated that a second system containing lanolin alcohol, acetylated lanolin alcohol, glycerol monostearate, and C_{18} DMAO was preserved with as little as 500 ppm total of a 50:50 mixture of alkyl dimethyl benzyl and alkyl dimethyl ethylbenzyl ammonium chlorides (BTC 2125M). A third system with mineral oil, stearic acid, glycerol monostearate, cholesterol, polyoxyethylene (24) cholesterol ether, and C_{18} DMAO emulsifier was also protected from microbiological attack with 1000 ppm of the same quaternary. Additionally, testing against fungi, specifically *Aspergillis niger* and *Penicillium notatum* has been done. Two thousand ppm of a 50:50 mixture of alkyl dimethyl benzyl and alkyl dimethyl ethylbenzyl ammonium chlorides in the test system has given protection for 8 weeks, including a reinoculation after 4 weeks.

From the preceding work it was determined that readily available, relatively inexpensive quaternaries can be used as effective preservatives for amine oxide emulsified products.

Irritation Studies

It is essential that the formulators of cosmetics avoid substances in their products which would be harmful to the user. Therefore, we tested the skin lotion for eye and primary skin irritation. All eye and primary skin irritation studies were carried out according to the Draize methods.

The product was evaluated with and without quaternary, the quat being a 50:50 mixture of alkyl dimethyl benzyl ammonium chloride and alkyl dimethyl ethylbenzyl ammonium chloride (Quaternary I) at a total level of 2000 ppm active. The formulations are shown in Table VIII.

The results (Table IX) indicated that the control system without quat did not cause irritation, either with or without washout, in any of the rabbit eyes. Table X results showed that the system with 2000 ppm quaternary did produce a very mild, transient irritation, some redness of the conjunctivae, clearing on the second day, in two of the three unwashed eyes. There was no involvement of the cornea or iris. The third rabbit showed no irritation whatsoever. The eyes that were washed 4 seconds after product installation did not show any irritation at all.

The results of primary skin irritation studies (Table XI) showed that without quaternary there was some erythema and eschar formation after 24 and

Table VIII
Skin Lotion Formulation for Irritation Studies

Ingredient	Control	2000 ppm quat
	Per Cent	
Mineral oil	35.0	35.0
Cetyl alcohol	1.0	1.0
Lanolin	1.0	1.0
C_{18}DMAO	2.5	2.5
Quaternary (100%)	...	0.2
Water	60.5	60.3
Total	100.0	100.0

Table IX
Eye Irritation Results for Control System (No Quat)

	Days After Installation				
	1	2	3	4	7
I. Cornea					
A. Opacity	0	0	0	0	0
B. Area of cornea involved	0	0	0	0	0
Score = A × B × 5 max = 80	0 × 0 × 5 = 0				
II. Iris					
A. Values	0	0	0	0	0
Score = A × 5 max = 10	0 × 5 = 0				
III. Conjunctivae					
A. Redness	0	0	0	0	0
B. Chemosis	0	0	0	0	0
C. Discharge	0	0	0	0	0
Score = (A × B × C) × 2 max = 20	(0 + 0 + 0) × 2 = 0				

72 hours, although no edema formation was noted, giving a primary irritation score of 4. With quaternary, there was erythema and eschar formation after 24 and 72 hours, and edema formation on intact and abraded skin after 24 hours, giving a primary skin irritation score of 6.

Further toxicological tests led us to the conclusion that mineral oil by itself was the cause of this irritation. The lotion was then remade, unpreserved and preserved, with 2000 ppm quat, using an ester, isopropyl palmitate, in place of the mineral oil (Table XII).

Results of skin irritation testing using this system showed no irritation at all on intact or abraded skin (Table XIII). Also, eye irritation testing according to the Draize Method indicated that the product with isopropyl palmitate was not an irritant either with or without quaternary.

Table X
Eye Irritation Results for System with 2000 Quaternary

	Days After Installation				
	1	2	3	4	7
I. Cornea					
A. Opacity	0	0	0	0	0
B. Area of cornea involved	**0**	**0**	0	0	0
Score = A × B × 5 max = 80	0 × 0 × 5 = 0				
II. Iris					
A. Values	0	0	0	0	0
Score = A × 5 max = 10	0 × 5 = 0				
III. Conjunctivae					
A. Redness	**1**	**0**	0	0	0
B. Chemosis	0	0	0	0	0
C. Discharge	**0**	**0**	0	0	0
Score = (A × B × C) × 2 max = 20	(1 + 0 + 0) × 2 = 2				

Table XI
Results of Primary Skin Irritation Studies

		Av. Exposure Value	
	Exposure Time (Hours)	No Quat	2000 ppm Quat
Erythema and eschar formation			
Intact skin	24	1	1
	72	1	1
Abraded skin	24	1	1
	72	1	1
Subtotal		4	4
Edema formation			
Intact skin	24	0	1
	72	0	0
Abraded skin	24	0	1
	72	0	0
Subtotal		0	2
Primary irritation		4	6

Commercially available stearyl dimethyl amine oxide of good quality, namely that containing low peroxide and free amine (Ammonyx SO), was tested by itself at 5% active for skin and eye irritation. These results indicated that this material is neither a primary skin irritant nor an occular irritant.

Table XII
Formulation for Modified Skin Lotion

Ingredient	Control	2000 ppm Quat
	Per Cent	
Isopropyl palmitate	35.0	35.0
Cetyl alcohol	1.0	1.0
Lanolin	1.0	1.0
C_{18} DMAO	2.5	2.5
Quaternary (100%)	...	0.2
Water	60.5	60.3
Total	100.0	100.0

Table XIII
Results of Primary Skin Irritation Studies with Modified Skin Lotion

		Av. Exposure Value	
	Exposure Time (Hours)	No Quat	2000 ppm Quat
Erythema and eschar formation			
Intact skin	24	0	0
	72	0	0
Abraded skin	24	0	0
	72	0	0
Edema formation			
Intact skin	24	0	0
	72	0	0
Abraded skin	24	0	0
	72	0	0
Primary irritation		0	0

Summary

In conclusion, we have shown that amine oxides can successfully be used as emulsifiers in cosmetic products. It is possible that one amine oxide can replace a number of surfactants in a specific system and perhaps at a lower total active level. The amine oxides do not present a problem from a toxicological viewpoint, and when formulated with inexpensive, readily available quaternary ammonium compounds as preservatives, freedom from microbiological attack is achieved. These systems, when properly formulated, are innocuous from an irritation potential. We hope that the information presented will be helpful to the formulator of cosmetic products and opens new areas for investigation.

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Occurrence of Pseudomonas Aeruginosa and Staphylococcus Aureus in the Orbital Area

JOHN F. McCONVILLE, B.S., and DAVID W. ANDERSON, Jr., Ph.D.*

Synopsis—PSEUDOMONAS AERUGINOSA and STAPHYLOCOCCUS AUREUS were recovered in large numbers from the ORBITAL AREA of two female subjects. Further investigation of these two subjects revealed that the source of these microorganisms appeared to be an INFECTION of the EXTERNAL AUDITORY CANAL. Medical diagnosis confirmed the existence of chronic ear infection in both subjects caused by *P. aeruginosa* in one case and S. *aureus* in the other.

Introduction

An investigation of the flora of the face and orbital area of selected subjects in Los Angeles, Calif. (1) showed large numbers of pathogens to be present in the orbital area of two subjects. Further study of these two subjects is reported here.

Wilson *et al.* (2), in a study on microbial contamination of eye cosmetics, pointed out that pathogens and other microorganisms are a potential hazard to the injured eye. The case histories described here indicate that infections of the ear may be a source of pathogens for the orbital area.

Material and Methods

The external eye areas of 26 subjects were swabbed to determine the microbial flora by the method previously reported by McConville and Anderson (1). Two subjects were found to have large numbers of S. *aureus* and *P. aeruginosa*, respectively, around the orbital area. To determine the extent to which these organisms had populated the facial area, both subjects were swabbed weekly around the eyes, on the cheeks and forehead, and in and around the ears for a period of several months.

* Max Factor & Co., 1655 N. McCadden Place, Hollywood, Calif. 90028.

Results

Subject 20 was female, age 31, and used heavy application of eye and face make-up regularly. Swabs of her eye area yielded approximately 6×10^5 microorganisms per in^2. The great majority of these bacteria were mannitol-fermenting, coagulase-positive, DNase-positive *Staphylococcus*. Questioning of this subject revealed chronic untreated ear problems. She complained of itching and discharge from both ears. Swabs taken of the external auditory canal gave confluent growth when streaked on Trypticase Soy Agar (TSA) (BBL). Subsequent gram stains and differential tests showed the organisms to be coagulase-positive *Staphylococcus aureus*.

Swabs of the subject's cosmetics recovered coagulase-positive S. *aureus* when tested immediately after use. But when the cosmetics were retested after 48 hours, no recovery of the bacteria was found.

The subject was treated for Staphylococcal infection of the middle ear by a physician. After one month of treatments the infection disappeared. Swabs of her facial area then showed S. *epidermidis*, *Micrococcus*, and *Bacillus* species. S. *aureus* was not recovered from either her ears or her face.

Subject 22 was female, age 29, who used eye and face make-up regularly with moderate application. Swabs of her upper and lower eyelids yielded approximately 2×10^4 organisms per in^2. Gram stains showed a mixed flora of gram-positive cocci and gram-negative rods. The gram-negative rods were the predominant organisms. Subsequent isolation and identification showed the gram-positive cocci to be *Staphylococcus epidermidis* and the gram-negative rods to be a mixture of fluorescent and nonfluorescent strains of *Pseudomonas aeruginosa*. Further swabbing showed that the pseudomonads could be recovered from her cheeks and external auditory canal.

This subject also reported an untreated ear problem. She had been experiencing itching and occasional discharge from both ears for several years. Swabs of the external auditory canal yielded confluent growth of *P. aeruginosa* when streaked on TSA.

Swabs of the subject's cosmetics were negative when streaked on TSA. The subject underwent medical treatment and the condition appeared to improve. *P. aeruginosa* was no longer recovered from her face or ears after repeated swabbings.

Approximately 6 weeks later, the subject again reported itching and discharge from her right ear. Swabs of the external auditory canal gave confluent growth of *P. aeruginosa* when streaked on TSA. Swabs of her facial area did not reveal the presence of this organism. Further treatment by a physician alleviated the condition. However, 5 weeks after discontinuing medication, the symptoms reappeared.

The infection has persisted for 15 months with only intermittent relief during and shortly after the application of otic medication. The extent to which the infection recurs depends upon the time elapsed between treatments.

Discussion

We have reported two case histories in which large numbers of potential pathogens were recovered from the face and orbital area with no detectable ocular infection. Kuehne *et al.* (3) report an instance where the mascara of a women with a corneal ulcer caused by *Fusarium solani* contained the same fungus. The implication was that the mascara was the source of the fungus. Neither the time elapsed between consumer use and microbial testing of the mascara, nor the number of fungi per gram of product was reported.

Immediately after subject 20 used her eye shadow, *S. aureus* was recovered from the product. Forty-eight hours later the organisms could not be recovered by plate count or enrichment which indicated the preservative system was bactericidal.

If this subject had ocular infection, and her eye cosmetics were sampled immediately after use, it might be interpreted that her eye make-up was the cause of the infection. Actually, the subject's ear infection was the source of the *Staphylococcus* population of her orbital area. Subsequent addition of these organisms to the orbital area from her cosmetics may not have been more significant and possibly less hazardous than rubbing her eyes with a finger.

Pseudomonads have been implicated in corneal infections (4, 5). The eye area of subject 22 contained thousands of these organisms per in^2. A corneal abrasion could have occurred from a tweezer, a cosmetic applicator, or a fingernail, possibly resulting in an infection. If subject 22 scratched her eye with a cosmetic applicator allowing entry of *P. aeruginosa* already present on the orbital area, the cosmetic manufacturer may have been implicated, particularly if the organism was found in the cosmetic, even in low numbers.

Eye cosmetics should have adequate preservative systems to prevent proliferation of microorganisms which are transferred from the eye area to the cosmetic during use. The microbial content of used eye cosmetics has been reported (2, 5–10). We have shown (7, 9) that eye cosmetics collected from users in Los Angeles were of good microbial quality. However, low numbers of microorganisms in an eye cosmetic may not be significant because of the small amount applied. The CTFA recommended microbiological limits (11) for eye cosmetics is 500 microorganisms per gram of product. Microorganisms should not proliferate in an adequately preserved product beyond this microbial limit density (8, 9). An eye liner with the microbial load of 500 per gram, would transfer 2 microorganisms per applicator to the eyelid (9). The natural flora of the eyelid far exceeds this number.

From 1952 to 1968, Locatcher-Khorazo and Gutierrez (12) determined the bacterial flora of noninfected eyes of 10,271 individuals 1 to 90 years of age. *Staphylococcus aureus* was found on the eyelid margins and conjunctivas of more than 30% of the subjects. Other pathogens recovered included *Escherichia coli* and several species of *Klebsiella* and *Proteus*.

On the basis of the findings reported here, we suggest that in future cases of ocular infections the source of the pathogens be fully investigated. In conjunction with testing the subject's cosmetics, the investigator should consider swabbing areas of the face, hands, forehead, cheeks, and particularly the external auditory canal.

Conclusion

We have shown that the presence of pathogens around the orbital eye area may be related to chronic ear infections. We have also shown (9) that if an eye product is adequately preserved, these organisms will not proliferate.

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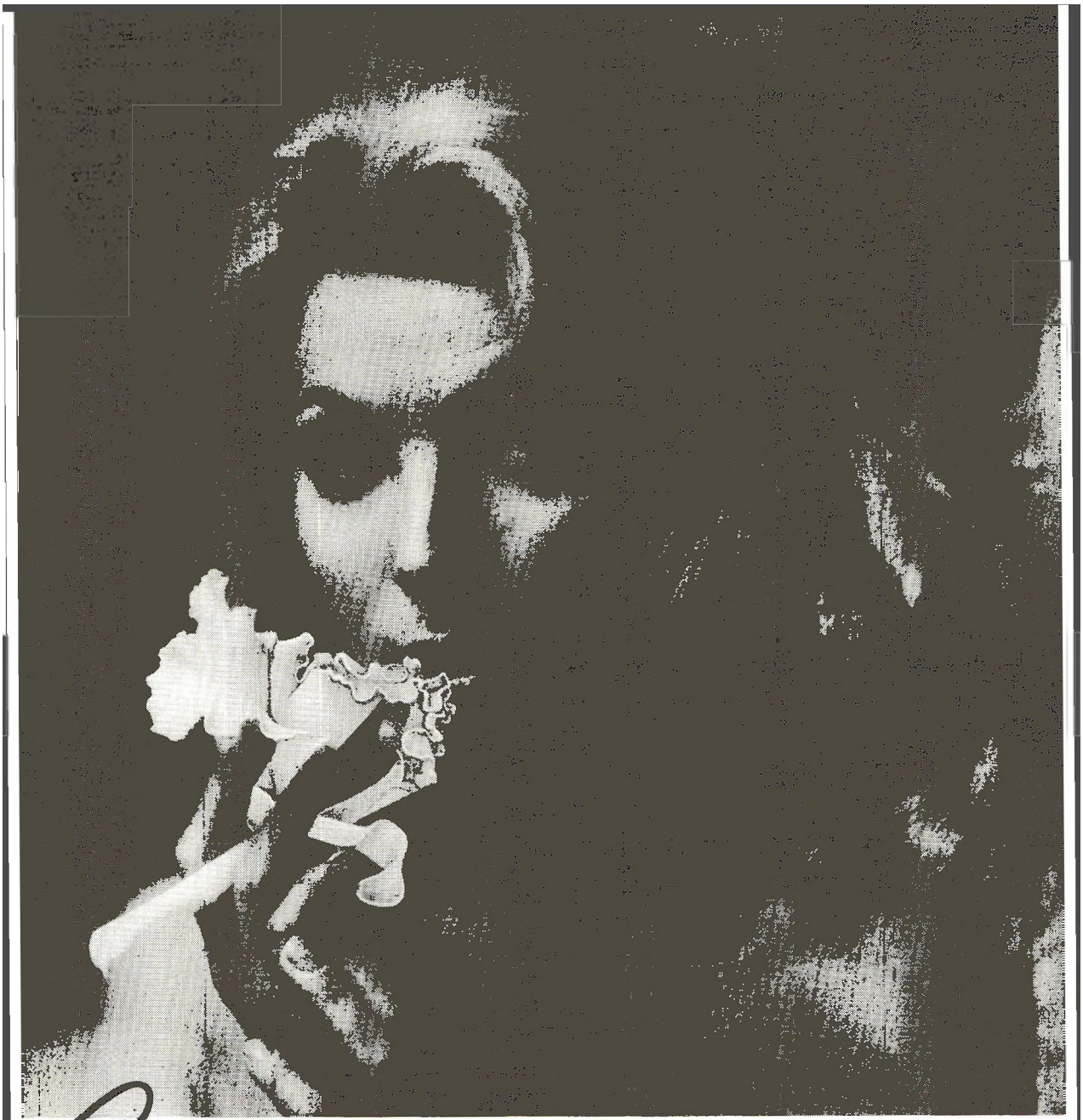

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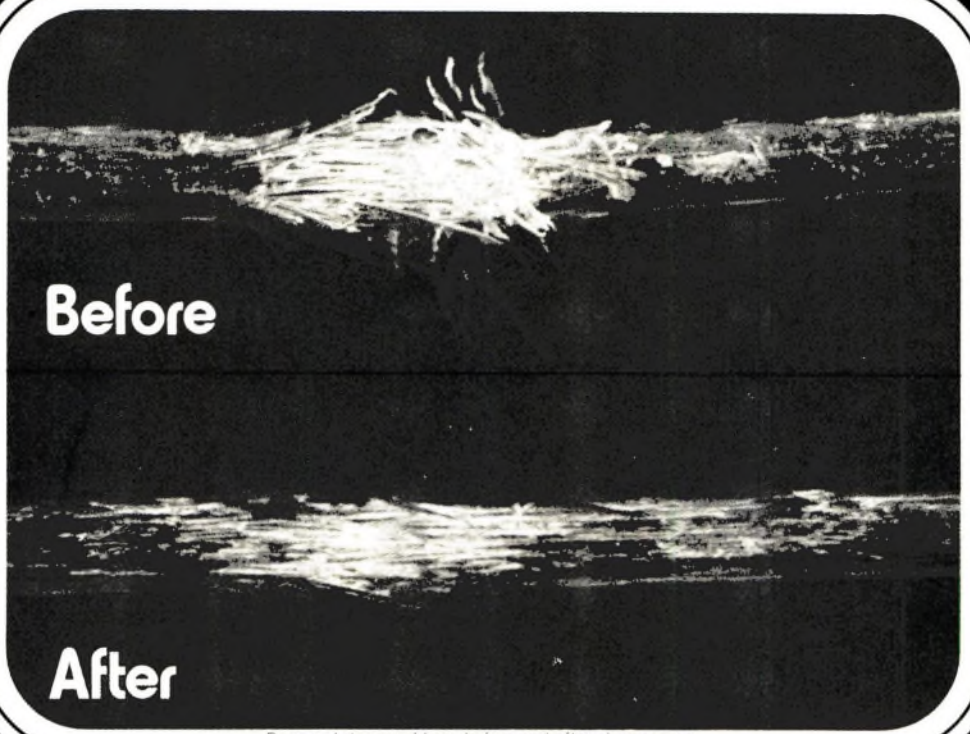

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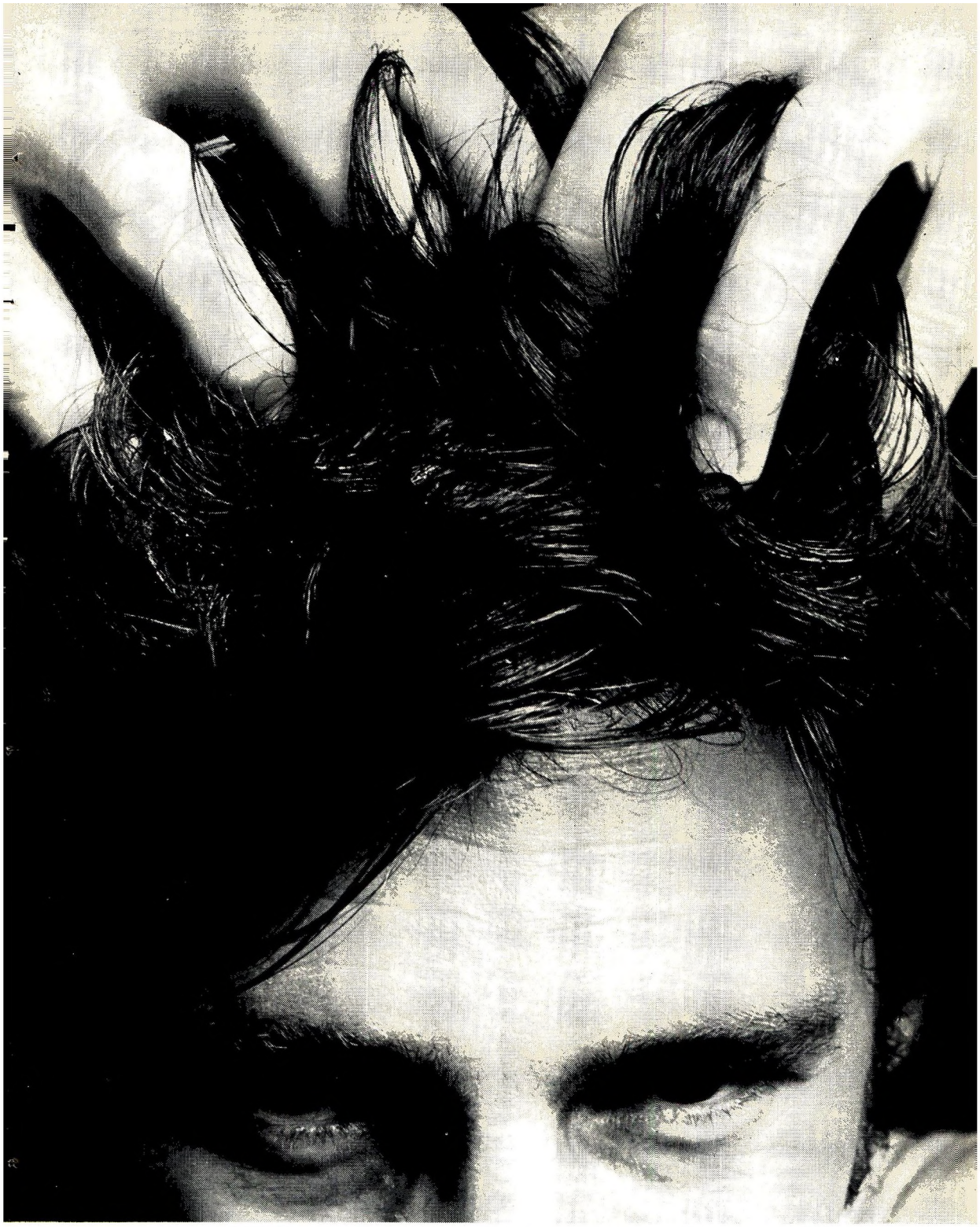

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