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Journal of the Society of Cosmetic Chemists

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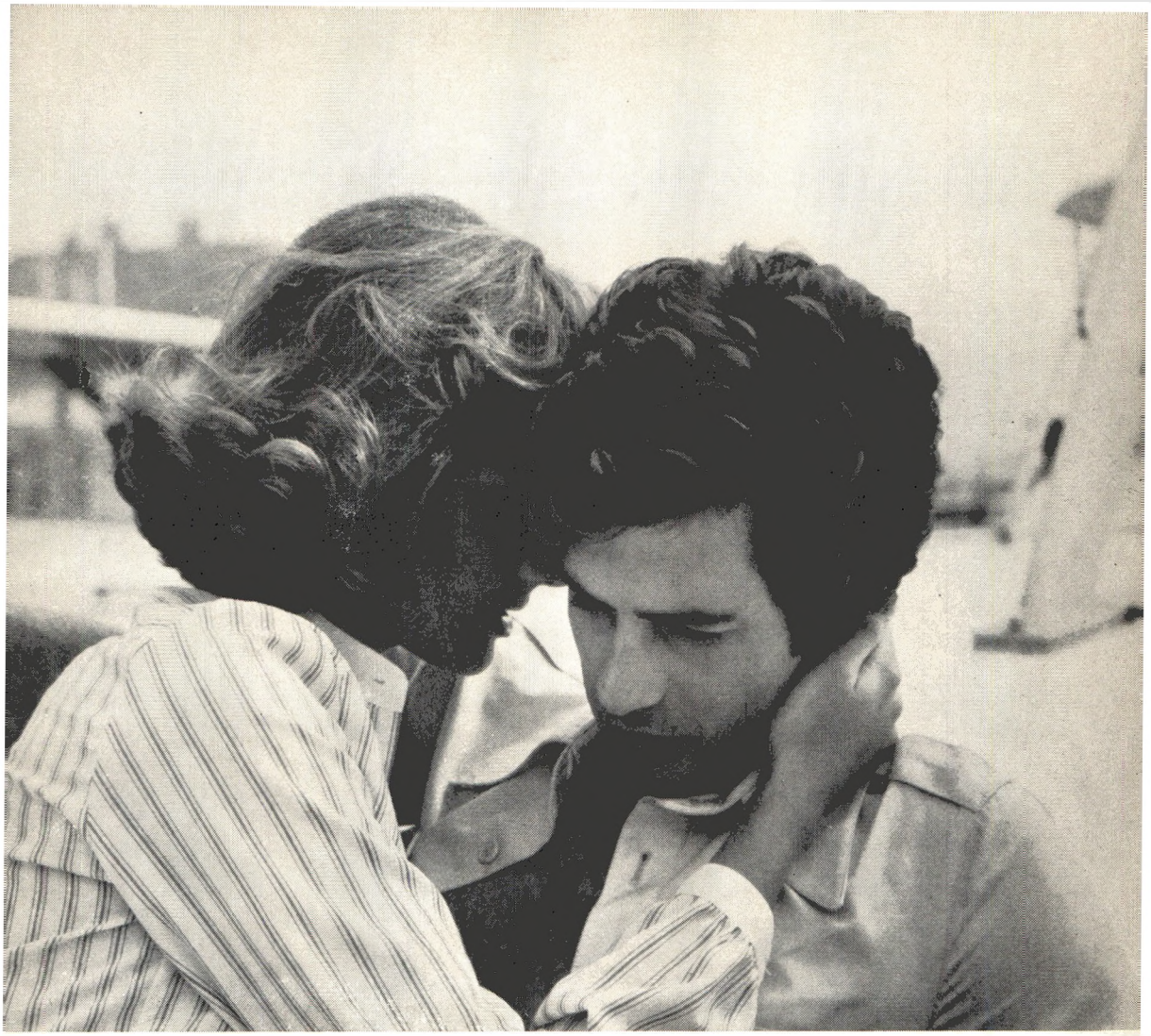

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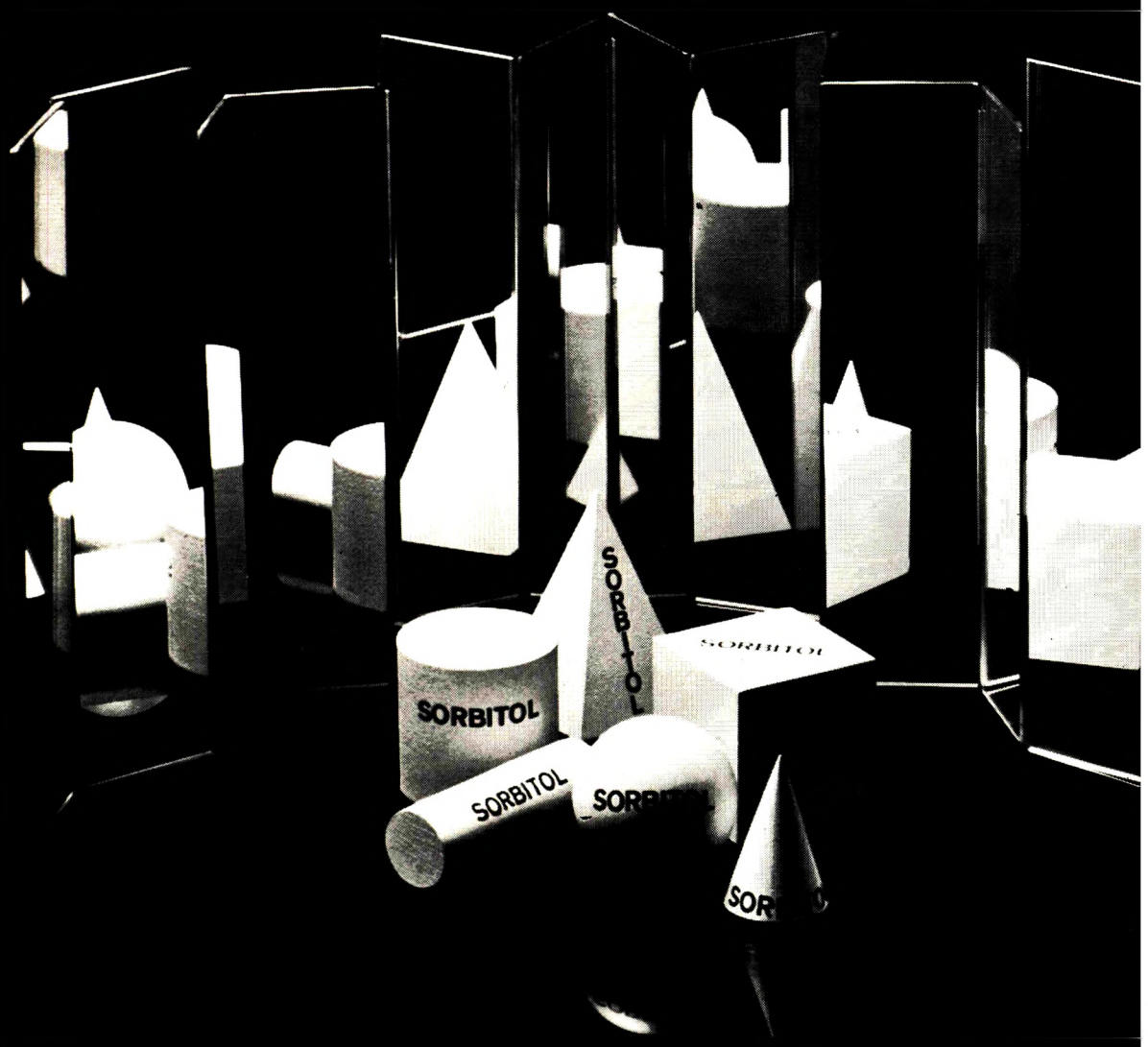

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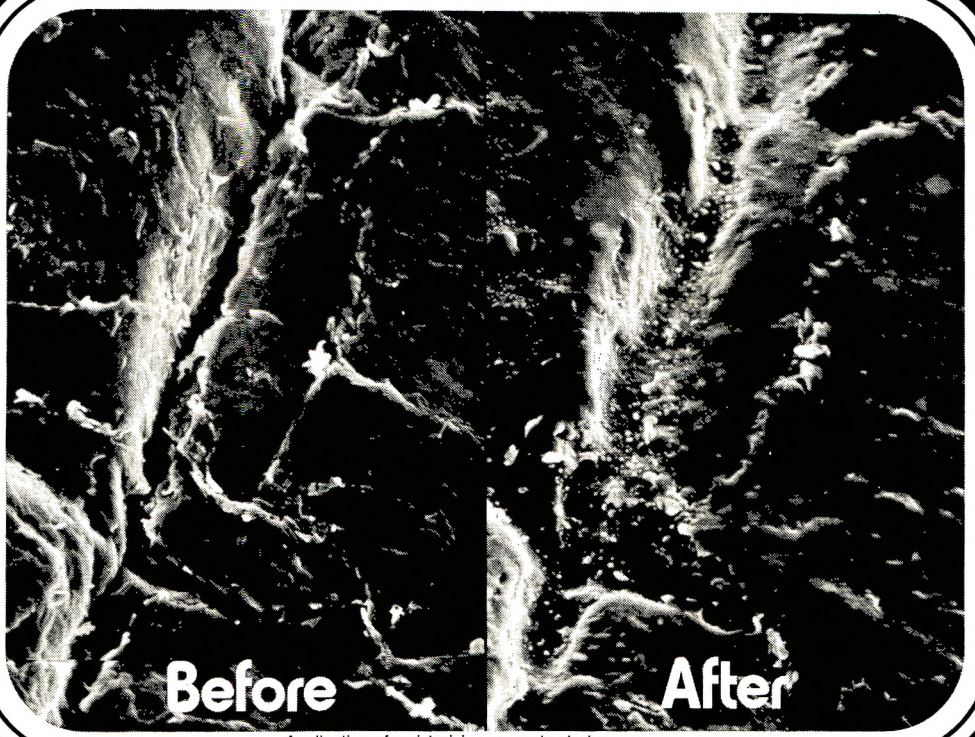

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Observations on the cutting of beard hair: Donald E. Deem and Martin M. Rieger. *Journal of the Society of Cosmetic Chemists* **27**, 579 (December 1976)

Synopsis—A device is described which permits measurement of the force required to cut a beard hair fiber under a variety of conditions. Studies with this device show that the force required to cut wet beard fibers with commercial razor blades is about 65 per cent less than that of dry fibers. Beard hair is almost completely hydrated by exposure to water for about 2 minutes at room temperature, and this hydration is accelerated by an increase in temperature. The force required to cut a beard hair increases with increasing fiber cross-sectional area, but this correlation is not perfect. The force required to cut beard hair is not lowered below that in water by the presence of a wetting agent, a shaving cream, or a soap solution. The force required to cut wet beard hair with a razor blade is lowered significantly by very severe attack on the fiber. On the other hand, the force required to cut beard hair increases as the rate of the blade travel increases.

Fluoride ions in toothpastes: M. O. Schmitz-Masse, M. Hanocq, and M. Herpol-Borremans. *Journal of the Society of Cosmetic Chemists* **27**, 593 (December 1976)

Synopsis—In the first part of this study the conditions for the separation of fluoride ion in toothpastes are examined. The method which depends on the codistillation of hexafluorosilicic acid with super-heated steam, which is generated in a suitable apparatus, has proved itself useful. The procedure is independent of the method of preparation of the toothpaste and of the type fluoride derivative present in the product. The second part is a comparison of two assay procedures. The first one—a spectrophotometric procedure—utilizes formation of a complex between cerium, alizarin, Complexon, and fluoride. The other—a potentiometric assay—depends on the use of a lanthanum fluoride membrane electrode. From the point of view of sensitivity, reproducibility, and precision the two methods are comparable.

Physical techniques for assessing skin moisturization: Alfred J. Quattrone and Karl Laden. *Journal of the Society of Cosmetic Chemists* **27**, 607 (December 1976)

Synopsis—An overview is presented of some physical techniques currently available for use in skin moisturization studies. Water soaking of unmodified versus ether-extracted stratum corneum, for example, causes a marked alteration in the biomechanical properties of these tissues (i.e., swelling capacity, elastic modulus, relaxation function, and work index). Differences in moisture binding properties as measured by gravimetric and scanning calorimetric analyses of the tissue at various relative humidities are related. The correlation of changes in these traits with changes in the pliability and strength of corneum tissue and its capacity to retain moisture is discussed. Criteria for judging dry versus hydrated skin *in vivo* are also reviewed through the utilization of transpirometry, photography, and scanning electron microscopy (SEM). Analysis via these techniques of the effect of humectants and occlusive oils on water retention within skin is presented.

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Observations on the Cutting of Beard Hair

DONALD E. DEEM, M. S. and MARTIN M. RIEGER, Ph.D.*

Synopsis: A device is described which permits measurement of the force required to CUT a BEARD HAIR FIBER under a variety of conditions. Studies with this device show that the force required to cut wet beard fibers with commercial razor blades is about 65 per cent less than that of dry fibers. Beard hair is almost completely hydrated by exposure to water for about 2 minutes at room temperature, and this hydration is accelerated by an increase in temperature. The force required to cut a beard hair increases with increasing fiber cross-sectional area, but this correlation is not perfect. The force required to cut beard hair is not lowered below that in water by the presence of a wetting agent, a shaving cream, or a soap solution. The force required to cut wet beard hair with a razor blade is lowered significantly by very severe attack on the fiber. On the other hand, the force required to cut beard hair increases as the rate of blade travel increases.

Introduction

Since the classical study by Hollander and Casselman (1) very few publications have dealt with the physics of shaving or the cutting of beard hair. Their study was based mainly on subjective evaluations by shaving panelists and included only minimal objective information obtained by mechanical testing (creep measurements) and microscopic examination.

The present study was designed to measure the force required to cut single beard fibers with commercial razor blades under conditions approximating the *in vivo* situation.

No attempt was made to analyze the problem of hair cutting mathematically. In accordance with the classical concepts of Dupré (2) one approach might be to equate the work-to-cut to the work to create two new cross-sectional hair surfaces plus frictional effects. Another approach would utilize a mathematical treatment of the elasticity of anisotropic materials, which according to Muskhelishoili (3) is extremely complex.

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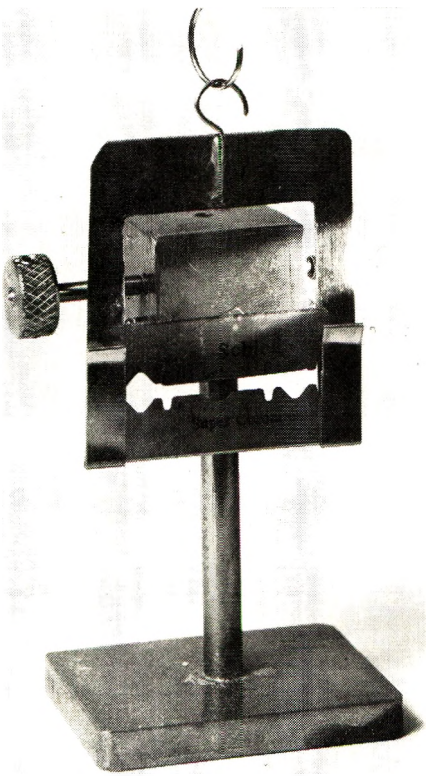
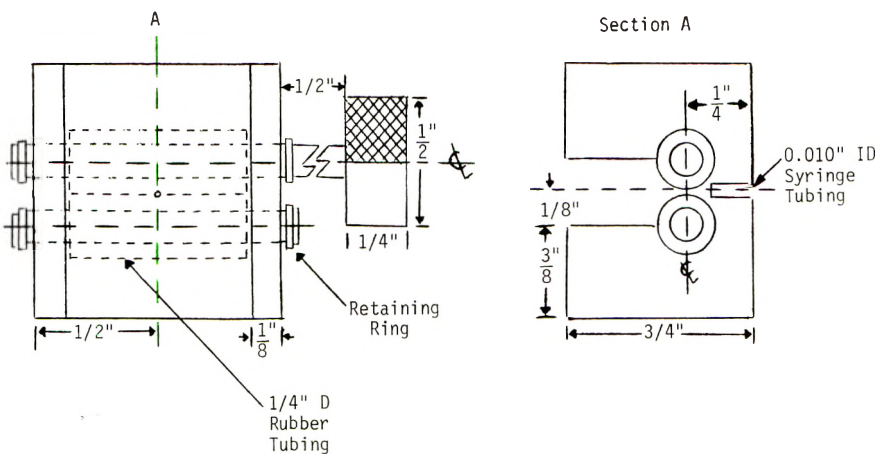


(a)



(b)

Figure 1. Beard Cutting Jig: (a) Overall view; (b) construction details. Jig is constructed of brass and supported with brass rod and base. Rollers are ⅛ in. i.d. rubber tubing with 1/16 in. walls. Syringe tubing is set into hole flush with front face

Experimental

Preliminary investigations involved selection of the most useful geometry for cutting fibers. Anvil cutting, i.e., forcing a blade through a fiber, placed on a compression cell of an Instron® tester,* was found to require very precise positioning of the blade to prevent premature contact of the blade with the cell. Catenary cutting, i.e., pulling a blade through a fiber held firmly at both ends, but without any tension, was found to produce scraping along the fiber before cutting unless the blade was centered exactly.

The method finally selected was cantilever cutting in which the beard fiber is allowed to protrude through a hole from the face of a brass jig (Fig. 1) placed on the crosshead of an Instron tester. The jig consists of a brass face, into which a 0.010-in. i.d. syringe tube bushing was inserted, and a set of rubber rollers to advance the fiber rapidly between cuts. The cutting blade was placed in a stirrup which was hung from the low range transducer (full scale 2 to 50 g) of a model TM Instron Tester. The fiber is cut by movement of the jig relative to the stationary blade. The angle between the blade and the face of the jig is approximately 3 degrees in order to prevent the blade from moving away from the face of the fixture. During and between cuts, the fibers were maintained "in" the solution of interest by pumping the solution from a constant temperature bath through a capillary pipette which directed the stream onto the fiber and into the syringe tubing in the face of the fixture.

The blades were randomly selected from one production lot of double edge

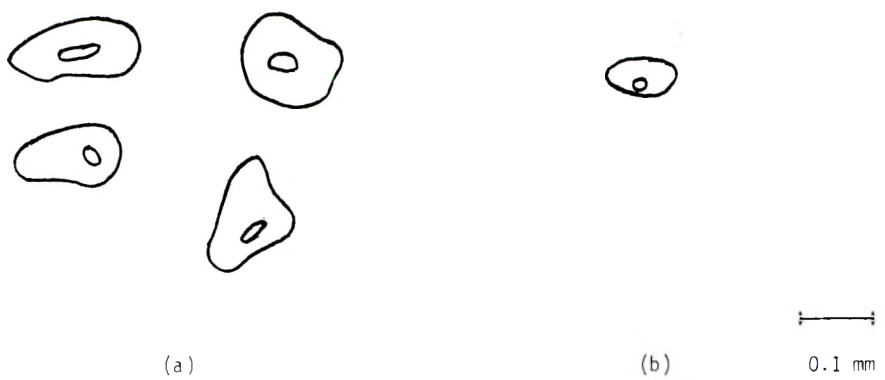


Figure 2. Typical cross sections of (a) beard and (b) head fibers

*Instron Corp., Canton, MA.

Schick blades coated with Vydax®* and were discarded after 5 to 10 cuts. No effort was made to examine or control variations in honing from blade to blade. Since only a few cuts of one fiber were made with each blade, and since these cuts occurred randomly over a distance of several millimeters along the blade edge, blade damage due to cutting was assumed to be minimal and neglected.

The TM Instron Tester recording system was used for measuring the cutting forces at speeds up to 5 in./min. A Tectronix®† storage oxcilloscope (Model RM564) fitted with a strain bridge was used to measure forces at cutting speeds up to 50 in./min. By combining both methods, cutting forces were measured at speeds ranging from 0.1 in./min to 50 in./min.

The use of scalp hair as a model for beard hair proved unacceptable, since the diameter of scalp hair is appreciably smaller and the cross-section much more regular than that of beard hair. Cutting force measurements in our laboratories demonstrated conclusively that scalp hair requires less force-to-cut (f-t-c) than beard hair. This may be merely a reflection of the fiber diameter or may be the result of the unexpected irregularity of beard hair cross-sections as shown in Fig. 2. Although Hollander and Casselman (1) reported that white beard fibers are more difficult to cut than dark fibers, it was found here that the presence or absence of melanin had no effect on the f-t-c.

The majority of the fibers used in this study were plucked from the beard of one subject. However, this was done only after comparing these fibers with those of four other volunteers and ascertaining that they were representative of beard fibers in general.

All chemicals used were analytical reagents except in the case of detergents, which were of commercial quality.

Results and Discussion

f-t-c versus Cross-sectional Area

Beard fibers removed by plucking from 5 subjects were completely hydrated and then cut 10 times at a rate of 0.5 in./min, while a stream of water at about 25°C was played on the fiber in the jig. The snippets created by the cuts were less than 1 mm long, and beard fibers as short as 3 cm suffice to yield statistically meaningful data. After each cut, the cross-section of the freshly cut surface was established by quickly photomicrographing the fiber in the jig and then determining the area with a planimeter. A plot of the f-t-c against the cross-sectional area is not very revealing (Fig. 3). Nevertheless, the slope for each fiber was determined by a linear regression analysis which forces the

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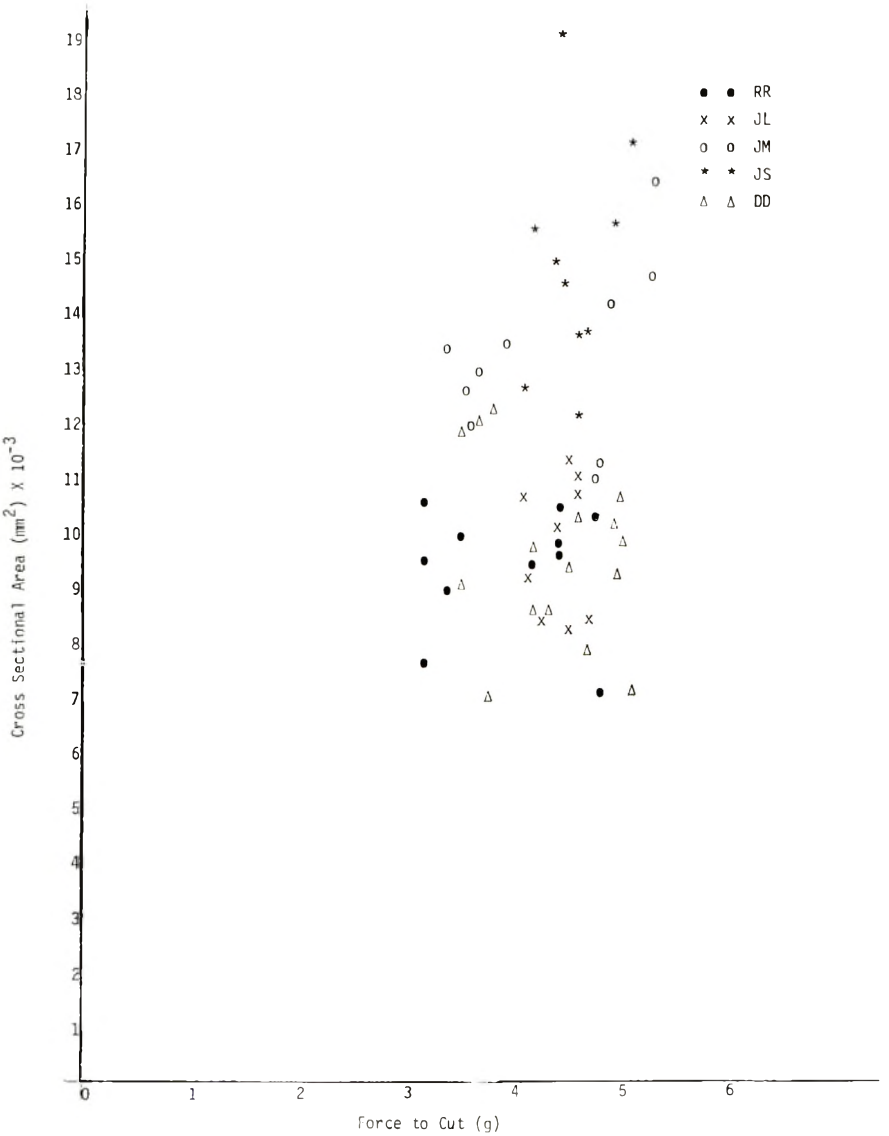

Figure 3. Cross-sectional area vs. cutting force

line through the origin (4), and the slope of this line is included in the tabulation of the data in Table I.

An examination of Fig. 3 and of the data in Table I suggests that there is some correlation between fiber diameter and f-t-c. The average standard error of the slope is about 5 per cent, while the average standard error of the

Table I
f-t-c of Beard Hair

Subject	Cross-Sectional Area $mm^2 \times 10^4$ ± Std Error	Cutting Force in (g) ± Std Error	Slope (g/mm^2) ± Std Error
JM(a)	122 ± 3.9	3.93 ± 0.23	322 ± 27
(b)	147 ± 6.2	4.85 ± 0.33	332 ± 15
RR(a)	99 ± 2.1	4.43 ± 0.09	445 ± 7.7
(b)	94 ± 4.9	3.36 ± 0.07	358 ± 14
JL(a)	115 ± 5.3	4.49 ± 0.11	389 ± 15
(b)	89 ± 3.4	4.40 ± 0.10	494 ± 24
JS(a)	142 ± 9.3	4.52 ± 0.18	318 ± 17
(b)	160 ± 10.5	4.56 ± 0.11	285 ± 21
DD(a)	82 ± 5.8	4.50 ± 0.22	550 ± 45
(b)	121 ± 1.2	3.67 ± 0.09	303 ± 4.4
(c)	96 ± 2.3	4.68 ± 0.12	487 ± 11

f-t-c is about 4 per cent. Since the errors are of the same order of magnitude, there appears to be no need to utilize the time-consuming determination of the cross-sectional area. Instead, the f-t-c suffices for all practical purposes.

F-t-c versus Rate of Cutting

In order to determine how the speed of travel of the blade through the fiber affects the f-t-c. two fibers were cut at 5 different cutting rates varying between 0.1 in./min to 50 in./min. Although there is a spread in the data (Fig. 4), linearity is assumed over the range studied.

For practical purposes, it appears that the f-t-c increases significantly as the rate of cutting is increased. It is noted that a rate of cutting of 50 in./min approaches normal shaving conditions. Nevertheless, the much slower rate of 0.5 in./min was routinely used here in order to avoid the time-consuming complication of employing a storage oscilloscope.

Effect of Moisture on Cutting Force

It is an axiom of shaving tradition that the presence of water facilitates shaving and reduces discomfort. This tradition finds scientific support in the observations that the shear and tensile moduli of keratin fibers are functions of the relative humidity (5) and that hair is appreciably weakened by complete hydration.

The influence of relative humidity on the f-t-c was established by conditioning beard hairs at various humidities and cutting them at that humidity in an environmental chamber positioned on the Instron Tester. The cutting forces (normalized to the value at 0 per cent R.H.) are plotted in Fig. 5, which also includes curves for the shear and tensile moduli computed from

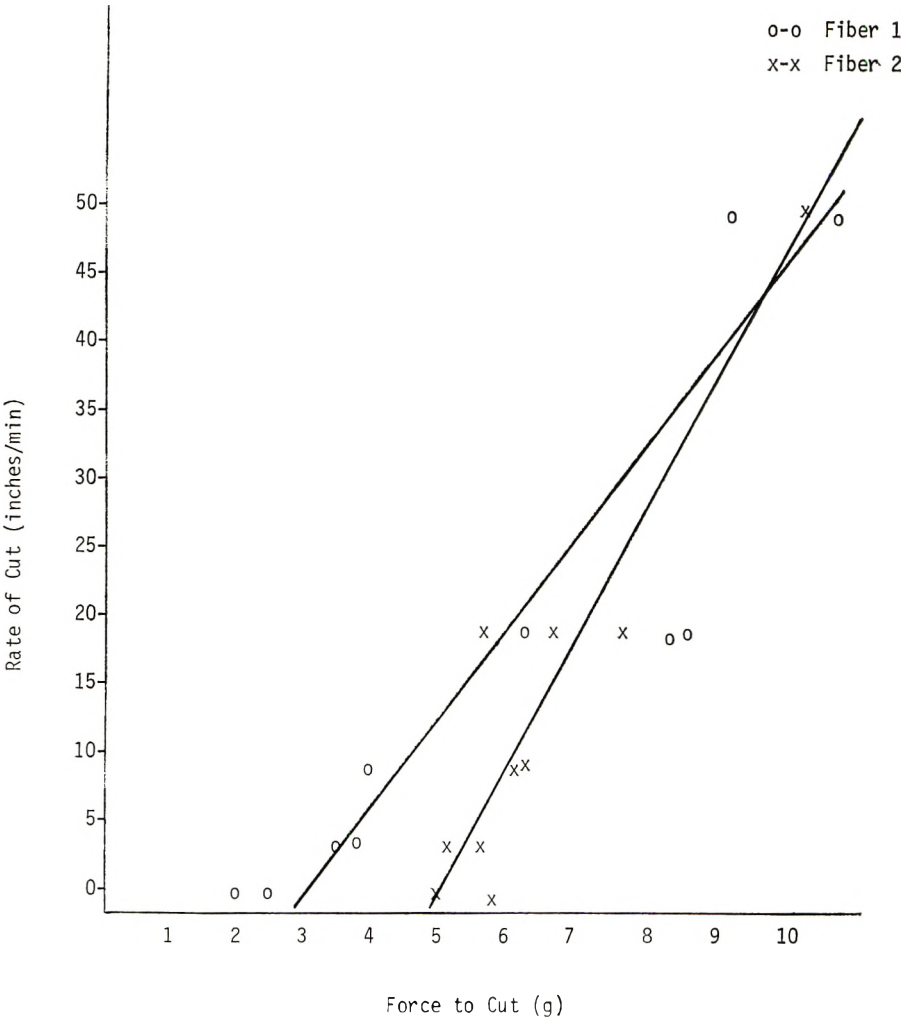

Figure 4. Cutting rate vs. cutting force (cantilever)

the data of Mitchell and Feughelman (5). The fact that the cutting force is less dependent on relative humidity than the shear or tensile modulus suggests that these moduli—even at a rate of 0.5 in./min—are not the predominant factors in beard hair cutting. Instead, the f-t-c might be more closely related to stress propagation or to the creation of new surface area, than to the viscoelastic properties of the fiber.

Hollander and Casselman (1) made creep measurements on scalp hair, which they then extrapolated to beard hair, to determine the rate of softening

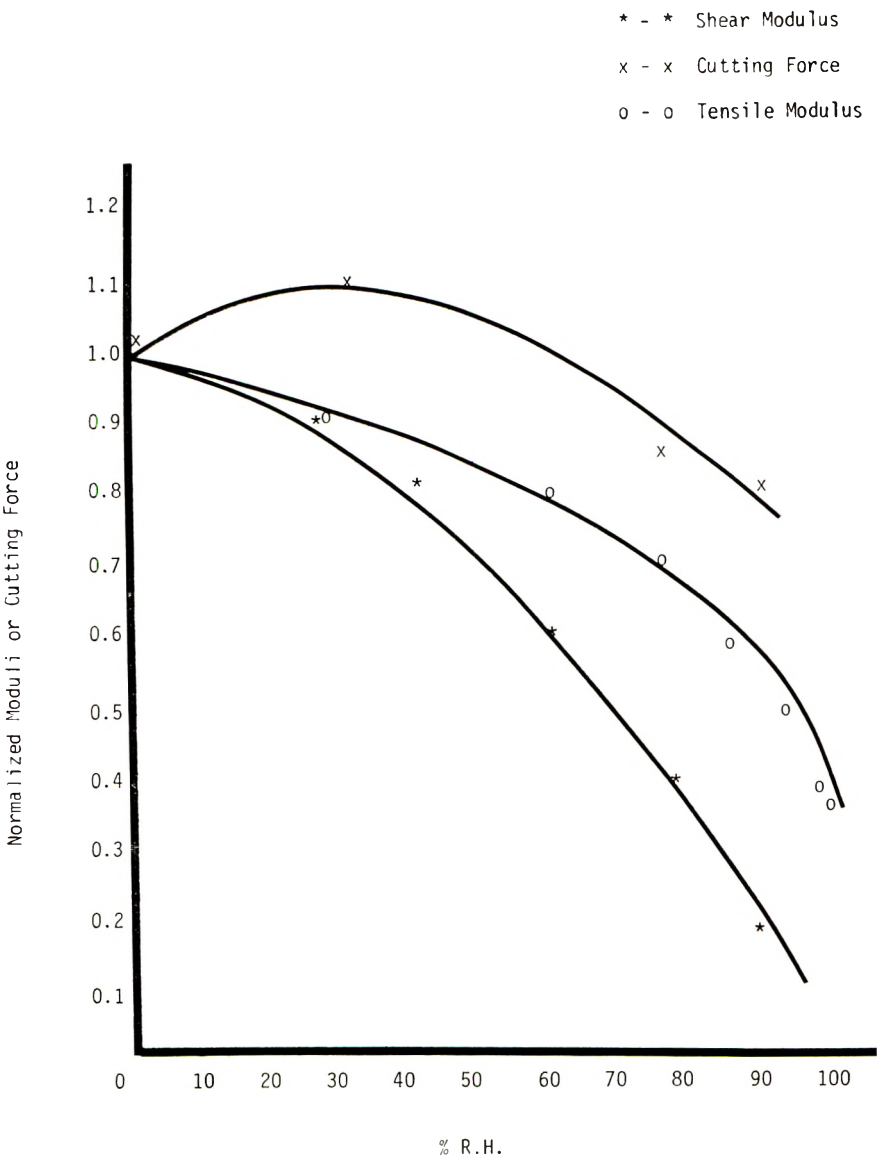

Figure 5. Normalized moduli or cutting force vs. per cent R.H.

of the hair with increased temperature. Their extrapolated value was 2½ to 3 min for complete hydration at 120°F.

The hydration studies reported below were performed as follows: (1) a fiber was cut 10 times at ambient humidity to determine an equilibrium dry

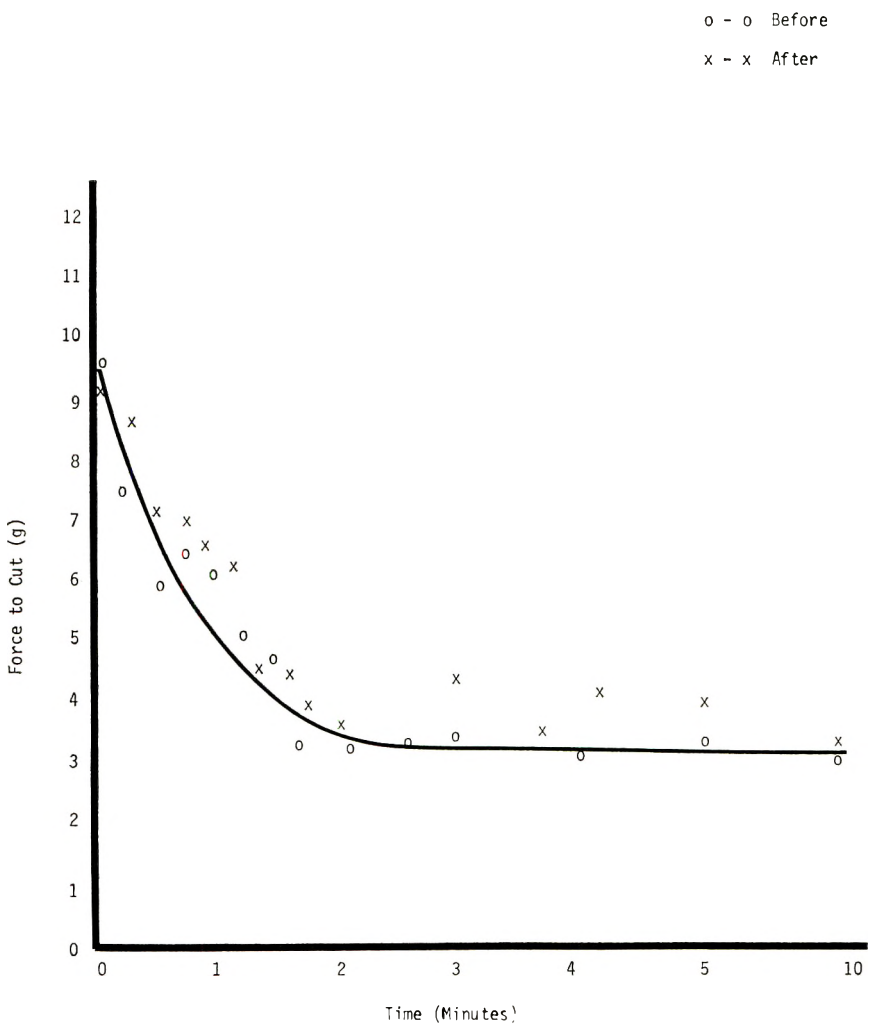

Figure 6. Hydration time before and after washing with sodium lauryl sulfate (0.5 per cent) at 23° C

value;* (2) a stream of water or of a test solution was directed at the fiber, and cutting forces were determined as rapidly as possible during the first 2 min; after this, cuts were made at 1 min intervals; (3) after 10 min a series of 10 cuts were made as a measure of the equilibrium cutting force in the wet state.

*Fig. 5 shows that the "dry" f-t-c is relatively insensitive to variations in the range of 10 to 60 per cent relative humidity.

Table II
Hydration Time and Cutting Force as a Function of pH

pH	9.1	6.84	4.01
Buffer	0.01 *M* borax	0.05 *M* phosphate	0.05 *M* phthalate
Hydration time (min) ± std dev.	2.60 ± 0.47	2.13 ± 0.26	2.16 ± 1.21
Force to cut wet (g) ± std dev.	4.01 ± 0.40	3.91 ± 0.39	4.31 ± 0.31
Ionic Strength of buffer	0.060	0.091	0.053

The results of a typical experiment are shown in Fig. 6. Following the determination of the initial hydration curve at 23°C (Fig. 6, before), the fiber was washed in 0.5 per cent aqueous sodium lauryl sulfate solution, rinsed with distilled water, and dried for 48 h in a desiccator over P_2O_5; a second hydration curve was then determined (Fig. 6, after). The absence of any significant difference suggests that the rate of hydration is not altered by the removal of surface lipids. Based on the f-t-c, beard hair fibers appear to have been completely hydrated by exposure to water at room temperature within about 2 to 3 min.

The rate of hydration of beard hair and the f-t-c could be expected to be dependent on the pH of the aqueous medium. Accordingly, cutting measurements were made in various buffer solutions, and the results are summarized in Table II.

Any differences between the hydration times or between the f-t-c are statistically insignificant. The ionic strengths and chemical composition of the buffers are different, but the variations are not likely to affect the results. These rather unexpected data indicate that the pH has little or no effect on the f-t-c or on the rate of hydration of beard hair.

Effect of Temperature on Cutting Force

It is part of shaving folklore that the use of cold water during shaving leads to an uncomfortable shave. It seemed important, therefore, to measure the effect of water temperature on the cutting force. Three types of experiments were performed on wet fibers and on dry fibers. For measurements on dry fibers, the fixture used for cutting beard fibers was fitted with a heating tape and a surface thermocouple. A single hair was cut (dry) 10 times at each of 4 temperatures. The average cutting forces and the standard deviations are tabulated in the chart below and indicate that the f-t-c of dry fibers is lowered by raising the temperature.

Temperature (°C)	23	56	65	75
f-t-c (g) ± std dev.	17.6 ± 1.5	14.5 ± 1.3	12.3 ± 0.80	13.1 ± 0.84

Significance (Values connected by underlining are significantly different)

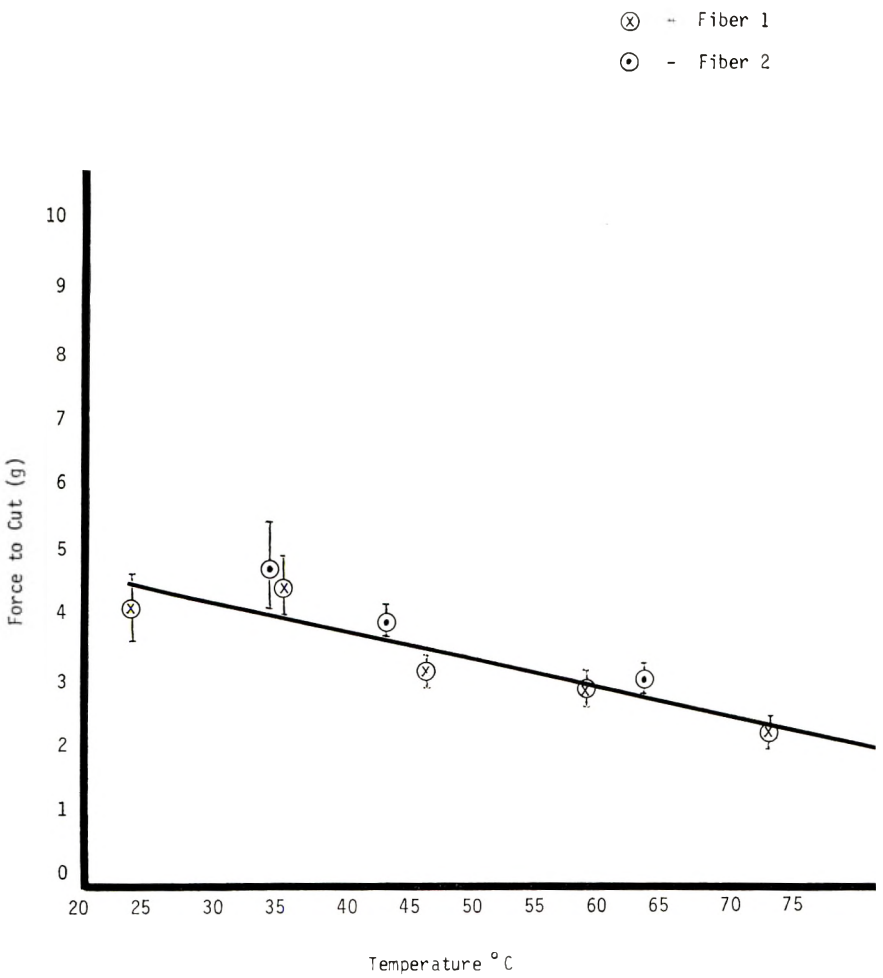

Figure 7. Temperature (°C) vs. force to cut (g) on same fiber (wet)

Similar studies of wet beard hair were conducted by measuring the cutting force of thoroughly hydrated fibers which were maintained at the desired temperature by playing a stream of warm water on the fiber positioned in the jig. Two fibers were each cut about 10 times at each of 4 or 5 temperatures, and the results are shown in Fig. 7. The computed least square slope indicates that the f-t-c is lowered by 0.051 g/°C. These data demonstrate that shaving should be easier at elevated temperatures.

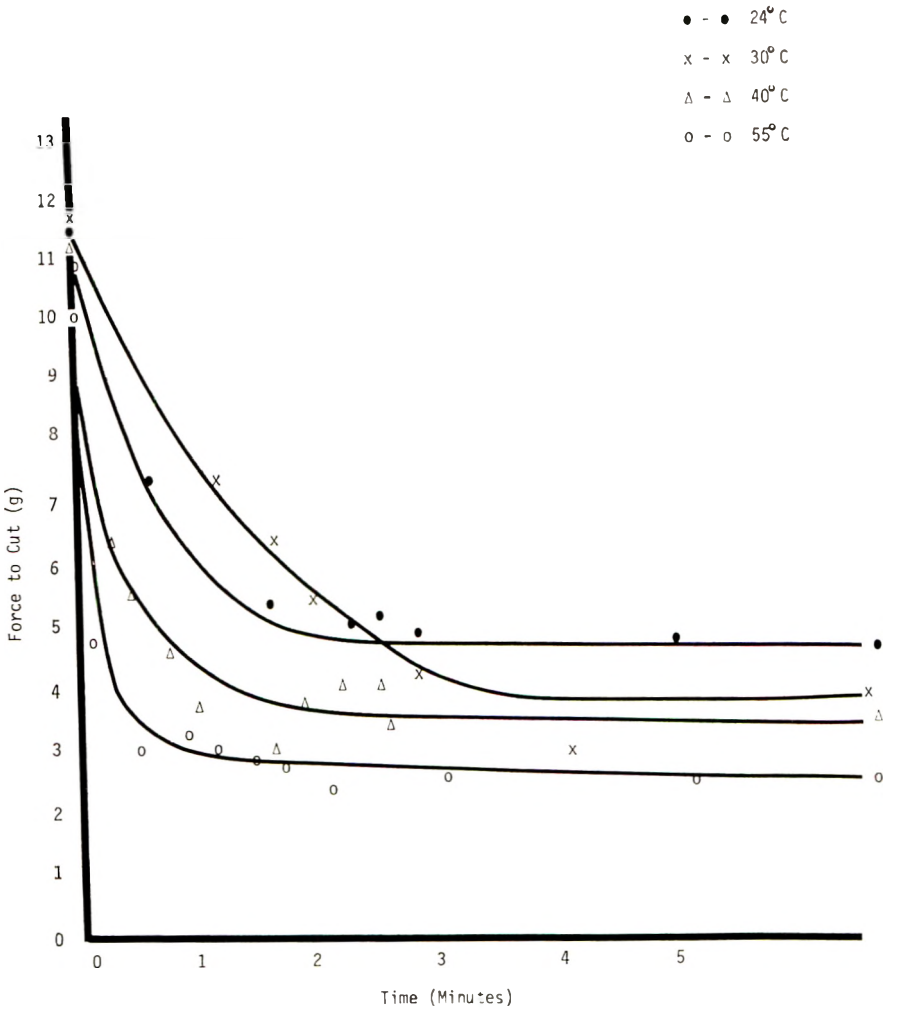

Figure 8. Force to cut as a function of time of hydration of beard hair in dilute (0.5 per cent) sodium lauryl sulfate at different temperatures

In a third study the influence of temperature on beard hair hydration time was determined in the presence of a wetting agent (0.5 per cent sodium lauryl sulfate). In view of the number of cuts required, it was necessary to use different hairs at different temperatures; the fibers were selected to exhibit the same dry cutting force at room temperature. Nevertheless, one anomaly occurred in the data at 30°C. All the data are summarized in Fig. 8. It is apparent that the time required for full hydration is shortened by increasing

the temperature. Again (see Fig. 6), the f-t-c completely hydrated fibers decreases as the temperature is raised.

Effect of Chemical Treatments

The results of the studies reported so far indicate that the rate of hydration of beard hair is relatively fast and that softening—as measured by f-t-c—is not significantly altered by modest changes in pH or the presence of a wetting agent. In view of this, the effect of potassium stearate solution, aerosol shaving cream concentrates, and several finished commercial creams on the f-t-c hair was determined. None of these materials showed any reduction of the hydration time or of the f-t-c the fiber beyond that effected by water. Therefore, it was decided to utilize a few more drastic chemical treatments.

Since 1-propanol/water mixtures are known to make hair easier to extend (6) a 45:55 (w/w) 1-propanol/water mixture was directed on the fiber during cutting. The data given in the chart below show that the difference between the wet and dry cutting force (47 per cent lowering) is about the same as that of samples treated with distilled water (51 per cent lowering). The hydration time is comparable to that of water-treated fibers.

	Dry	Wet (propanol/water)
Average f-t-c (g) ± std dev.	6.20 ± 1.40	3.27 ± 0.82
Hydration times (min) ± std dev.	—	2.76 ± 1.44

In a more drastic procedure, beard fibers of known wet f-t-c were soaked in a commercial waving lotion (6.0 per cent thioglycolic acid, pH 9.3) for 5, 7, and 10 min, rinsed in several changes of distilled water, and then cut under a stream of water. The results tabulated in the chart below confirm again that significant chemical attack (7 min in waving lotion) on the fiber causes only a minor (13 per cent) reduction in the cutting force (98 per cent confidence level). Fibers exposed to the waving lotion for 10 min could not be cut because of excessive damage to the fiber, which allowed it to bend and to be split axially.

	f-t-c ± Std Dev. (in g)	
Time of Waving Lotion Treatment	Control	Treated
5 min	3.95 ± 1.06	4.20 ± 0.65
7 min	5.20 ± 0.57	4.51 ± 0.54

McLaren (7) has shown that wool is reduced more drastically in thioglycolic acid in aqueous 1-propanol (45:55 w/w) than aqueous thioglycolate. Therefore, it was decided to measure the cutting force of fibers soaked (for 2 or 5 min) in 1-propanol/water (45:55) containing either 0.5 per cent or

6.0 per cent thioglycolic acid adjusted to pH 9.2 with ammonia. In a separate set of experiments fibers were also treated with either 0.5 per cent or 6.0 per cent thioglycolic acid at pH 11.3 (with sodium hydroxide) for 2 or 5 min. Only exposure to 6 per cent thioglycolic acid at pH 11.3 for 5 min effected any lowering of the cutting force over that of water. The majority of the fibers with this treatment disintegrated before they could be cut, and the two fibers which could be cut bent during cutting.

The conclusions, which must be drawn from these studies, are that even the most severe chemical (covalent bond) damage, which is known to lower the tensile modulus drastically, has almost no effect on the force required to cut beard hair. In addition, rupture of hydrophobic bonds by 1-propanol/water also appears to have almost no effect on the f-t-c.

Conclusion

A device is described which permits measurement of the force required to cut a beard hair fiber under a variety of conditions. Studies with this device show that the force required to cut wet beard fibers with commercial razor blades is about 65 per cent less than that of dry fibers. Beard hair is almost completely hydrated by exposure to water for about 2 min at room temperature, and this hydration is accelerated by an increase in temperature. The force required to cut beard hair is not lowered below that of water by the presence of a wetting agent, shaving cream, or soap solution. The force required to cut wet beard hair with a razor blade is lowered significantly by very severe chemical attack on the fiber. On the other hand, the force required to cut beard hair increases as the rate of blade travel increases.

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Methoden zur Bestimmung von Fluoridionen in Zahnpasten

II. Studie zur Trennung der zu bestimmenden Ionen durch Destillation mit überhitztem Wasserdampf

M. O. SCHMITZ-MASSE**, M. HANOCQ* und
M. HERPOL-BORREMANS**

Synopsis — Methods for the Determination of Fluoride Ions in Toothpastes. II: Study of the Separation of the Ionic Species of Interest with the Aid of Super-heated Systems. — In the first part of this study the conditions for the separation of fluoride ion in toothpastes are examined. The method which depends on the codistillation of hexafluorosilicic acid with super-heated steam, which is generated in a suitable apparatus, has proved itself useful. The procedure is independent of the method of preparation of the toothpaste and of the type of fluoride derivative present in the product. The second part is a comparison of two assay procedures. The first one — a spectrophotometric procedure — utilizes formation of a complex between cerium***, alizarin, Complexon, and fluoride. The other — a potentiometric assay — depends on the use of a lanthanum fluoride membrane electrode. From the point of view of sensitivity, reproducibility, and precision the two methods are comparable.

Einleitung

Im Verlauf des ersten Teils der vergleichenden Arbeit (1) haben wir gezeigt, daß die Bestimmung des Aluminiumfluorids nach seiner Abtrennung vom Anion durch Mikrodiffusion nicht quantitativ war.

Unser Ziel bei der Inangriffnahme dieser neuen Arbeit ist es, eine brauchbare Bestimmungsmethode von Fluoridionen in Zahnpasten vorzuschlagen, und

* Laboratoire de Chimie Analytique, Chimie Pharmaceutique inorganique et de Toxicologie (Professeur L. Molle). Institut de Pharmacie, Université Libre de Bruxelles, Campus de la Plaine, 1050 Bruxelles.

** Ministère de la Santé Publique. Institut d'Hygiène et d'Epidémiologie (Professeur Dr. Lafontaine). Département Pharmaco-Toxicologie. 14, rue J. Wytsman, 1050 Bruxelles.

zwar ganz gleich, welches Fluor-Derivat verwendet wird und welcher Art die Zusammensetzung des kosmetischen Präparates ist, die dem Bearbeiter sehr oft unbekannt ist.

Um das zu erreichen, benutzten wir die Trennungsmethode von Willard und Winter (2), die darin besteht, mit Hilfe eines Wasserdampfstromes Hexafluorkieselsäure überzutreiben, die durch Einwirkung einer starken Mineralsäure (HCl, ClO_4, H_2SO_4) in Gegenwart von Kieselsäure auf das Fluoridion erhalten wird. Das so isolierte Fluorid wird dann im Destillat bestimmt.

Sehr viele Bearbeiter nehmen ihre Zuflucht zu dieser Technik (3 bis 6). Sie erfordert eine Reihe von Vorsichtsmaßnahmen, vor allem eine konstante Einstellung der Destillationstemperatur auf ungefähr 135—140°. Um Nachteile zu vermeiden, haben wir eine Apparatur benutzt, die auf das von J. M. Icken und B. M. Blank (7) und von R. Truhaut (8) vorgeschlagene Gerät zurückgeht.

Nach Vergleich der beiden Trennungsmethoden, die wir studiert haben, Destillation und Mikrodiffusion, betrachten wir nachfolgend diese zwei Bestimmungsmethoden.

Die erste, spektrophotometrische, basiert auf der Bildung des Komplexes Cerium(III)-Alizarin-complexon-fluorid in Anwesenheit einer 25 %igen wäßrigen Dimethylsulfoxidlösung; diese empfindliche und spezifische Methode war schon Gegenstand früherer Arbeiten (9 bis 11).

Für die zweite, die potentiometrische Methode, braucht man eine spezifische Lanthanfluorid-Membranelektrode nach Frant und Ross (12), mit der man die Konzentration von Fluoridionen in einer Lösung genau so bequem mißt, wie man den pH-Wert nach der klassischen Methode mit der Glas-Elektrode bestimmt. Es sei betont, daß diese spezifische Elektrode bereits Gegenstand zahlreicher Arbeiten geworden ist. Sie wurde angewendet, um durch direkte Messung Spuren von Fluor in den Zähnen (13), in Grundstoffen (14), in Gewässern (15, 16) und in der Luft (17) zu bestimmen. Diese Methode ist ständig bei der Analyse solcher Arzneimittel wie Multivitaminpräparaten (18) und Zahnpasten verwendet worden, die Fluoride von Natrium und Zinn enthielten (19).

Trotz der sehr großen Spezifität einer solchen Elektrode ist es möglich, theoretisch verschiedene Arten von Abweichungen durch bestimmte Ionen zu erhalten:

Kationen, wie die von Silicium (IV), Aluminium (III), Eisen (III), Zirconium (IV), Thor (IV) etc., bilden Komplexe oder fällen das Fluoridion.

Die Anwesenheit fremder Ionen — ob sie nun das Fluoridion binden oder nicht — bewirkt eine Änderung der Ionenkonzentration der Lösung.

Bestimmte Kationen oder Anionen können auf der Membranfläche mit einem der gebildeten Ionen der letzteren eine unlösliche Verbindung eingehen.

Diese verschiedenen Möglichkeiten sind von dem einen von uns (9) experimentell untersucht und seitdem von verschiedenen Forschern bestätigt worden. So hat auch H. Zentner (20) in Anwesenheit großer Mengen von Ca^{++}-Ionen ein nicht typisches Ergebnis mit der Lanthanfluorid-Membranelektrode festgestellt. Er führt dieses Phänomen auf eine Adsorption sich überlagernder Ionen auf der Membranfläche zurück.

Manche Autoren empfehlen den Gebrauch eines Puffers, allgemein TISAB* genannt, dessen Aufgabe es ist, den pH-Wert und die Ionen-Konzentration konstant zu halten. Er dient durchweg dazu, gewisse störende Ionen komplex zu binden, wie die Kationen Si (IV), Al (III) und Fe (III). Diese Wirksamkeit der komplexierenden Stoffe ist jedoch eine Funktion des Verhältnisses zwischen der Menge der Fluoridionen und der der Kationen, die ausgeschaltet werden müssen (21). Nun ist, worauf wir bereits hingewiesen haben, die genaue Zusammensetzung der Zahnpaste dem Analytiker sehr oft unbekannt. Darum erscheint es uns unerläßlich, selbst wenn man von einer spezifischen Lanthanfluorid-Membranelektrode Gebrauch macht, vorab die zu bestimmenden Ionen zu isolieren.

Experimenteller Teil

Geräte und Reagenzien

Geräte

— Spektrophotometer Beckman Acta V mit 1 cm Quarz-Küvette
— pH-Meter-Millivoltmeter Radiometer PHM 52
— Orion Lanthanfluorid-Membranelektrode Modell 94-09
— Calomel-Elektrode Beckman, Modell 39170
— Hamilton-Spritze zu 100 µl
— Apparatur zur Entwicklung von überhitztem Wasserdampf zwecks Abführung des Fluorwasserstoffes

Schema der letzteren ist in Abbildung 1 wiedergegeben. Die Apparatur besteht aus einem Glaskolben B 1, in dem der aus der Analysenprobe und Kieselsäure in stark saurem Milieu gebildete Silicofluorwasserstoff in der Hitze zu Fluorwasserstoff hydrolysiert wird; dieser wird dann durch einen Wasser-

* Eisessig, Kochsalz, Natriumzitrat

dampfstrom, der im Kolben B 2 entwickelt wird, mitgerissen. Die Temperatur muß im Verlauf einer solchen Destillation so genau geregelt werden, daß eine vollständige Mitnahme des Fluorwasserstoffs erreicht, ein Übergehen der verwendeten Perchlorsäure aber vermieden wird. Die Gleichmäßigkeit der Destillation wird durch das Tetrachloräthan erreicht, das im Glaskolben B 3 am Kochen gehalten wird (146°). Dieses Lösungsmittel destilliert so in den Mantel B 4, daß der übergehende Wasserdampf überhitzt wird, während er die Glaswindungen durchströmt, die den Behälter B 1 umgeben.

Im Verlauf der verschiedenen Versuche, die wir mit einer solchen Apparatur durchgeführt haben, stellten wir fest, daß das Übertreiben von Fluorwasserstoff, welcher aus der Säureeinwirkung auf Aluminiumfluorid stammte, nicht quantitativ erfolgte. Um diesen Nachteil, der schon andernorts in der Literatur erwähnt wird (22, 23), abzustellen, haben wir einen Versuch durchgeführt,

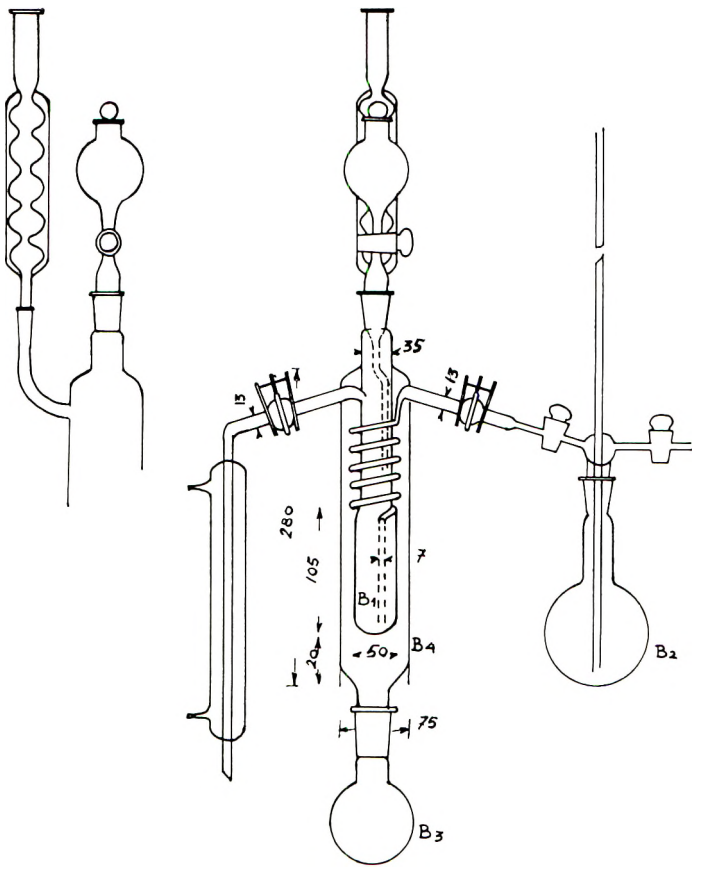

Abbildung 1

der den Einfluß der Konzentration der Perchlorsäure, das Volumen des Destillats, die Dauer der Destillation und vor allem die Maße der Apparatur berücksichtigt. Aus diesem Grunde ist das Schema des Destillationsapparates, den wir empfehlen, in *Abbildung 1* genau dargestellt.

Reagenzien

— Außer den Reagenzien, die im Verlauf des 1. Teils der Arbeit (1) verwendet wurden, sind folgende benutzt worden:

— Wäßrige Natronlauge 0,02 N

4 g Natriumhydroxid (Merck p. a.) in etwa 200ml doppelt destilliertem Wasser lösen. Nach Abkühlung auf 500 ml mit doppelt destilliertem Wasser auffüllen und 10fach verdünnen.

— Pufferlösung ph 5,7

294 g neutrales Natriumcitrat-Dihydrat (Merck p. a.) in etwa 500 ml doppelt destilliertem Wasser lösen; danach 33,6 ml einer 70 %igen Perchlorsäurelösung (Merck p. a.) hinzufügen und mit doppelt destilliertem Wasser auf 1 Liter auffüllen.

— Oleyl-Alkohol (BDH)*

Arbeitsweise

Trennung der Fluoridionen durch Destillation

Zunächst wird ein Leerversuch durchgeführt. Zu diesem Zweck füllt man in den Kolben B 1 einige Glaskugeln, 500 mg Silbersulfat (das dazu dient, eventuell vorhandene Chloride zu binden), 0,5—2 ml Oleyl-Alkohol (Antischaummittel) und 25 ml der 70 %igen Perchlorsäurelösung. Man bringt das im Glaskolben B 3 enthaltene Tetrachloräthan zum Kochen (146°) und regelt den Wasserdampfstrom so, daß in einer Stunde etwa 180 ml abdestilliert werden, die in einem geeichten 200-ml-Glaskolben aufgefangen werden, der 10 ml einer 0,02 N Natronlauge enthält. Man wäscht den Kühler R *(Abb. 1)* mit einigen ml doppelt destilliertem Wasser und füllt bis zum Eichstrich auf. Nach Abkühlung der Apparatur wird eine Versuchsprobe der Zahnpasta von ungefähr 500 mg auf einem Cellophanpapier genau abgewogen und mit dem Papier in den Kolben B 1 gegeben. Man führt die Destillation genau so aus wie bei dem Leerversuch. Nach Filtrierung des Destillats durch Papier What-

* British Drug House.

man Nr. 1 trocken wird die Gehaltsbestimmung mit 10 ml des Filtrats nach einer der beschriebenen Methoden ausgeführt.

Spektrophotometrische Bestimmung

Die Technik ist weiter oben beschrieben worden (1).

Potentiometrische Bestimmung

In einen Polyäthylenbecher von 50 ml gibt man 10 ml des Destillats und 10 ml Pufferlösung pH 5,7. Nachdem man die Calomel-Elektrode darin angebracht und geeicht hat, wird unter stetigem Schütteln das Millivoltmeter so eingestellt, daß es eine gleichbleibende Abweichung von 100 anzeigt; letztere entspricht einer unbekannten Menge von Fluoridionen x µg. Wenn das Gerät auf diese Weise eingestellt ist, führt man mit der Hamilton-Spritze 100 µl der Lösung von 100 ppm Fluoridionen ein, entsprechend 10 µg Anionen. Unter diesen Bedingungen zeigt das Millivoltmeter eine neue Abweichung an:

Bei $100 = x\ \mu g$

und $y = x\ \mu g + 10\ \mu g$

läßt sich die unbekannte Menge Fluoridionen x, ausgedrückt in µg, mit Hilfe folgender Gleichung berechnen:

$$\frac{100}{y} = \frac{x\ \mu g}{x\ \mu g + 10\ \mu g}$$

Alle Messungen werden bei 25° ± 0,1° ausgeführt.

Versuchsergebnisse

Diese Trennungsmethode wurde gleich zu Anfang bei verschiedenen reinen Fluor-Derivaten angewendet. Die verschiedenen Resultate, die man mit der spektrophotometrischen Bestimmung der Ionen im Destillat erhielt, werden auf *Tabelle 1* wiedergegeben; sie sind, da die Messungen genau und reproduzierbar sind, sehr zufriedenstellend.

Die Analyse dieser Tabelle zeigt durchweg, daß im Gegensatz zur Methode der Mikrodiffusion, die wir früher betrachtet hatten (1), die Methode der Trennung mit überhitztem Wasserdampf in der Apparatur, die wir genau beschrieben haben, es erlaubt, das Aluminiumfluorid quantitativ zu trennen. Andererseits sind in gleicher Weise Versuche mit bekannten Mengen von Natriumfluorid zusammen mit fremden Stoffen, wie Kieselsäure und Tricalcium-

Tabelle I

Spektrophotometrische Bestimmung der Fluoridionen nach der Trennung durch überhitzten Wasserdampf.

Derivat	µg F⁻ theor.	Bearbeiter	n(1)	x, µg F⁻ gefunden (2)	$\bar{x}$, µg F⁻ gefunden	$\bar{x}$ - theor. Wert µg F⁻	% F⁻ gefunden	% F⁻ gefunden	s (3)
NaF	500	1	6	493.2-502	497.83	— 2.17	99.57	99.84	0.99
	500	2	5	494.8-501.9	498.58	— 1.42	99.70		
	382	2	6	374.0-390.0	383.00	+ 1.00	100.21		
AlF_3	500	1	6	492.2-497.2	494.66	— 5.34	98.93	99.25	1.15
	500	2	6	482 -503.1	494.53	— 5.47	98.91		
	485	1	3	486.6-489	487.7	+ 2.7	100.56		
SnF_2	500	1	7	495.2-503.3	499.77	— 0.23	99.95	99.97	0.47
	450	2	4	448 -452.0	450.02	+ 0.02	100.00		
PO_3FNa_2	500	1	7	497.2-503.2	499.61	— 0.39	99.22	98.61	2.54
	494	1	3	481 -499	487.2	— 6.8	99.33		
	501	2	2	475 -490	482.5	— 18.5	96.31		
$MgSiF_6$	507.4	1	6	491 -510	501.5	— 5.9	99.13	99.42	1.37
	461.8	2	4	460.1-466.8	463.2	+ 1.4	100.30		

(1) Anzahl der Versuche.

(2) Die beiden in dieser Kolonne eingeschriebenen Zahlen stellen die Extremwerte dar, die im Laufe einer Versuchsserie erhalten wurden.

(3) Standardabweichung $s = \sqrt{\Sigma(x-\bar{x})^2/(x-1)}$

phosphat, durchgeführt worden. Die Resultate, auf *Tabelle II* wiedergegeben, zeigen, daß unter diesen Bedingungen die Methode anwendbar bleibt, sogar in Anwesenheit von Natriumsilikat, das den normalen Prozeß der Mikrodiffusion behindert, worauf wir hingewiesen haben.

Die Trennungsmethode durch Mikrodestillation erscheint also durchführbar, welches auch immer das zu untersuchende Derivat und die beigegebene fremde Substanz sei. In allen Fällen kommen die Mittelwerte der Resultate, die durch zwei Prüfer in zwei verschiedenen Laboratorien erarbeitet und in Prozenten Fluoridionen ausgedrückt wurden, den theoretischen Konzentrationen sehr nahe. Die Fehler, die maximal 2,5 % erreichten, sind mit den in der Mikroanalyse gegebenen Normen vereinbar.

Unser Versuch hat zur Bestimmung von Fluor in vier Zahnpasten geführt, deren genaue Zusammensetzung im Anhang dieser Arbeit aufgeführt ist. Einige von ihnen sind im Handel (I, II), andere sind unter unserer Aufsicht hergestellt worden (III, IV): alle enthalten eine Mischung von zwei Fluor-Derivaten Zinn(II)fluorid — Natriumfluorid; Natriummonofluorphosphat — Natriumfluorid; Calciumfluorid — Natriumfluorid und Aluminiumfluorid — Natriummonofluorphosphat.

Tabelle II

Mikrodestillation von Fluorwasserstoff in Anwesenheit von Fremdsubstanzen (spektrophotometrische Bestimmung).

µg F^- (NaF) theor.	zugefügte Fremdsubstanzen	n (1)	x, µg F^- gefunden (2)	$\bar{x}$, µg F^- gefunden	$\bar{x}$ - theoret. Wert µg F^-	% F^- gefunden	% $\bar{F}^-$ gefunden	s (3)
500	$(PO_4)_2Ca_3$	3	495.6-499.1	497.7	— 2.4	99.5	99.8	0.63
250		3	248.5-252.4	250.1	+ 0.1	100.0		
500	SiO_2	3	497.3-502.7	499.5	— 0.5	99.9	99.85	0.41
250		3	248.8-250.3	249.5	— 0.5	99.8		
500	SiO_3Na_2	3	496.3-503.4	499.5	— 0.5	99.9	100.06	0.71
250		3	248.3-252	250.6	+ 0.6	100.2		

(1) Anzahl der Versuche.

(2) Die beiden in dieser Kolonne eingeschriebenen Zahlen stellen die Extremwerte dar, die im Laufe einer Versuchsserie erhalten wurden.

(3) Standardabweichung.

Tabelle III

Bestimmung der Fluoridionen in einer Zahncreme
(Mikrodestillation — Spektrophotometrie).

Bearbeiter	Zahncreme I (SnF_2-NaF) theoretisch 100 mg F^-/100 g				Zahncreme II (Na_2FPO_3-NaF) theoretisch 100 mg F^-/100 g			
	Versuchsmenge g	F^- mg/100 g gefunden x	x - theor. Wert mg/100 g	$\bar{F}^-$ mg/100 g	Versuchsmenge g	F^- mg/100 g gefunden x	x - theor. Wert mg/100 g	$\bar{F}^-$ mg/100 g
1	0.5082	102.2	+ 2.2	102.8	0.4920	98.8	— 1.2	97.6
		102.4	+ 2.4			98.1	— 1.9	
	0.5044	102.7	+ 2.7		0.4991	99.6	— 0.4	
		102.7	+ 2.7			99.8	— 0.2	
	0.4995	103.6	+ 3.6		0.4974	95.9	— 4.1	
		103.1	+ 3.1		0.4974	95.1	— 4.9	
2	0.5024	101.8	+ 1.8	102.5	0.5037	99.3	— 0.7	98.1
		102.7	+ 2.7			99.1	— 0.9	
	0.5018	102.2	+ 2.2		0.5083	96.2	— 3.8	
		103.1	+ 3.1			96.9	— 3.1	
	0.5035	102.6	+ 2.6		0.4962	98.1	— 1.9	
		102.8	+ 2.8			98.6	— 1.4	
Ergebnis	102.7 mg % g		s = 0.48		97.9 mg % g		s = 1.57	

Tabelle IV

Bestimmung der Fluoridionen in einer Zahncreme
(Mikrodestillation — Spektrophotometrie).

Bearbeiter	Zahncreme III	theoretisch 200,7 mg $\bar{F}$/100 g			Zahncreme IV	theoretisch 101,8 mg $\bar{F}$/100 g		
	Versuchsmenge g	$\bar{F}$ mg/100 g gefunden x	x - theor. Wert mg/100 g	$\overline{\bar{F}}$ mg/100 g	Versuchsmenge g	$\bar{F}$ mg/100 g gefunden x	x - theor. Wert mg/100 g	$\overline{\bar{F}}$ mg/100 g
1	0.4494	195.8	— 4.9	197.2	0.3730	99.9	— 1.9	100.8
	0.4388	198.7	— 2.0		0.3757	101.8	0	
2	0.5040	199.4	— 1.3		0.4284	100.5	— 1.3	
		199.4	— 1.3			101.4	— 0.4	
	0.4132	200.2	— 0.5	199.0	0.3910	100.3	— 1.5	100.4
		199.6	— 1.1			98.8	— 3.0	
	0.4287	198.9	— 1.8		0.3885	99.5	— 2.3	
		197.1	— 3.6			101.9	+ 0.1	
Ergebnis	198.6 mg $\bar{F}$ %/g		s = 1.44		100.5 mg $\bar{F}$ %/g		s = 1.12	

Die Ergebnisse der verschiedenen Versuche sind in den *Tabellen III* und *IV* zusammengefaßt. Ihr Studium erweist, daß sich die Methode für den angestrebten Zweck vollkommen eignet und bestätigt im besonderen, daß die Trennung auch in Anwesenheit kleiner Mengen von Aluminium-Kationen quantitativ ist. Der normale Analysengang wird selbst durch die Anwesenheit großer Mengen von Kieselsäure, Phosphat-, Calcium- und Magnesiumionen nicht gestört. Die mittleren Werte der verschiedenen Resultate sind den theoretischen Konzentrationen nahe; die Standard-Abweichung erreicht maximal nicht mehr als 1,6 %.

Vergleich der beiden vorgeschlagenen Bestimmungsmethoden

Es erschien uns interessant, die Genauigkeit der beiden Methoden zur Bestimmung von Fluoridionen nach ihrer Trennung durch Wasserdampfdestillation zu vergleichen. Zu diesem Zweck haben wir diese Anionen bestimmt, sowohl mit der spektrophotometrischen wie mit der potentiometrischen Methode mit Hilfe der Lanthanfluorid-Membranelektrode. Die Resultate, die wir nach der einen oder anderen Methode erhalten haben, sind auf *Tabelle V* abgebildet. Der Vergleich zeigt, daß die durch Spektrophotometrie erhaltenen Werte um

Tabelle V

Bestimmung des Fluoridions durch Spektrophotometrie und Potentiometrie nach Destillation. Ein Vergleich.

Theoretischer Wert 100 mg F^-/100 g								
Untersuchte Zahncreme	Spektrophotometrie				Potentiometrie			
	Versuchsmenge g	F^- mg/100 g gefunden x	x - theor. mg/100 g	$\overline{F^-}$ mg/100 g s	Versuchsmenge g	F^- mg/100 g gefunden x	x - theor. mg/100 g	$\overline{F^-}$ mg/100 g s
I	0.5082	102.3	+ 2.3		0.4905	105.6	+ 5.6	
	0.5044	102.7	+ 2.7		0.5044	104.3	+ 4.3	
	0.4995	103.3	+ 3.3	102.5	0.4981	102.7	+ 2.7	103.4
	0.5024	101.3	+ 1.3	s = 0.67	0.5135	101.2	+ 1.2	s = 1.66
	0.5018	102.7	+ 2.7		0.4804	103.1	+ 3.1	
	0.5035	102.7	+ 2.7					
II	0.4920	98.5	— 1.5		0.4991	102.7	+ 2.7	
	0.4991	99.7	— 0.3		0.4928	101.4	+ 1.4	
	0.4974	95.4	— 4.6	97.8	0.4974	100.5	+ 0.5	100.7
	0.5037	99.2	— 0.8	s = 1.66	0.5032	100.1	+ 0.1	s = 1.49
	0.5083	96.5	— 3.5		0.5188	98.7	— 1.3	
	0.4962	98.4	— 1.6					
V	0.4689	95.9	— 4.1		0.4689	99.2	— 0.8	
	0.4800	97.3	— 2.7		0.4685	99.3	— 0.7	
	0.4907	98.0	— 2.0	97.3	0.4800	99.2	— 0.8	98.0
	0.5109	96.9	— 3.1	s = 0.88	0.5008	96.3	— 3.7	s = 1.42
	0.4781	97.4	— 2.6		0.5042	97.2	— 2.8	
	0.5019	98.4	— 1.6		0.4981	96.6	— 3.4	

1,5 % niedriger sind als die durch Potentiometrie erzielten; die Reproduzierbarkeit beider Methoden scheint sonst identisch zu sein.

Wenn es auch nicht verwunderlich ist, daß zwei Analysen-Methoden, die sich überhaupt nicht ähneln, dennoch zu wenig unterschiedlichen Resultaten führen, scheint uns trotzdem die Versicherung erlaubt, daß sie sich sowohl vom Standpunkt der Spezifität als dem der Genauigkeit aus als gleichwertig erwiesen haben.

Schlußfolgerung

Wir meinen, folgendes bewiesen zu haben: Mit Hilfe einer Apparatur von angemessenen Ausmaßen ist die Methode einer Trennung durch Mikrodestillation, die durch eine spektrophotometrische oder potentiometrische Bestimmung ergänzt wird, besonders gut verwendbar zur Feststellung und Bestimmung von Fluoridionen in einer Zahnpasta, und zwar unabhängig von der Art der Zubereitung und der zu untersuchenden Derivate.

Hauptsächlich ist sie anwendbar für Serienmessungen, weil vom Augenblick an, in dem ein Muster der Wasserdampfdestillation unterliegt, schon eine zweite Versuchsmenge in die Apparatur eingeführt werden kann.

Wir haben allerdings festgestellt, daß die Anwesenheit einer erhöhten Menge von Aluminiumoxid (40—50 %) den Analysengang stört. Wir rechnen damit, im Laufe weiterer Untersuchungen die vorgeschlagene Methode diesem besonderen Fall anpassen zu können.

Zusammenfassung

Im ersten Teil dieser Arbeit werden die Bedingungen zur Trennung von Floridionen in Zahnpasten untersucht. Die Methode, die auf der Mitnahme von Hexafluorkieselsäure durch überhitzten Wasserdampf beruht, der in diesem Apparat von geeigneten Ausmaßen erzeugt wird, hat sich als brauchbar erwiesen, unabhängig von der Zubereitung der untersuchten Zahnpasten und der Natur des im Präparat enthaltenen Fluorderivats.

Der zweite Teil ist dem Vergleich der beiden Bestimmungsmethoden vorbehalten: Die eine, die spektrophotometrische, greift zurück auf die Bildung eines Komplexes von Cerium(III)-alizarin-complexon-fluorid in Anwesenheit einer 25 %igen (v/v) Lösung von Dimethylsulfoxid. Die andere, die potentiometrische Methode, basiert auf der Verwendung einer Lanthanfluorid-Membranelektrode. Unter dem Gesichtspunkt der Empfindlichkeit, der Reproduzierbarkeit und der Genauigkeit haben diese beiden Methoden sich als gleichwertig erwiesen.

Anhang

Zusammensetzung der verschiedenen analysierten Zahnpasten

Zahncreme 1

Zinn-II-fluorid 0,272 g, Natriumfluorid 0,075 g (100 mg Fluoridionen), Natriumbenzoat 4,0 g, Eugenol 0,025 g, Methyl-p-hydroxybenzoat 0,100 g, Excip. ad 100 g.

Zahncreme 2

Natriummonofluorphosphat 0,570 g, Natriumfluorid 0,055 g (Fluoridionen 100 mg), Natriumbenzoat 4,0 g, Eugenol 0,025 g, Methyl-p-hydroxybenzoat 0,100 g, Excip. ad 100 g.

Zahncreme 3

Calciumfluorid 0,180 g, Natriumfluorid 0,250 g (Fluoridionen 200,7 mg), Natriumbenzoat 4,0 g, Eugenol 0,025 g, Natriumcarragenat 1,25 g, Calciumcarbonat 26,00 g, gefällte Kieselsäure 6,00 g, Tricalciumphosphat 4,00 g, Natriumlaurylsulfat 2,00 g, Natriumhexametaphosphat 0,20 g, Methyl-p-hydroxybenzoat 0,100 g, Saccharin 0,040 g, Sorbitlösung 70%ig 25,00 g, äther. Öle 0,120 g, Menthol 0,070 g, Phenol (offizinell) 0,010 g, Farbe q. s., destill. Wasser ad 100,0 g.

Zahncreme 4

Natriummonofluorphosphat 0, 4545 g, Aluminiumfluoridtrihydrat 0,1013 g (Fluoridionen 101,8 mg), Dinatriumphosphat 0,1045 g, neutrales Natriumphosphat 12 H_2O 0,2015 g, Methyl-p-hydroxybenzoat 0,100 g, Saccharin 0,0398 g, Magnesiumtrisilikat 5,00 g, Natriumbenzoat 4,00 g, Natriumcarragenat 1,25 g, gefällte Kieselsäure 6,00 g, gefällte Kreide 26,00 g, Natriumlaurylsulfat 2,00 g, Sorbitlösung 70 %ig 25,00 g, ätherische Öle u. Farbe q. s., destill. Wasser ad 100 g.

Zahncreme 5

Handelsübliche Zahncreme, deren genaue Zusammensetzung uns nicht bekannt ist, mit Ausnahme des Gehalts an Fluoridionen, der 0,100 g F^- als Natriummonofluorphosphat betrug.

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A MESSAGE FROM THE EDITOR OF THE JSCC

As you know, The Journal of the Society of Cosmetic Chemists, along with all other publications, has been faced with increasing production costs over the past year. The cost of paper along with printing costs have increased to the point where some action must be taken in order to insure the continued scientific and technical integrity of The Journal. The Publications Committee has been considering this problem for the past several years and has found it necessary to increase its subscription rate to non-members, increase its advertising rates as well as to increase the total number of pages of advertising copy. We have now reached the point where we can no longer increase the number of advertising pages without proportionally increasing the number of pages devoted to scientific and technical articles. To do so would drastically change the nature of our Journal to the point where it would lose much of its professional status.

Therefore, in order to increase the number of pages devoted to scientific and technical papers, the Board of Directors have approved the institution of a modest page charge to be assessed each author of a published paper. While these page charges will be waived by the business office in cases of undo hardship, it is expected that sufficient income will be received so as to insure the continued viability of scientific and professionals journals.

If we recognize that publication is one of the goals of research, then the cost of publication should be included as part of the research funding.

Sincerely,
John J. Sciarra, Ph.D.
Editor

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Physical Techniques For Assessing Skin Moisturization

ALFRED J. QUATTRONE, Ph.D.* and KARL LADEN, Ph.D.†

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Synopsis: An overview is presented of some PHYSICAL TECHNIQUES currently available for use in SKIN MOISTURIZATION studies. Water soaking of unmodified versus ether-extracted stratum corneum, for example, causes a marked alteration in the BIOMECHANICAL properties of these tissues (i.e., swelling capacity, elastic modulus, relaxation function, and work index). Differences in moisture binding properties as measured by GRAVIMETRIC and SCANNING CALORIMETRIC analyses of the tissue at various relative humidities are related. The correlation of changes in these traits with changes in the pliability and strength of corneum tissue and its capacity to retain moisture is discussed. Criteria for judging dry versus hydrated skin *IN VIVO* are also reviewed through the utilization of TRANSPIROMETRY, PHOTOGRAPHY, and SCANNING ELECTRON MICROSCOPY (SEM). Analysis via these techniques of the effect of humectants and occlusive oils on water retention within skin is presented.

Introduction

A wide variety of *in vitro* and *in vivo* physical procedures are available for investigating phenomena associated with moisturization of the stratum corneum. This presentation will touch on the usefulness of gravimetric, scanning calorimetric and, mechanical techniques in quantitating levels of moisture retention and pliability obtained after treating corneum tissue with various materials *in vitro*. In addition, *in vivo* evaluations by means of transpirometry low magnification photography, and scanning electron microscopy (SEM) of skin replicas before and after treatment of human skin with moisturizing formulations will be reviewed in detail.

*Past address: Gillette Research Institute, 1413 Research Blvd., Rockville, MD 20850. Present address: Tracor-Jitco, Inc., 1776 E. Jefferson St., Rockville, MD 20852.
†Past address: Gillette Research Institute, 1413 Research Blvd., Rockville, MD 20850. Present address: Carter Products Div., Carter-Wallace, Inc., Cranbury, N.J. 08512.

In vitro methodologies

A. *Gravimetric Measurement of Water Binding*

This widely used method of assessing the affinity of isolated stratum corneum for water consists of equilibrating corneum tissue at a fixed temperature in a constant relative humidity (RH) chamber until a nonvarying weight is attained. Temperatures in the range of 0° to 35°C and RH in the range of 10 to 90 per cent have been commonly used (1-5). A period of 5 to 7 days is generally required for attaining constant weights at 10 to 90 per cent RH. The samples are then desiccated over a dehydrating agent until a dry weight is reached. The data are expressed as the per cent moisture uptake (i.e., weight per cent gained) with respect to the dry weight.

The capacity of callus tissue to remain soft and flexible was shown by gravimetric assay of water uptake to be directly correlated with the presence of natural moisturizing factors (NMF) in the tissue (1, 2). Human callus, extracted with diethyl ether and water, and then allowed to equilibrate in chambers at 35 per cent RH, gained 5 per cent less absolute weight (i.e., moisture) than callus which was just water-soaked and equilibrated. At 80 per cent RH, this differential increased to as much as 20 per cent water uptake. Laden and Spitzer were able to identify the major humectant in NMF as being sodium 2-pyrrolidone-5-carboxylate (2). Since that study, Middleton has further substantiated the role of an NMF (e.g., lactic acid) in influencing the state of stratum corneum hydration (6). These investigations support the hypotheses that NMF within the cornified cells of the epidermis maintain the flexibility of this tissue (a) through enhancing the rate of water migration from lower living cell layers and (b) by hindering the release of moisture from the skin surface by reinforcing the water retaining capacity at very low RH.

B. *Differential Scanning Calorimetry*

A direct measure of the levels of "bound" (nonfreezing) and "unbound" (freezing) water in animal and human corneum strips was described by Walkey (7) in 1972 using a differential scanning calorimeter.* She was able to quantitate the level of hydration after equilibration of dried strips at various RH from latent heat of melting curves. Walkley showed that when dry human corneum attained a 45 per cent moisture regain above dry weight, approximately two-thirds of that water (0.35 mg/mg dry corneum) was nonfreezable (i.e., bound). Her results were confirmed by the findings of Anderson *et al.* (5), based on proton magnetic resonance and infrared spectroscopy, which demonstrated the presence of 0.35 to 0.50 mg of bound water per mg

*Perkin Elmer Corp., Norwalk, Conn. 06856.

of dry corneum. Both Walkley (7) and Anderson *et al.* (5) hypothesized that the freezable fraction was held only by diffusional barriers, whereas the non-freezable (i.e., bound) fraction was strongly associated with the polar groups of corneum proteins and NMF. Walkley further found that the effect of extracting lipids with diethyl ether and NMF with water allowed for an increase in the portion of bound water from 0.29 to 0.41 mg/mg of dry animal foot pad corneum. Ether-water extraction caused a dramatic lowering of corneum diffusional barriers and allowed for a greater proportion of sorbed water to be bound by polar residues of the remaining proteins and lipids. In a study of swelling properties of unmodified and ether-water extracted stratum corneum via biomechanical analyses as is described below, Wolfram *et al.* (8) have confirmed Walkley's finding.

C. *Biomechanical Analyses*

As several investigators have pointed out (4, 6, 8, 9), the elastic modulus of stratum corneum is directly correlated with the amount of water retained in the tissue. Water retention, in turn, has been demonstrated to depend on the surrounding temperature and relative humidity and on the structural integrity of the cornified cells (10-14). It is widely observed that, in winter, the rate of moisture replacement from beneath the corneum becomes inadequate in comparison to the rate of transpiration from the surface. Moreover, exposure to organic solvents or aqueous detergents damages the skin and allows for dehydration of the outermost cell layers. As these cornified layers become progressively more dehydrated, they become inflexible and less extensible than the deeper layers causing the surface to stiffen, flake, and crack, while the person involved perceives the tight, drawn, and itching sensations of chapped and dry skin.

Changes in the reversible stretching properties of animal corneum may be evaluated by the method of Elfbaum and Wolfram (9) who used the extensometer.* Their results have been expressed as the work index (i.e., the ratio of the work required to reversibly stretch a strip of corneum to a 5 per cent displacement in a given solvent versus preliminary 5 per cent displacement of the same strip in water). In this way, aqueous dimethyl sulfoxide (DMSO) concentrations greater than 50 per cent cause a reversible stiffening (increase in the work index) of animal corneum together with extensive swelling in the cells of the cornified tissue. A concomitant increase in the tautness and hardness of the samples is observed at the macroscopic level. In a similar manner, ether-delipidized tissue has been water-swollen and reversibly stretched (8). Unlike the dimethyl sulfoxide treatment, exposure of stratum corneum to

*Instron Corp., Canton, Mass. 02021.

ether and then water causes about a 30 per cent dry weight loss and nearly a two-fold decrease in stiffness as measured by the work index.

A contacting probe balance, developed by E. M. Buras at our laboratory,* has been employed for measuring the cross-sectional swelling of specimens of stratum corneum (8). This instrument permits the rapid and accurate recording of the displacement of a probe placed in contact with the dry surface of dry cornified tissues. After measuring the dry state thickness, each sample is submerged in 0.1 per cent aqueous Triton X-100,† and the displacement due to swelling is continuously monitored. The final thickness is determined after equilibrium is reached, usually within 10 min. The percentage swelling is calculated by comparing the initial displacement with that following imbibition.

Changes in the remaining two dimensions (termed in-plane swelling) are measured directly for square samples (20 x 20 mm) before and after immersion in 0.1-per cent aqueous Triton X-100. After 16 h, all squares are removed, and their perimeters remeasured to the nearest 0.1 mm to determine the percentage change. To test the effects of delipidization, squares have been preextracted with diethyl ether for 1.5 h, air dried, incubated as above for 16 h, and then remeasured.

The pronounced weakening of the ether-pretreated specimens as reported in the Instron study correlates well with distinct increases in both cross-sectional and in-plane swelling. Ether-water extraction and concomitant loss of NMF causes about a 3-fold increase in thickness when subsequently reswollen, but only a 5 per cent enhancement in area (8), as compared with water-soaked stratum corneum which is not preextracted. Ether pretreatment, therefore, alters not just the lipid content and moisture retaining capacity through loss of NMF, but the physical dimensions, strength, elasticity, and membrane permeability as well. The high swelling and reduction in the rate of strain recovery (i.e., decrease in the viscous component of elasticity) of the ether-treated samples may be explained by marked alteration in the conformation of keratin molecules, which is brought about by the breakdown of hydrogen bonds and accompanying aqueous exposure of previously buried

*The probe balance consists of a freely moving aluminum arm suspended on aluminum-coated Mylar flexures. The balance has a 2 mm^2 probe at one end, which contacts the corneum specimen placed on a flat surface. At the other end of the balance arm, there is a position transducer which consists of a vane and a proximity probe. Displacement is measured by change in capacitance which varies with the length of a cylindrical probe inserted into the vane. The proximity probe of this dynamic balance is wired into a commercial driver unit, and then into the Y-axis of the recorder. An aluminum cylinder coil fastened to the balance arm above a magnet constitutes a damping system. The instrument has also been used to measure diametrical swelling of hairs.

†Rohm and Haas Co., Philadelphia, PA 19105.

hydrophilic and hydrophobic groups within the keratin of the delipidized corneum cells.

Instead of the work index, Rieger and Deem (13, 14) have analyzed the elastic modulus (i.e., the ratio of stress imposed on stratum corneum to the strain applied at a constant strain rate) and the relaxation function (i.e., the decay in the strain rate of the tissue while a constant stress is imposed). Both the elastic modulus and the time constant of relaxation of unmodified stratum corneum decreases with increasing RH, providing an objective characterization of pliability (13). Upon application of known humectants such as 4 per cent sodium pyrrolidone carboxylate and 50 per cent glycerol in water, there is a decided increase in elasticity and a faster relaxation time in comparison with dried tissue (14). Light mineral and safflower oils have the opposite effect, suggesting an increase in the stiffness and a decrease in the pliability of the treated samples.

In Vivo Methodologies

A. *Transpirometry*

Some of the current techniques, which we utilize *in vivo*, have aided us greatly in directly evaluating cutaneous moisturizers. Several types of instrumentation are described in the literature for application in the direct determination of transpiration rates (15-21). We employ an apparatus designed by Slegers and Dobson for measuring the rate of moisture release from the skin into a stream of dry nitrogen (Fig. 1).* This stream, passing in a flow-through chamber on the skin, and a stream of identical pressure flowing independently of the skin are compared for their thermal conductivity in a gas chromatograph. Two of these systems, each equipped with integrators, allow for simultaneous measurement of the rate of moisture loss at two separate sites (i.e., a control and a test).

We have observed all three sources of water (i.e., surface moisture, transpired water, and eccrine sweating) which Berube *et al.* have mentioned (15). After an equilibration and "calm-down" period of 30 min, surface moisture is eliminated and most panelists become sufficiently conditioned to a room tem-

*The transpirometer consists of two thermal conductivity gas chromatographs. For each unit, streams of dry nitrogen at 200 ml/min/cm^2 are split into two equal components, one passing directly into the chromatography unit, while the other streams into a flow-through probe on the skin before entering this thermal conductivity analyzer. The difference in the conductance between the split streams is measured, and a signal from each chromatograph is sent to a dual pen recorder. The latter is equipped with two repeating potentiometers allowing for integration of each signal. Standard curves are obtained for each system before use each day by application of known quantities of water (0.1 to 1.0 μl. for the 0.05 mV sensitivity range) to filter paper sealed within each chamber.

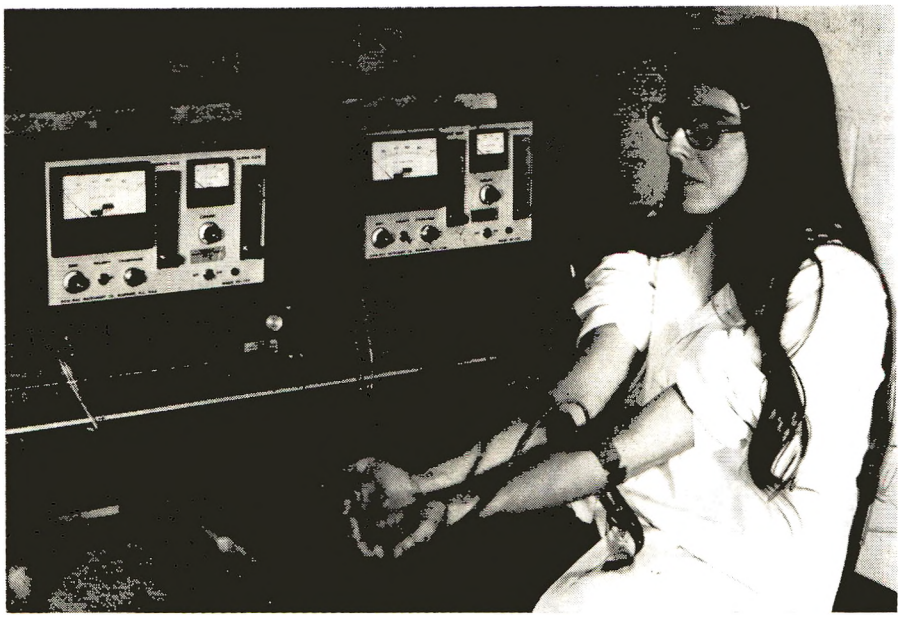

Figure 1. Dual-recording transpiration analyzer as designed by Slegers and Dobson (23)

perature of 20°C and RH of 50 per cent or less so as not to demonstrate significant eccrine sweating. Those panelists, who are not stabilized after this period, are eliminated from testing that day.

Upper forearms and the calves of the lower legs of 12 panelists of both sexes were examined, with one site serving as an untreated control and a contralateral site being used for the tests. Occlusion of the skin for 5 min with a water-saturated gauze patch followed by tissue blotting of the excess water produces an initial 20 to 40 per cent increase in water loss, which decreases steadily over a 30-60 min period to the level of transpiration on the opposite side. Similar application and blotting of a commercial emollient cream reverses this trend, giving a 20 per cent decrease in transpired water after 1 h and about a 10 per cent decrease after 4 to 6 h.

We interpret these results with an emollient cream as indicative of temporary retardation of water loss afforded by an oil barrier. Once the dry corneum imbibes moisture from lower living cell layers and attains a new equilibrium water content, the temporary effect of the diffusional barrier of the cream is slowly overcome, and the original transpiration rate is reestablished. The effect of the barrier cream is then to raise the moisture concentration within the dead cells without effecting the final equilibrium transpiration rate too much.

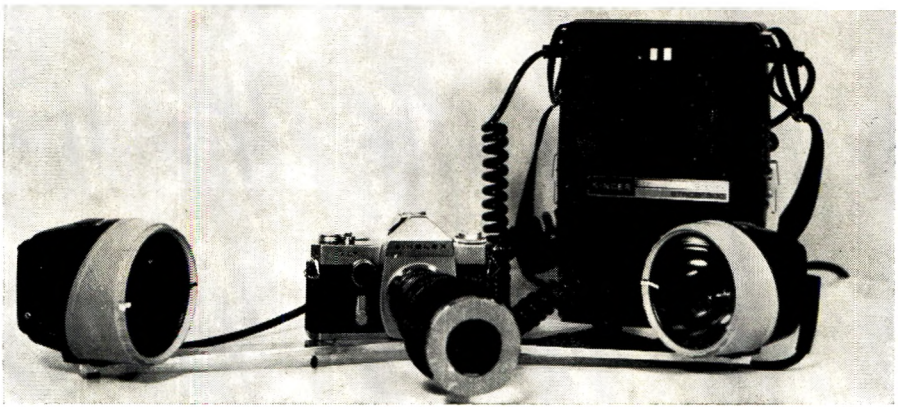

Figure 2. Photographic apparatus for producing low magnification prints

B. *Low Magnification Photography*

Low magnification photography of skin sites before and at specific intervals after treatment provides a rapid subjective means of evaluating the moisturizing potency of emollient creams. Kodacolor II® (ASA 80) film* and a Ricoh® 35 mm reflex camera† with a 55 mm lens and extension tube system give a final magnification of 3.5-fold. The front of the lens is equipped with a gridded disc fixed onto 95 mm spacing bars. This system allows the correct focus to be obtained for an area of 33 x 23 mm^2 with minimal readjustment (Fig. 2). A camera aperture of f16 gives optimal exposure and depth of field at 1/60 of a second. Two strobe heads (7100 ecps) on a Graflex 500® flash unit‡ are fixed to the camera so as to be set 220 mm from the photographic site at an angle of 25° above the plane of the skin. To minimize glare, polarizing filters are placed over the strobe heads and oriented perpendicular to a polarizing filter placed over the camera lens.

A panel of three independent judges evaluates the photographs based on the level of white lines and flakes discernible in these pictures (Fig. 3). A rating scale (from 6 equals no white margins nor scales to 0 equals only white margins and many lifted scales) is utilized by the assessors. This rating scheme is similar, but opposite in value to that described by Gibson (22) and Middleton (6). The major advantage of our technique is to provide an extremely helpful low magnification (3.5 X) of each site studied (Fig. 3).

*Eastman Kodak Co.,Rochester, NY 14650.
†Braun North America, Cambridge, MA 02141.
‡The Singer Co., Graphics Systems Division, San Leandro, CA 94577.

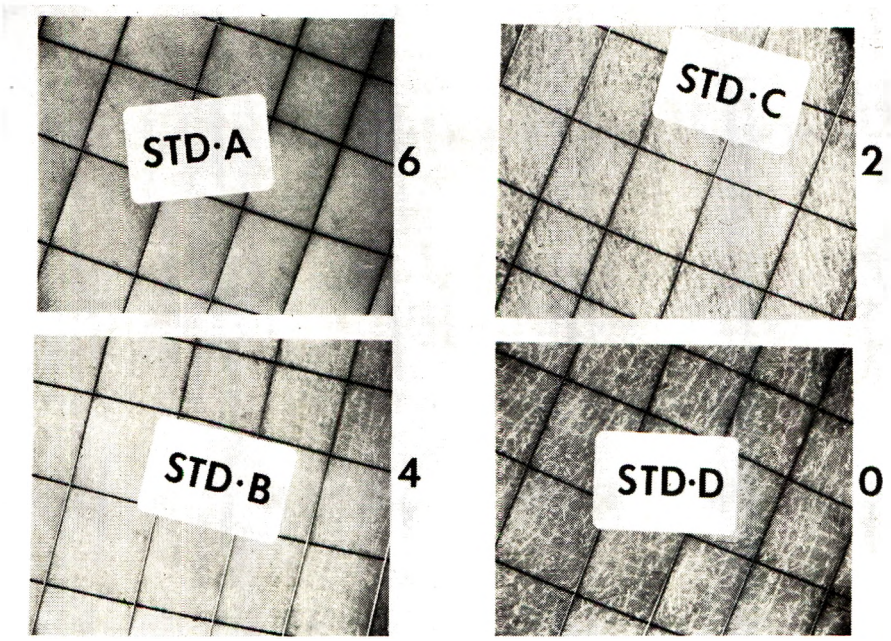

Figure 3. Photographic rating scale: Standard A: 6 equals no white margins nor uplifted dry flakes; Standard B: 4 equals a few white margins, but no uplifted dry flakes; Standard C: 2 equals many white margins and a few uplifted dry flakes; Standard D: 0 equals totally distinct white margins and many uplifted dry flakes

In an initial investigation, 30 panelists were treated at 1 skin area with an emollient cream, while a site on the opposite side remained untreated. Photographs were taken of both sites after 6 and 24 h, and evaluated by 3 judges. Their scores were averaged and analyzed by the t-test statistic (23). The treated sites were scored nearly 3 points higher at 6 h and 1 point higher at 24 h (Table I), indicating that the visual benefits provided by the emollient cream could be readily discerned from the photographs.

More recently, we have been examining the effect of various camera color filters on the quality of black-and-white photographs. Dent, in 1941, published an elegant study (24) on skin photography as a function of the wavelength of light reflected from the skin. He determined that detailed texture, definition and lines discerned under violet-blue lighting (300-450 nm) result from the fact that very little of this light penetrates below the stratum granulosum.

Table 1

Mean Photographic Scores

Hours After Start	Skin Sites: Treated	Skin Sites: Untreated	Null Probability[a]
6	5.06	2.43	< .001
24	3.72	2.55	< .001

[a]Based on the t-test statistical comparison.

Thus, light directed into the camera can only be reflected from within the corneum, the lucidium and to a lesser extent the granulosum layers. Light of the green through red wavelengths (480 to 800 nm), however, is able to penetrate further into the dermis. Photographs obtained under this light show veins and blood vessels, but no surface detail or texture. Gibson (25) has reported on a direct viewing system of goggles equipped with a monochromatic vision filter (MV812)* which converts colors to shades of gray. A gray photographic scale is then used for evaluating levels of erythema or changes in skin pigmentation.

We have confirmed Dent's observations, in particular, for Caucasian skin after elution with acetone for 30 sec. Photography using Panatomic-X® black-and-white film† and a Kodak CC50C-cyan filter† (passing mainly <550 nm and >740 nm) gives pictures with significantly more surface detail than photography using the same film but with a yellow filter equivalent to Kodak 81C (passing mainly >450 nm) (Fig. 4).

C. SEM

SEM investigations of panelists' skin have been used to correlate the influence of the chemical composition of various preparations with the moisturizing efficacy of these formulations *in vivo*. Bernstein and Jones (26, 27) have developed a replication method which would neither damage the skin nor become destroyed by the electron beam. Initially, a negative impression of the skin is formed by polymerization of 10:1 mixture of Silastic® 382 Elastomer‡ and RTV-Thinner‡ with stannous octoate. From this impression,

*Ilford Inc., Ciba-Geigy Co., Paramus, NJ 07652.
†Eastman Kodak, Rochester, NY 02142.
‡Dow Corning Corp., Midland, MI 48640.

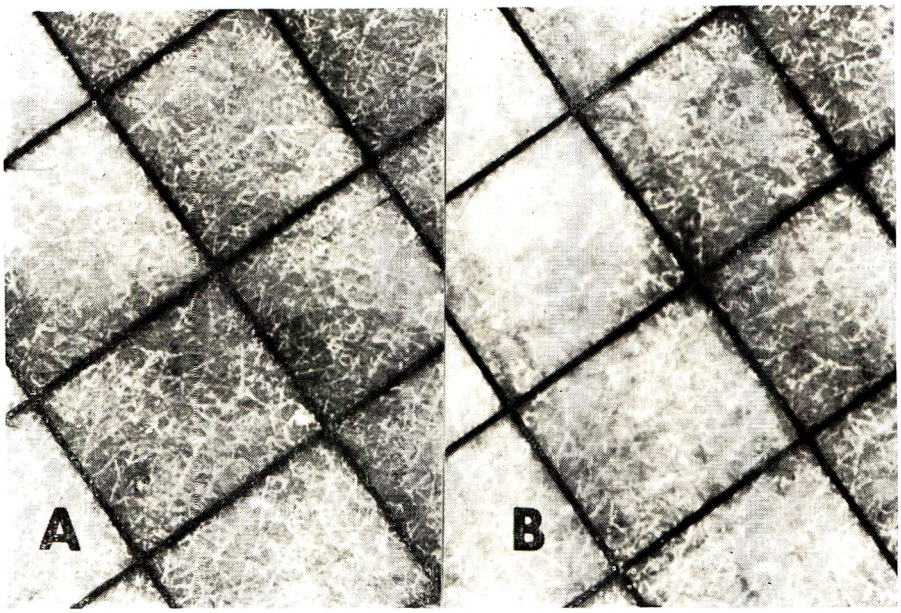

Figure 4. Repetitive photography of a site on Caucasian skin. Recorded with (A) a Kodak CC50C-cyan filter; (B) a yellow filter equivalent to Kodak 81C

a positive replica is made by melting polyethylene pellets° *in vacuo* at 180°C. After shadowing with Au-Pd to a thickness of 150 Å as was previously described (26), metal coated 9 mm punches of the polyethylene replicas are examined with a JEOL JSM-2† instrument at 30 to 3,000 X magnifications with accurate reproduction of details and greatly enhanced depth of field compared to conventional light microscopy.

Unmodified and solvent-extracted human skin *in vivo* have been examined in this manner (8). Impressions are obtained from the backs of panelists' hands before treatment in order to obtain control photographs. Half of each hand is then extracted with diethyl ether for 60 sec. The hands are dried and water-soaked with a damp flannel patch for 1 h. Following removal of the patches and blotting away the excess water with a paper tissue, Silastic‡ negative impressions are taken of the ether-water and water-only soaked areas. Together with the adjacent samples derived from unmodified skin, positive polyethylene casts are taken of each of the two extracted skin specimens.

°Union Carbide, Chemicals and Plastics Division, New York City, NY 10017.
†JEOL U.S.A. Inc., Medford, MA 02155.
‡Dow Corning Corp., Midland, MI 48640.

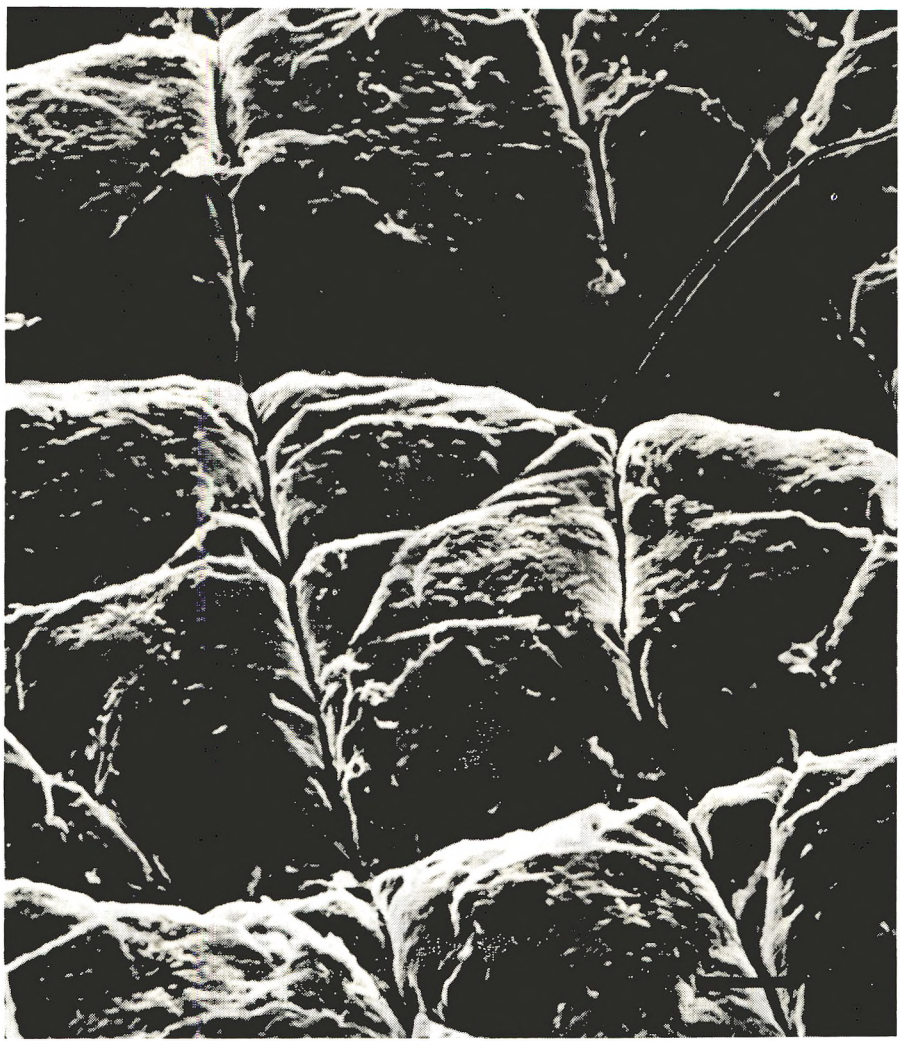

Figure 5. Scanning electron micrograph of untreated skin. Wolfram, Wolejsza and Laden (8). Bar represents 167μm

Photographs of these specimens under SEM examination reveal a surprising level of detail and contrast (8). Triangular, roughly rectangular, and polygonal cell clusters, having lengths of 600 to 1,200 μm, can be seen readily in the unmodified skin sample (Fig. 5). Earlier, Bernstein (28) demonstrated an enhanced rounding in the divisional contours with progressive increases in the degree of hydration. Water soaking the skin compared to the unmodified skin causes an increased plumping up of the subdivisional contours, while

Figure 6. Scanning electron micrograh of water-soaked skin. Wolfram, Wolejsza and Laden (8). Bar represents 167 μm

simultaneously enhancing the roughness and bumpiness within each cluster (Fig. 6). Ether extraction followed by water elution causes a further elevation in the height and number of protuberances (Fig. 7). The grooves between the subdivisions spread and have become accentuated, while the surface has become stretched and made taut. These SEM observations (8) strongly substantiate the conclusion drawn from gravimetric (1, 2) and biomechanical (7-9) analyses that removal of lipids and NMF results in enhanced water

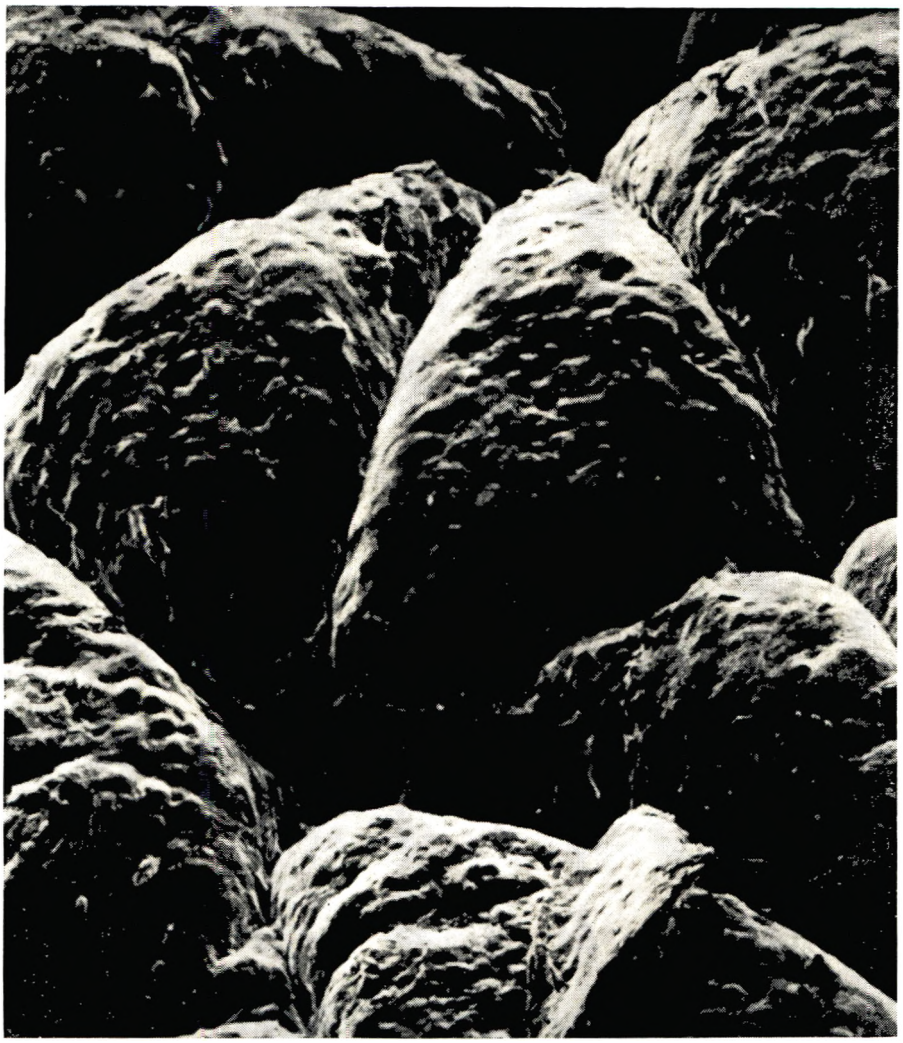

Figure 7. Scanning electron micrograph of ether-extracted and water-soaked skin. Wolfram, Wolejsza and Laden (8). Bar represents 167 μm

permeability, roughness, and swelling of the corneum cells concomitant with a weakening of the cellular membranes. This swelling, however, disappears within 1 h.

In order to avoid removal of desquamous material from winter-dried skin through adhesion to the Silastic polymer upon repetitive replication of a single area, we have routinely chosen to reproduce immediately adjacent

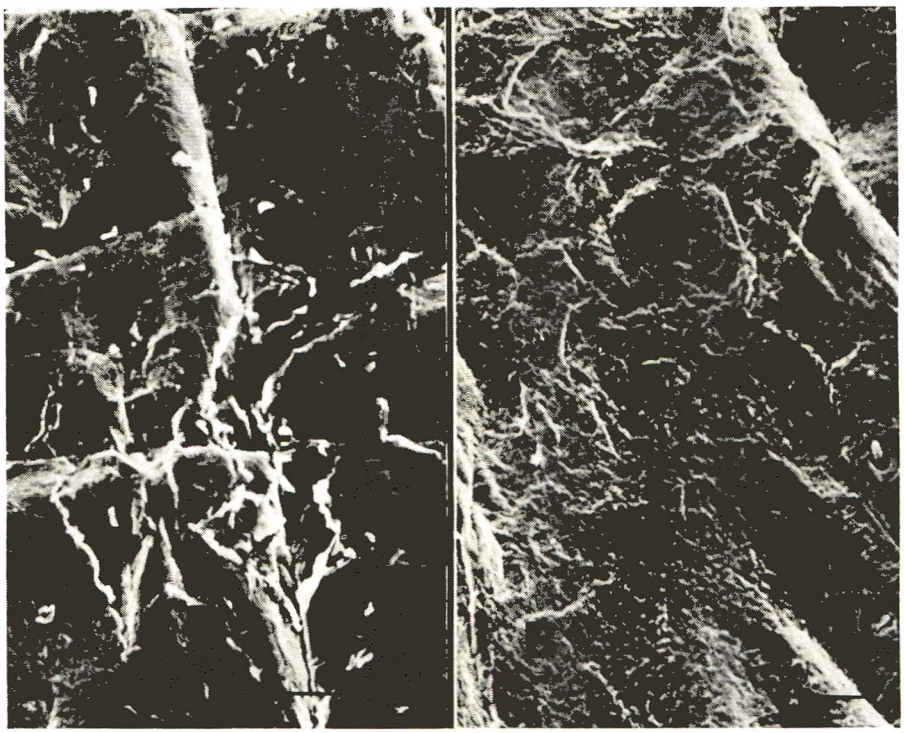

Figure 8. Scanning electron micrographs of immediately adjacent lateral sites on the calf of woman's leg. Each site was replicated simultaneously with (A) no treatment, and (B) 8 h after application of a commercial emollient cream. Each bar represents 167 μm

sites from grossly dry areas (e.g., the calf of a leg). A panel of women with chapped legs have recently been examined via this SEM technique before and up to 8 h after application of an emollient cream. Representative photographs of a single individual's dry untreated leg show a severely desquamous and fissured surface embossed with laminae of accumulated dried cells (Fig. 8(A)). At higher magnification, the shrunken, scaly and fractured vista is more pronounced (Fig. 9(A)). About 8 h after application of an emollient cream, the subdivisional interstices and white edges of uplifted corneum plaques had nearly disappeared, while the overall topography appeared to be partially coated and distinctly granular in texture (Fig. 8(B)). Enhanced magnification demonstrated the near absence of crevices, flaky edges and flatten scales, and accentuated a swollen, indented texture (Fig. 9(B)). An evaluation of the duration of relief and the efficacy of skin moisturization afforded by a product can thus be made by extending the periods of replication so as to provide several adjacent samples for an SEM time study.

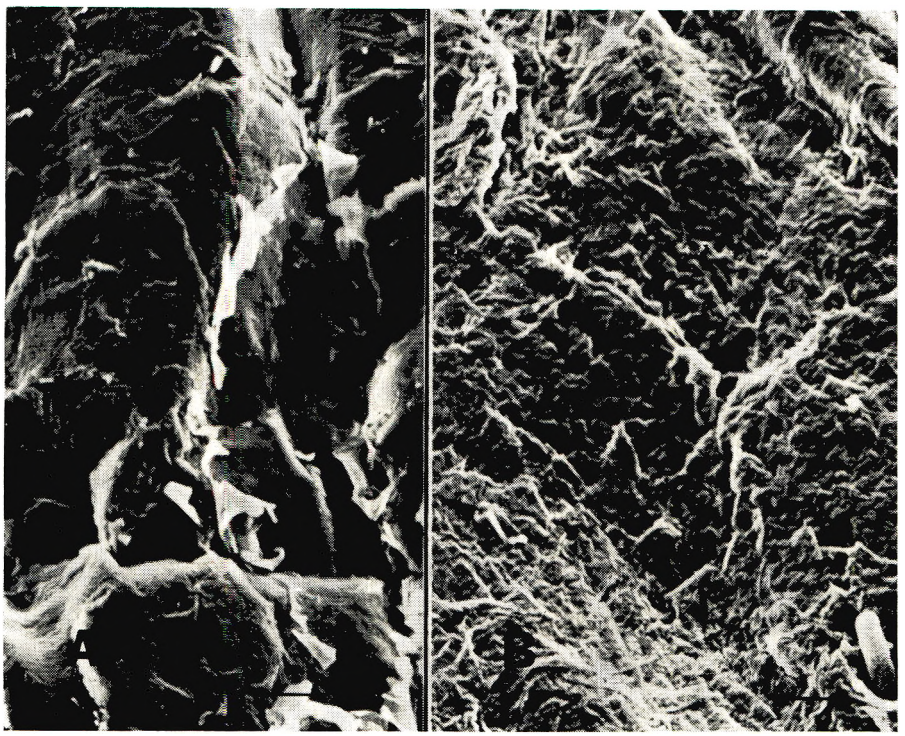

Figure 9. Higher magnification scanning electron micrographs of skin sites in Fig 8. Each bar represents 55.6 μm

Conclusions

In conclusion, the *in vitro* techniques of biomechanical, gravimetric and scanning calorimetric analyses provide valuable background information concerning the substances and dynamics involved in moisture uptake and retention by stratum corneum. Moreover, *in vivo* investigations by means of transpirometry and particularly low magnification photography and scanning electron microscopy are quite critical in the assessment of benefits derived from any cutaneous moisturizer or treatment. These latter methodologies afford a direct assessment of the physical condition of the living skin, and provide very nearly objective means for evaluating the efficacy of present and new formulations designed to moisturize or relieve dry and chapped skin.

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Book Reviews

HANDBOOK OF MOISTURE DETERMINATION AND CONTROL—PRINCIPALS, TECHNIQUES, APPLICATIONS, Vol 3, by A. Pande. Marcel Dekker, Inc., New York, 1975, XI + 289 pages. Price $33.50.

This is the third volume in a four volume series which uses continuous pagination. The index appears only in the final volume. Volumes 1 and 2 were reviewed in previous issues of the Journal (**26**,429 (1975); **27**,244 (1976)).

The four chapters contained in this volume are "Moisture in Textiles," Moisture in Bagasse, Wood, and Paper," "Moisture in Foods and Allied Agricultural Products," and "Moisure in Soils, Sands, Concrete and Silica, and Silicates." Although, each chapter does contain specific needs unique to that particular area of expertise, there is much of a general nature that is potentially of great interest to cosmetic chemists. The study of water in wool has always had obvious similarities in the hydration properties of human hair. Even some aspects of skin hydration find parallels in the dehydration and rehydration of polymers, paper, etc. Those working with talc and other silicates may find that some unique problems of moisture content may have already been resolved in very different contexts of other industries.

Obviously, the cosmetic chemist is probably not likely to find his specific application problem clearly laid out for him. He will, however, find similar problems and needs clearly developed. Recognizing the analogy, he may find an obvious solution to his specific need. Certainly, a perusal of these chapters should lead to a better understanding of equivalent truly cosmetic applications in moisture determinations in both finished product and in raw materials.

Although, the post 1965 literature may be minimal, it should be mentioned, as for a review of an earlier volume, that this is the apparent cut-off date for virtually all references.—JOHN H. WOOD—School of Pharmacy, Virginia Commonwealth University.

TOXICOLOGY ANNUAL 1974, Edited by Charles L. Winek. Marcel Dekker, Inc., New York, 1975, 344 pages (illustrated). Price: $29.50.

Toxicology Annual 1974 is a compilation of papers on selected topics of current interests and represents several disciplines covering a wide range of topics in the field of toxicology.

Included in the volume are articles on veterinary toxicology, narcotic drug dependence, the current status of saccharin, and postmortem drug level changes.

An excellent chapter by E. Buehler in this book, which is of interest to cosmetic chemists, is on test methods to predict potential occular hazards of household substances.

The index appears adequate, and the wide range of topics covered will make the book of interest to toxicologists in many fields. The articles are written by outstanding scientists and researchers, and the book should be a useful, though not vital, addition to the libraries of pharmacologists, toxicologists, physicians, pharmacists, and veterinarians.—CHARLES O. WARD, Ph.D.—Huntingdon Research Center.

THE THEORY AND PRACTICE OF INDUSTRIAL PHARMACY, 2nd Ed., by Leon Lachman, Herbert A. Lieberman, and Joseph L. Kanig. Lea and Febiger, Philadelphia, PA., 787 pages. Price $38.50.

The comprehensive coverage of the area of industrial pharmacy by 44 tenured nationally and internationally recognized experts including industrial scientists, pharmaceutical educators, and research and development managers makes this book exceptionally valuable for individuals seeking a thorough orientation in contemporary industrial practice. The editors, in this second edition of their book, include 4 new chapters discussing preformulation, production, packaging, and drug regulatory affairs, which supplement 22 extensively revised and rearranged chapters to reflect the numerous advances in the technology and regulatory activities affecting modern industrial pharmacy practices. All 26 chapters are extremely well illustrated with numerous charts, tables, diagrams, photographs, etc., and painstakingly referenced.

The chapters titled "Theories of Dispersion Techniques;" "Pharmaceutical Suspensions;" "Emulsions;" "Semisolids;" "Pharmaceutical Aerosols;" and "Sterile Products" should be of particular interest to cosmetic chemists who are involved in basic formulation work. Since these chapters stress the development of theoretical concepts and principles, rather than simply review the subject matter, they become extremely useful to the formulation chemists who desire to endeavor outside their own area of specific expertise. Lacking, however, from these chapters is a thorough discussion of the rheological properties governing polyphasic systems, and the cosmetic chemist should not turn to this book in search of a quantitative indepth interpreta-

tion or analysis of a material's texture or consistency qualities. The last four chapters of the book titled "Production Management;" "Packaging Materials Science;" "Quality Control: Process and Dosage Form;" and "Drug Regulatory Affairs," are well written without excessive detail and should prove to be useful and informative to those cosmetic chemists involved in the managerial and marketing aspects of cosmetic products.

There is some overlap of material in the book, but in view of the large number of notable contributors and the scope of the text, the editors should be commended for keeping it to a minimum. In summary, the book most certainly will well serve those involved in industrial practices and would be an excellent addition to their personal libraries.—ANTHONY J. CUTIE—Brooklyn College of Pharmacy.

LIPID CHROMATOGRAPHIC ANALYSIS, Vol. 1, 2nd Ed., Edited by Guido V. Marinetti. Marcel Dekker, Inc., New York, 1976. IX + 337 pages. Price $29.50.

The expanded second edition of this informative work consists of three volumes, which collectively represent an attempt to compile the major chromatographic methods in the lipid field.

Volume 1, reviewed here, contains six chapters by competent specialists. There is one chapter on column chromatography of neutral glycerides and fatty acids, which includes a brief discussion of the value of high-pressure liquid chromatography in lipid analysis.

Another chapter deals with gas chromatography of neutral acylglycerols. These include ordinary fats and oils (triglycerides) as well as any glycerol esters in which one or more hydroxyl groups have been combined with fatty acids and any remaining hydroxyl groups may be combined through an ether linkage to an aliphatic alcohol, aldehyde, or saccharide.

Other chapters cover thin-layer chromatography of phospholipids and glycolipids, the use of silica-gel-loaded-paper chromatography, the chromatographic analysis of alkyl ether lipids and their derivatives, and the analysis of phosphatides and glycolipids by chromatography of their partial hydrolysis or alcoholysis products.

Even though a major emphasis in this book is on elucidating the biochemical nature of complex fatty mixtures of natural origin, there is much material of interest to the cosmetic chemist. Most of the chapters have excellent detailed instructions and comments on experimental procedures, which are helpful to the practicing analytical chemist. There are ample literature references, and also a few typographical errors. An unfortunate deficiency is the absence of an index.—ALFRED WEISSLER—Consultant.

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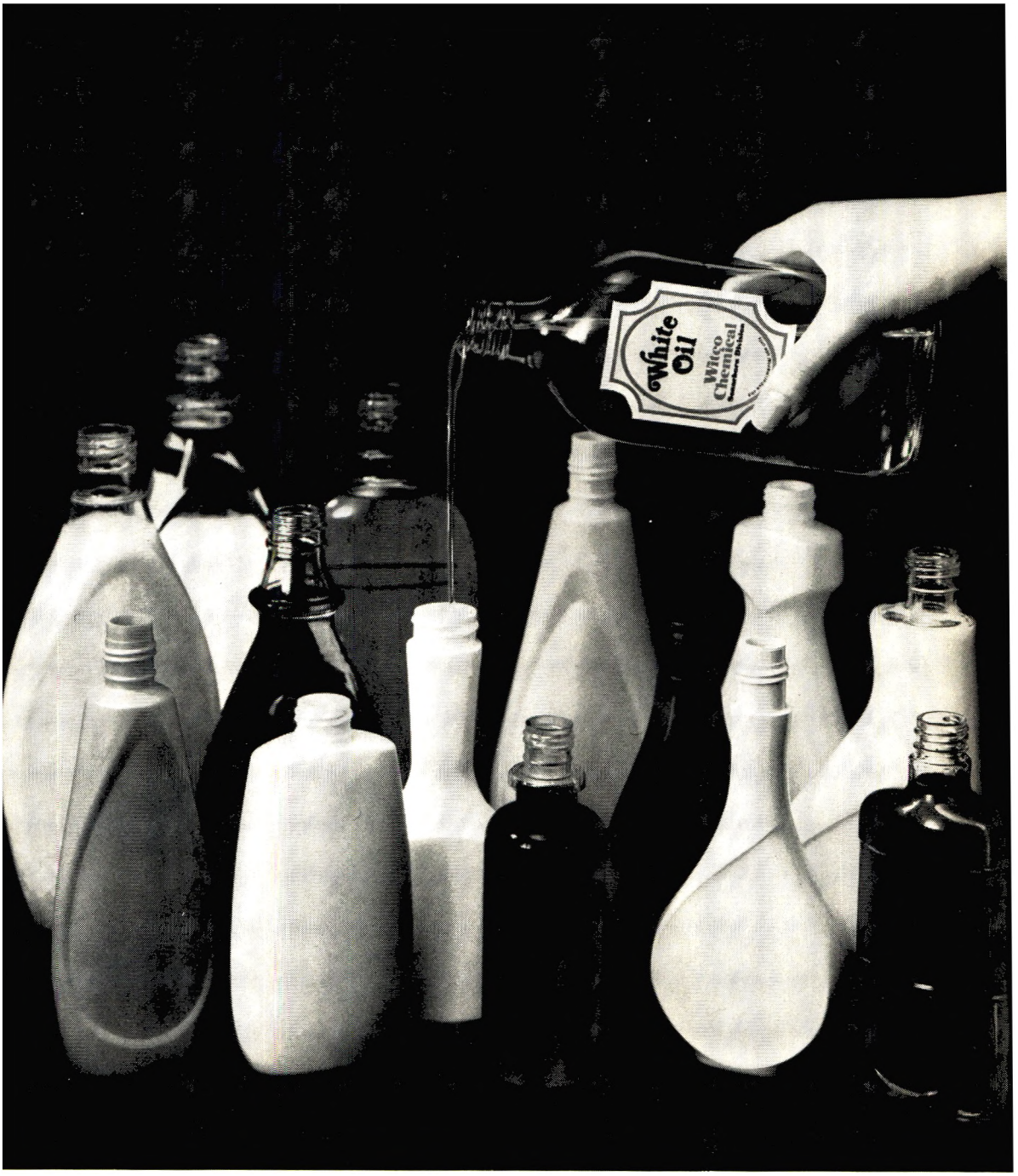

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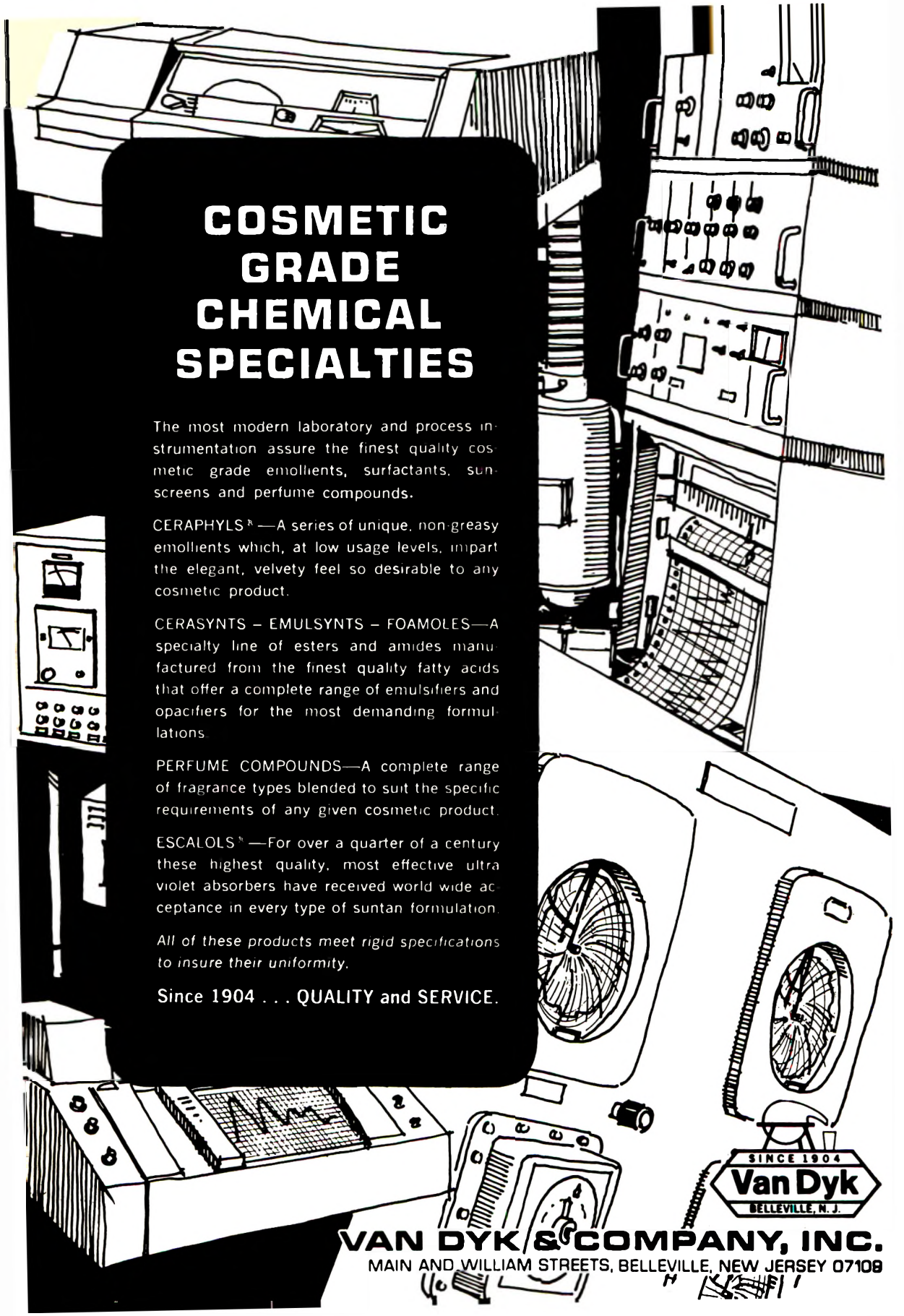

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