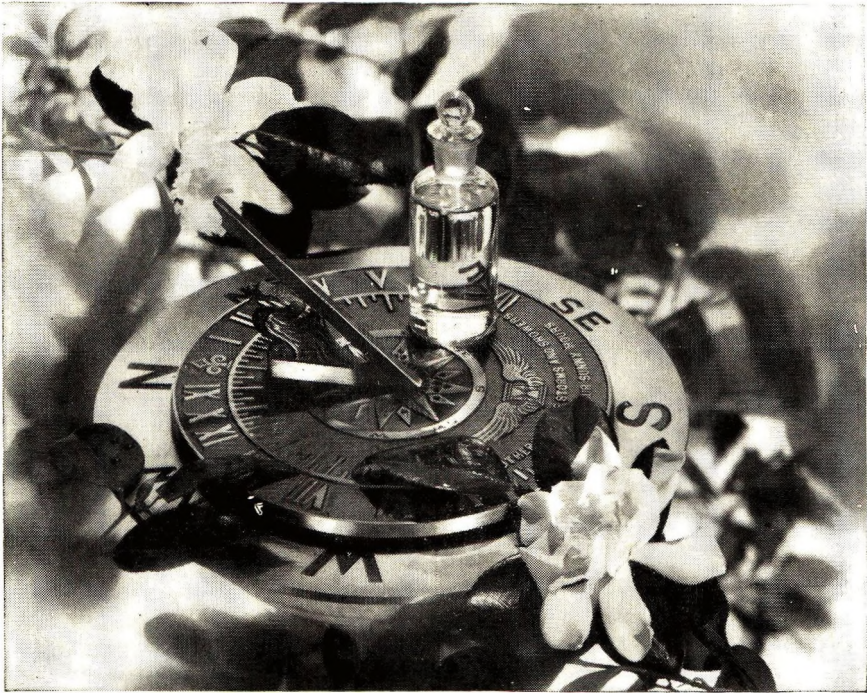


Journal of the Society of Cosmetic Chemists

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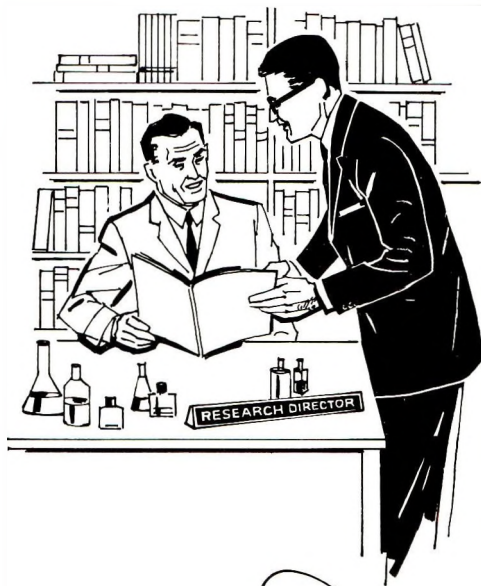
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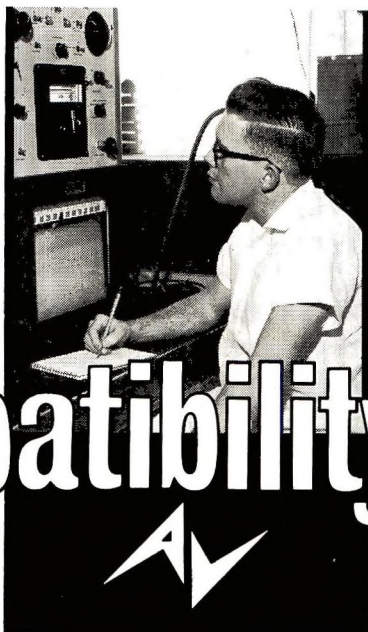
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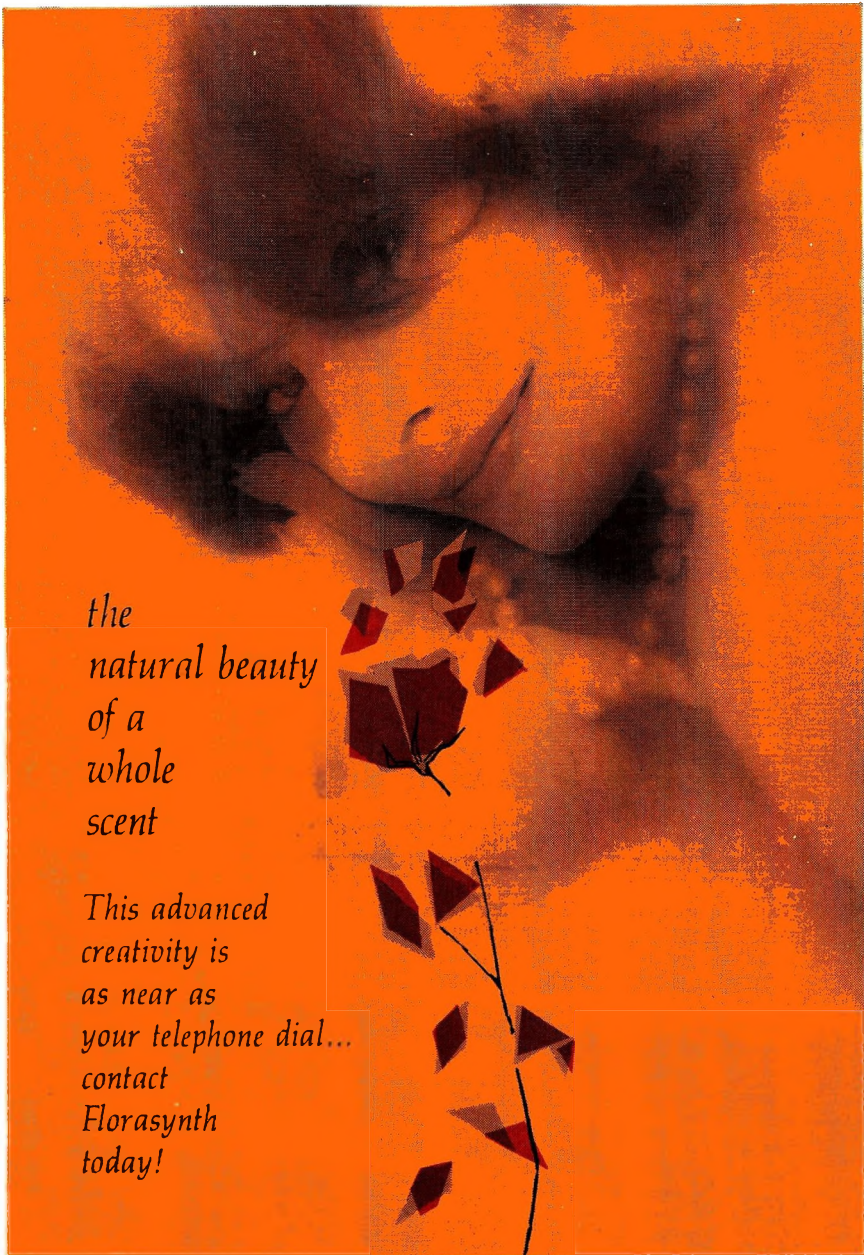


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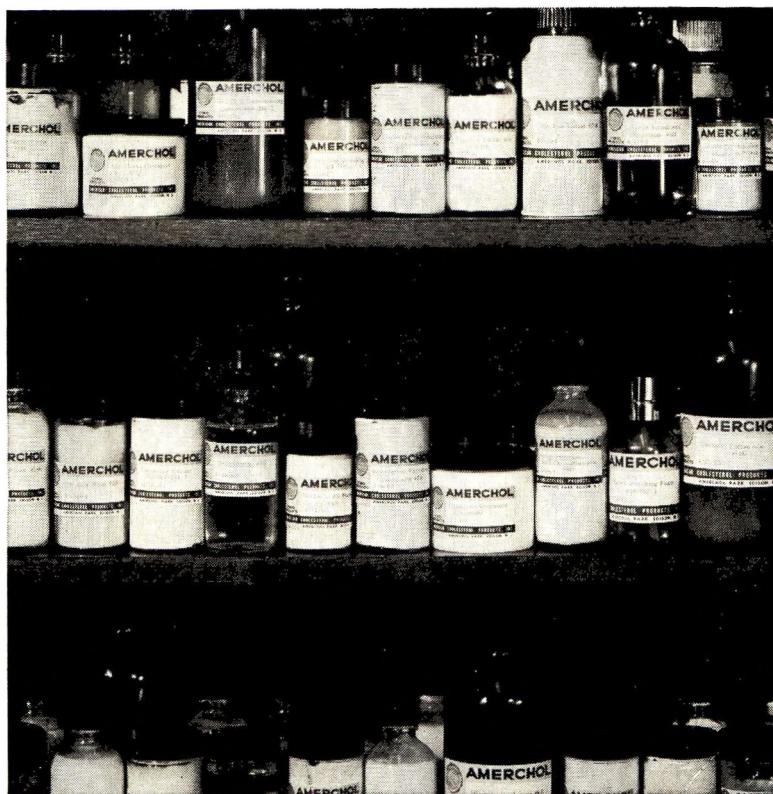
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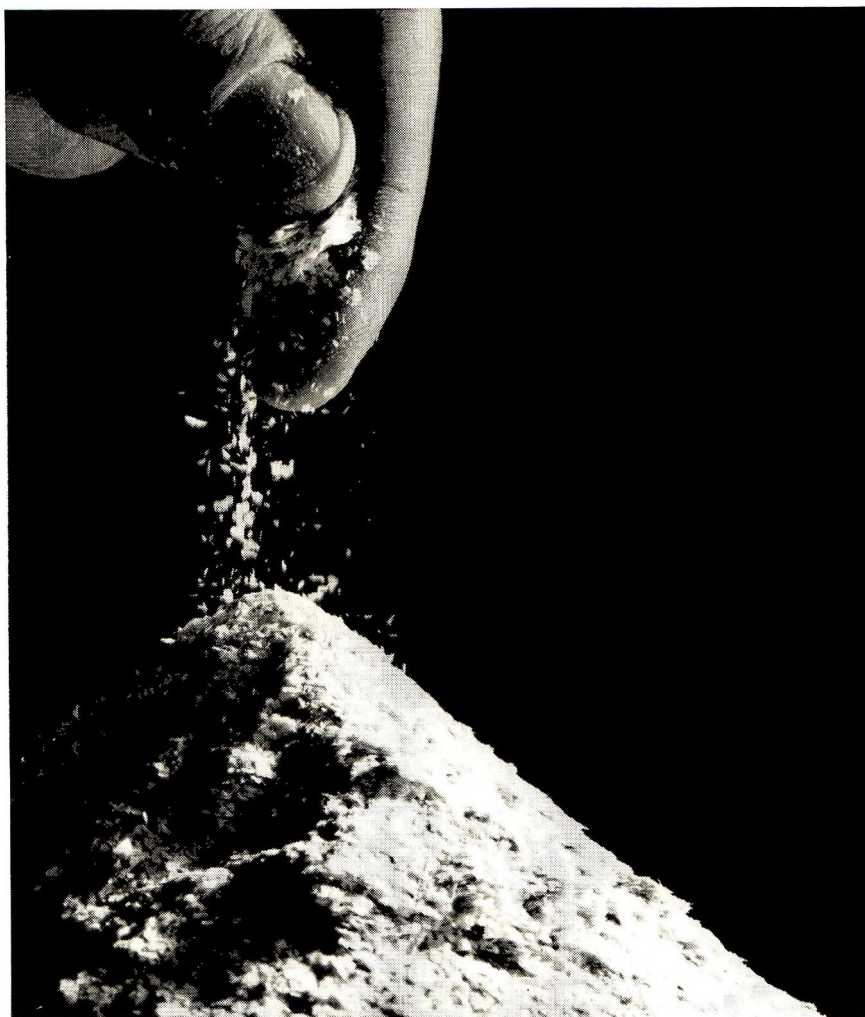
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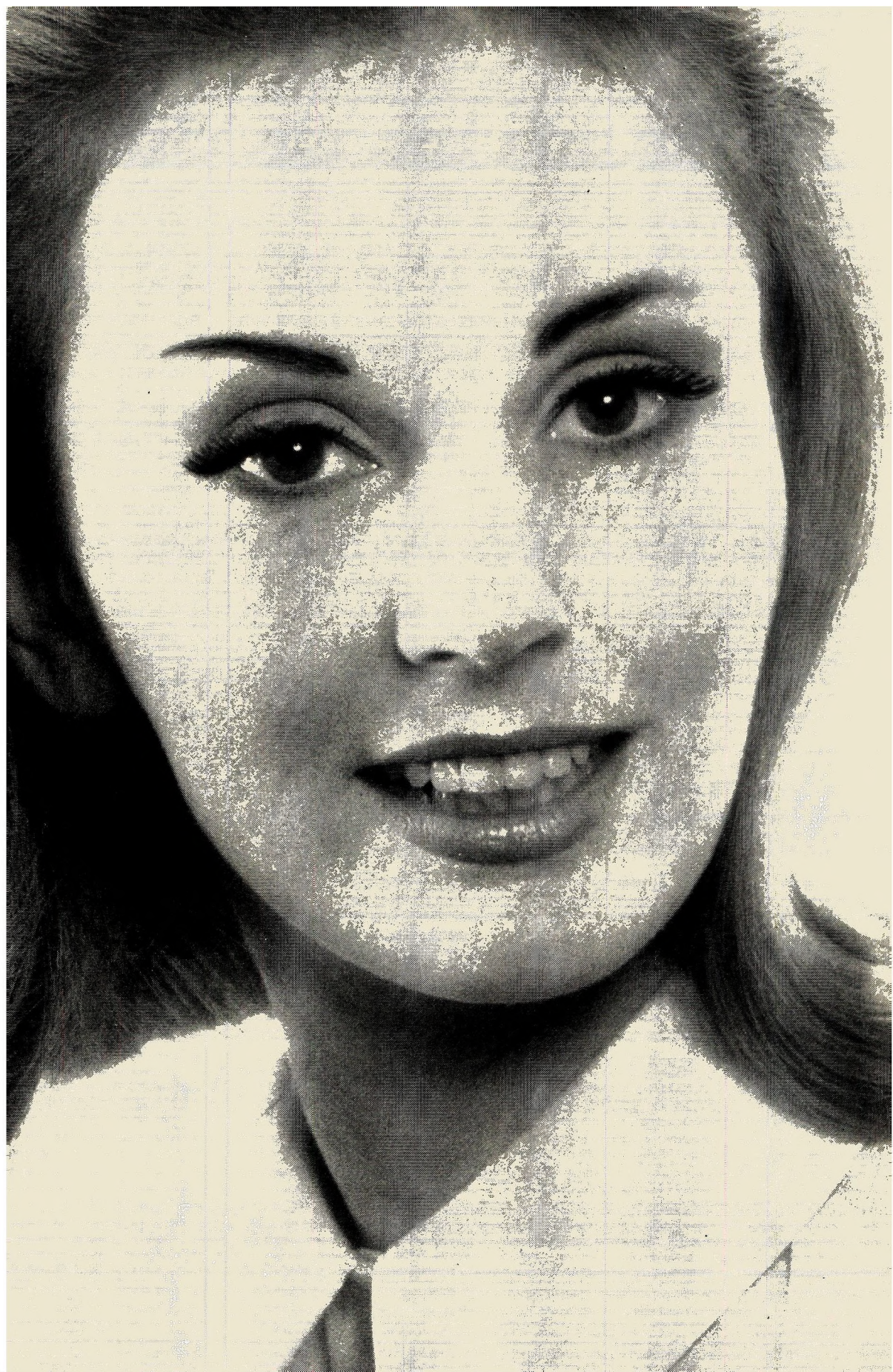
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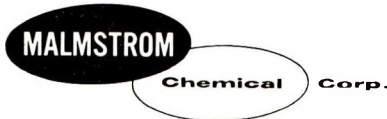
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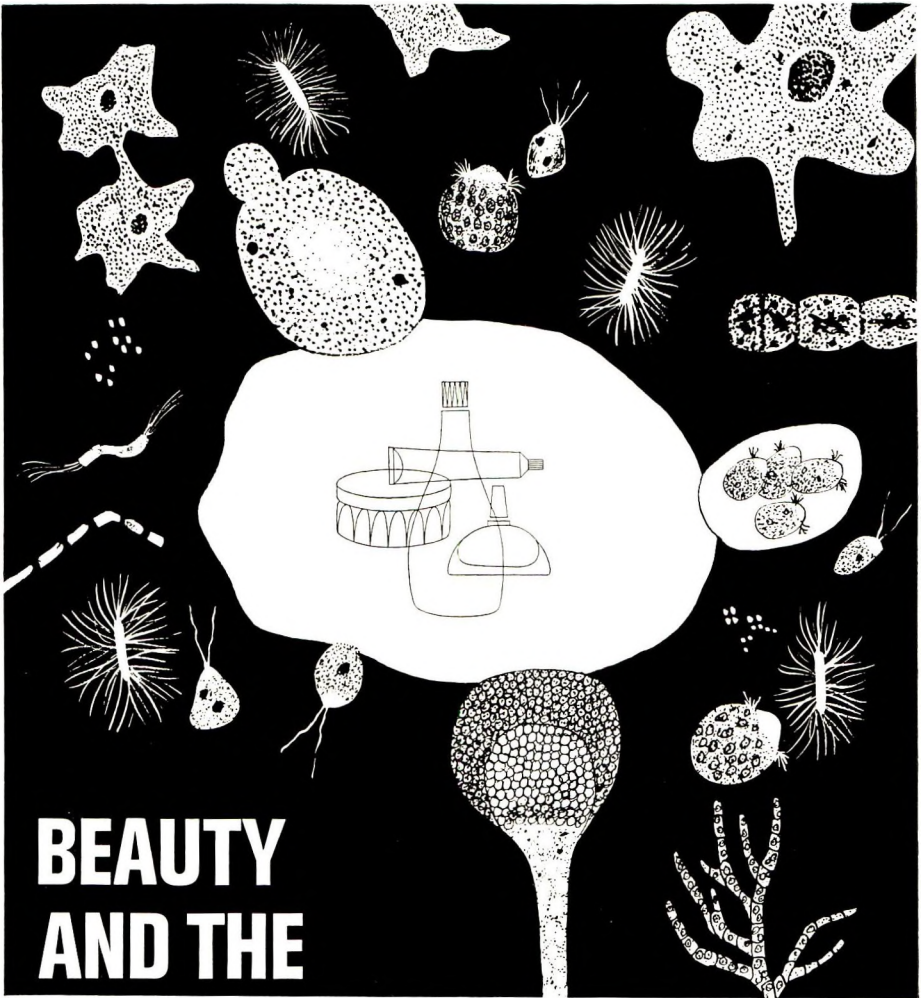
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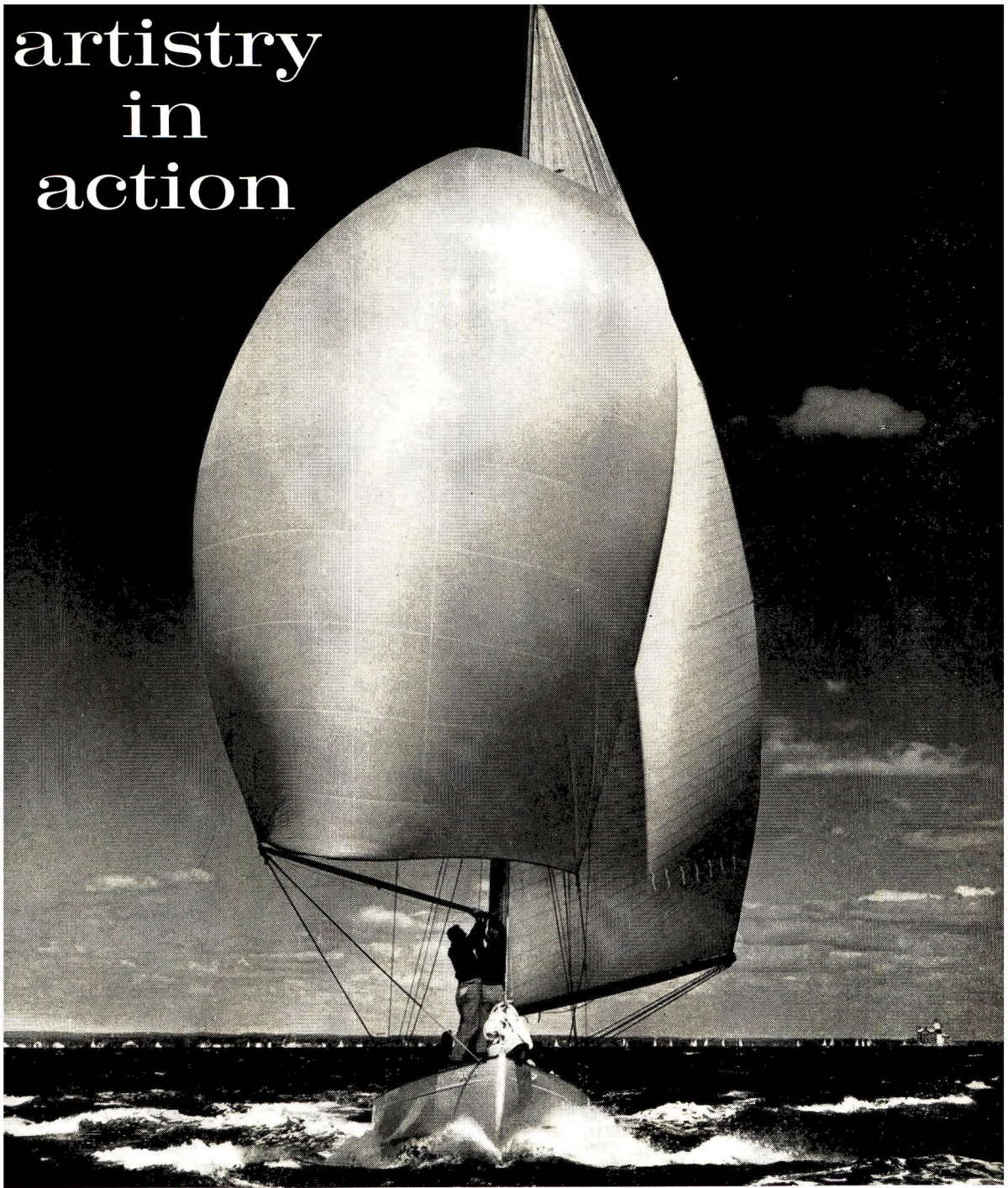
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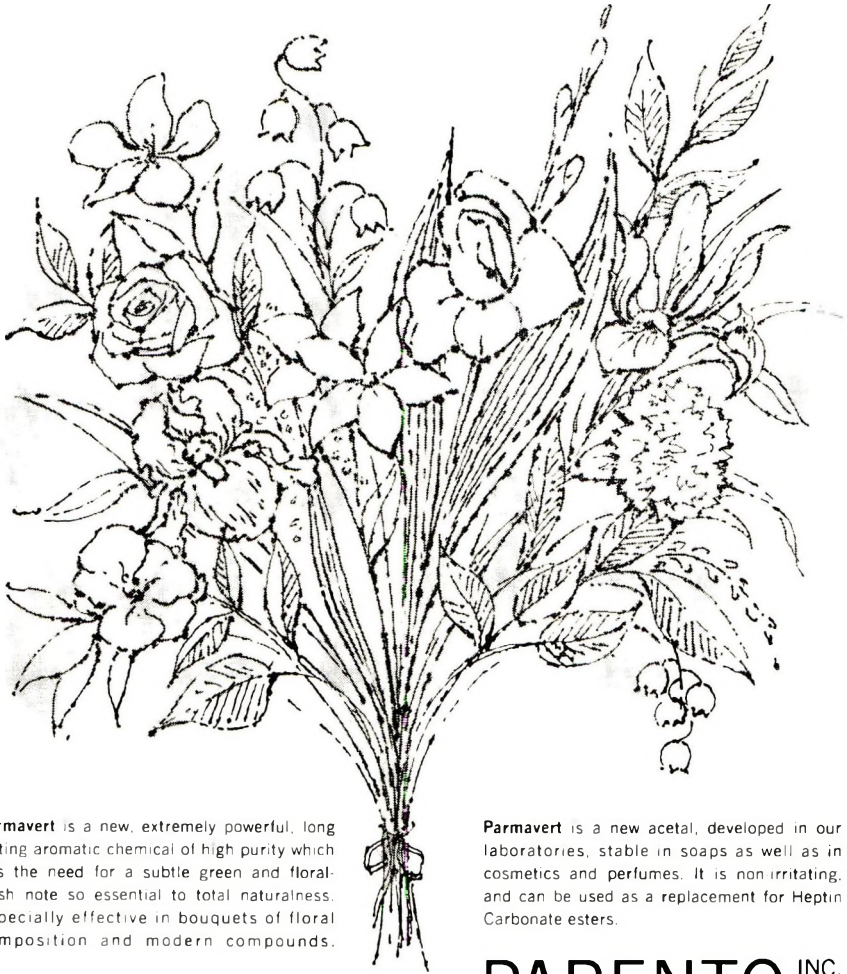
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SYNOPSIS FOR CARD INDEXES

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

Cosmetic uses of a new synthetic antifungal agent: F. Gialdi, A. Baruffini, R. Ponci, and P. Caccialanza. *Journal of the Society of Cosmetic Chemists* **17**, 575 (1966)

Synopsis—It is shown that 2,2'-dithiobis (*N*-butylbenzamide) is an effective topical antifungal agent for therapeutic, prophylactic, and cosmetic use. The results of clinical tests with cosmetic products containing this agent are detailed.

New evaluation techniques for sunscreens: Karson J. Master, Robert M. Sayre, and Mark Allen Everett. *Journal of the Society of Cosmetic Chemists* **17**, 581 (1966)

Synopsis—New techniques contributing to the laboratory evaluation of chemical, physical, and chemical/physical combination sunscreen products are presented. These enable determination of direct and total transmission, absorption, and reflection of ultraviolet and visible light through transparent or translucent thin films similar to those applied on the skin. The classical solution-dilution method using a Beckman Model DB Spectrophotometer and a new thin film technique using a xenon arc Monochromator and Cary Model 14 Spectrophotometer with integrating spheres were investigated. *In vitro* and *in vivo* measurements were made.

Hygroscopicity and hardness of hair spray resins at varying humidities: Morris J. Root and Stanley Bohac. *Journal of the Society of Cosmetic Chemists* **17**, 595 (1966)

Synopsis—An apparatus is described for measuring moisture pick-up, drying rate, and Sward Hardness of various resin films at relative humidities of from 50 to 90%. Data are given on films laid down from anhydrous SDA #40 ethyl alcohol. Resins used include: Polyvinyl Pyrrolidone (PVP K-30), Vinylpyrrolidone/Vinyl Acetate Copolymer (PVP/VA 735), VEM Resins, 90, 50, and 0% neutralized with 2-amino-2-methyl-1,3-propanediol (AMPD), and National Starch Resyn 28-1310, 90, 70, and 0% neutralized with AMPD. One set of data used the unplasticized films, and a second set used the film plasticized with 3.5% DC-555 Silicone Oil. Although this apparatus has been useful for collecting data on resins used in hair sprays, the method and apparatus can be used for obtaining data with these and other resins for applications such as adhesives and coatings.

Evaluation of certain factors influencing oil deposition on skin after immersion in an oil bath: M. E. Stolar. *Journal of the Society of Cosmetic Chemists* **17**, 607 (1966)

Synopsis—Previously developed methods for determining quantitatively the amount of oil and recording the oil film deposited on the skin after immersion in oil baths were utilized to evaluate various oils of potential use in bath oil formulations. The effect of the presence of various surfactants in mineral oil on the amount and skin coverage of the deposited oil film was determined. The existence of a relationship between spreading coefficient values and the amount of skin coverage of the deposited oil film were investigated.

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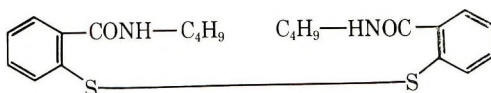
Cosmetic Uses of a New Synthetic Antifungal Agent

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ORD.‡

Synopsis—It is shown that 2,2-dithiobis (*N*-butylbenzamide) is an effective topical antifungal agent for therapeutic, prophylactic, and cosmetic use. The results of clinical tests with cosmetic products containing this agent are detailed.

INTRODUCTION

For several years, studies have been carried out toward the synthesis of antifungal agents related to aromatic sulfides (1, 2). Among the new substances synthesized, 2,2-dithiobis[*N*-butylbenzamide] (OD-507) has been found to be particularly interesting:



This substance possesses high antifungal activity and is also active against some bacteria. Table I gives some data on minimum inhibitory concentrations of this compound. Comparative microbiological tests have demonstrated that OD-507 possesses activity equal or superior to that of the best known natural or synthetic antifungal agents (2).

Pharmacological studies on the toxicity of OD-507 have revealed that this substance is very well tolerated (3). Furthermore, a series of

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Table I
Minimum Inhibitory Concentrations

Microorganism	MIC (mcg./ml.)
<i>Trichophyton mentagrophytes</i> (ATCC 8757)	1
<i>Trichophyton mentagrophytes</i> (ATCC 9129)	1
<i>Epidermophyton floccosum</i> (ATCC 10227)	2
<i>Microsporum audouini</i> (ATCC 9079)	5
<i>Microsporum gypseum</i> (ATCC 11658)	5
<i>Microsporum canis</i> (ATCC 10214)	2
<i>Candida albicans</i> (ATCC 10231)	10
<i>Candida albicans</i> (LCP)	5
<i>Candida tropicalis</i> (ATCC 1369)	1
<i>Saccharomyces cerevisiae</i> (ATCC 9763)	10
<i>Saccharomyces carlsbergensis</i> (ATCC 9080)	5
<i>Kloeckera brevis</i> (ATCC 977)	0.5
<i>Cryptococcus neoformans</i> (SKF 1110)	2
<i>Nocardia asteroides</i> (CBS)	5
<i>Staphylococcus aureus</i> (ATCC 6538)	2
<i>Streptococcus pyogenes</i> (C-203)	1
<i>Streptococcus faecalis</i> (ATCC 10541)	5
<i>Bacillus subtilis</i> (ATCC 6633)	2
<i>Pseudomonas aeruginosa</i> (ATCC 10145)	20
<i>Mycobacterium tuberculosis</i> (H 37 RV)	1

experimental "patch tests" and "scratch tests" in humans indicated no irritation or sensitization reactions by this substance.

In view of all these facts, the utility of OD-507 as an antifungal medication for dermatological use was studied in a preliminary clinical trial, and the results obtained have been reported (2). The substance has proved to be generally well tolerated both by healthy human skin and in areas of epidermomycotic lesions. The antifungal activity of OD-507 tested in various types of epidermomycotic infections (inguinal epidermophytosis, erythrasma, athlete's foot, etc.) has been found to be extensive and efficient, superior or comparable to that of the best-known modern therapeutic antifungal preparations.

PURPOSE OF STUDY

All the results of studies so far performed emphasize the fact that OD-507 is both highly active and always well tolerated. In fact, the rare cases of intolerances noted in the earlier clinical experiments (2) have turned out to be related to the composition of the vehicle, not to the active antifungal agent. The study of OD-507 has now been extended to the cosmetic field. The basic requirement for the application of an active substance in cosmetics is its tolerance over prolonged periods

of use. In other words, the possibility of including OD-507 in a series of preparations, either as a prophylactic against widespread epidermomycotic states or as a useful adjuvant to therapeutic treatment, was studied.

EXPERIMENTAL

Three preparations were used: a body powder, a solid skin detergent, and a shampoo.

The body powder was formulated with 2% micronized OD-507 in a base consisting of talc, isopropyl myristate, and Carbowax 1500 to assure adhesion of the preparation.

The solid skin detergent was formulated with the objective of using detergents capable of giving aqueous solutions at weakly acid pH values. A solid skin detergent based on anionic surfactants, polyethylene glycols, alcohols, and higher fatty acids was therefore prepared. Lactic acid was used to achieve the acidic pH. OD-507 was incorporated at a concentration of 2%. This preparation, dispersed in water at 1% concentration, gives a pH of 6.5.

The shampoo was formulated at a neutral pH, choosing anionic and non-ionic surfactants as detergents. The OD-507 was incorporated at 0.5% concentration. The formulations are shown below:

Body Powder

Talc USP XVII	95.0 g
Isopropyl myristate (Italcolloid-Italy)	1.0 g
Polyethylene glycol 1500 (Carbowax 1500; Union Carbide-USA)	2.0 g
OD-507	2.0 g

Solid Skin Detergent

Sodium laurylsulfate (Empicol L.Z.G.V.; Marchon Italiana-Italy)	11.0 g
Sodium <i>N</i> -oleyl- <i>N</i> -methyltaurate (Arkopon T.; Hoechst-Germany)	31.0 g
Stearic acid	16.5 g
Stearyl alcohol	12.0 g
Polyethylene glycol (Polyglycol E 4000; Dow Chemical-USA)	11.0 g
Talc USP XVII	16.0 g
Lactic acid	0.5 g
OD-507	2.0 g

Shampoo

Sodium laurylpolyoxyethylene sulfate (Salvo E020; Chemy-Holland)	35.0 g
Polyoxyethylene stearate (Myrj 52; Atlas-U.S.A.)	5.0 g
Sodium <i>N</i> -alkyl- <i>N</i> -methyltaurate (Hostapon C.T.; Hoechst-Germany)	10.0 g
OD-507	0.5 g
Water to make	100.0 ml

RESULTS

The cosmetic preparations under test were used by subjects suffering from a variety of clinical skin mycoses which, according to past personal history, had all chronically relapsed. The case list was, therefore, a selected one and comprised primarily refractory patients.

The *first group* consisted of 18 subjects, all suffering from dermatophytosis between the toes (athlete's foot) chronically relapsing during the spring/summer season for three to five years.

After complete clinical recovery (through use of OD-507 cream), 12 of these subjects started maintenance treatment using the solid detergent two to three times weekly and applying the talcum powder daily or on alternate days. The other six subjects were not treated with the cream but instead started prophylactic treatment with the solid detergent and the talcum powder at the start of spring, a period which, according to past personal history, consistently coincided with relapse of the mycosis. The results indicate that no fungal manifestations reappeared in any of the 18 subjects so treated. Patient tolerance of the solid detergent and the talcum powder was excellent.

The *second group* consisted of 11 subjects with inguinal epidermomycosis (bilateral in 9 cases) which had repeatedly relapsed for years. All subjects applied the detergent and talcum powder systematically every day for a long period instead of ordinary soap and bath talc. As in the first group, no relapses occurred in any of the cases.

The *third group* comprised ten subjects suffering from *pityriasis versicolor* which consistently relapsed in spite of varied and prolonged earlier treatments. In several cases the lesions still showed obvious fluorescence under ultraviolet light. All ten subjects made systematic and prolonged use of the solid detergent and talcum powder under test. None of the patients showed relapse of the chromophytosis.

The *fourth group* comprised seven obese subjects, suffering from relapsing candidiasis of the large body folds. Two cases suffered from

diabetes, and all subjects suffered from frequent and abundant sweating. Regular use of the solid detergent and of the talcum powder under test gave excellent, rapid and long-lasting results. As above, tolerance of the preparations was excellent.

The *fifth group* comprised six cases of severe chronic seborrheic dermatitis of the mid-chest in adults. Systematic and prolonged use of the solid detergent and talcum powder led to recovery from the symptoms.

The *sixth group* comprised six cases of so-called dry pityriasis of the scalp, nine cases of pityriasis steatoides of the scalp, and two cases of tinea amiantacea. All of these subjects, many of whom had had severe relapses or were chronic, greatly benefited from use of the shampoo. This preparation, included as an adjuvant with other routine medications (UV rays, corticosteroids, etc.), was found to be a practical and beneficial topical therapeutic means. After termination of clinical treatment, the regular use of the shampoo (instead of the usual shampoos) favored continuing recovery and evidently prevented the onset of the easily occurring relapses in all subjects.

Throughout these clinical studies, the use of placebos as controls was not considered necessary.

CONCLUSIONS

The clinical results obtained in the 67 cases studied confirm that OD-507 is active and well tolerated. It may be considered a synthetic antifungal agent which is perfectly suitable also for the formulation of cosmetic preparations.

Formulations such as those tested above have been shown to be extremely efficient prophylactics against several frequently relapsing mycotic states. These preparations are also believed to be useful adjuvants for use during medical treatment of mycotic infections.

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Translations Available

English translations of the following papers may be obtained by writing to Mr. Robert A. Kramer, Evans Chemicals, Inc., 250 East Forty-third Street, New York, New York 10017.

“Studies of the Phenomenon of Permanent Waving of Human Hair,”
by Dr. Hans Freytag.

“Alteration of Hair Keratin by Cosmetic Processing and Natural
Environmental Influences,” by Dr. Peter Berth and Dr. Gunter
Reese.

“New Information about the Morphological Structure of the Hair,”
by Dr. Rudolf Randebrock.

“The Application of the Analytical Methods of Sulfur Chemistry to
Permanently Waved Hair,” by Prof. Dr.-Ing Helmut Zahn, Dr.
Tarsilla Gerthsen, and Dipl.-Chem. Marie-Luise Kehren.

New Evaluation Techniques for Sunscreens

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and MARK ALLEN EVERETT, M.D.†

Presented December 1, 1965, New York City

Synopsis—New techniques contributing to the laboratory evaluation of chemical, physical, and chemical/physical combination sunscreen products are presented. These enable determination of direct and total transmission, absorption, and reflection of ultraviolet and visible light through transparent or translucent thin films similar to those applied on the skin. The classical solution-dilution method using a Beckman Model DB Spectrophotometer and a new thin film technique using a xenon arc Monochromator and Cary Model 14 Spectrophotometer with integrating spheres were investigated. *In vitro* and *in vivo* measurements were made.

INTRODUCTION

Sunburn, photosensitivity reactions, aging, and carcinogenesis are some of the undesirable cutaneous responses to sunlight most commonly encountered by the dermatologist. Blum (1), Bachem (2), Everett (3), and Knox (4) have reviewed the physiological and the pathological cutaneous reactions to sunlight resulting from its broad action spectrum from the ultraviolet through the visible (290 to 740 $m\mu$). In recent years, considerable activity has been evidenced in the cosmetic and pharmaceutical industry in developing products capable of efficiently screening out the harmful portion of the spectrum of light. This has resulted in the investigation of more effective and broad spec-

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trum physical and chemical sunscreens and their combinations in cosmetically elegant and functionally effective bases. Laboratory evaluation of such products, utilizing the classical methods, has failed to keep pace with the progress achieved in other areas in the field. Presented below are studies leading to a more adequate and meaningful evaluation of sunscreens and techniques devised to obtain representative, practical, and meaningful information regarding their physiological effectiveness.

CHEMICAL AND PHYSICAL SUNSCREENS

Chemical sunscreens are agents that protect the skin by absorbing and dissipating the energy of the damaging ultraviolet sunburn rays. Organic compounds which have absorbed radiation are either raised to a higher energy level or dissociated. The excited molecules may dissipate their absorbed energy by collision, fluorescence, or a reaction with other molecules at collision (5). *p*-Aminobenzoic acid and its derivatives have been the most widely employed suncreening agents.

Physical sunscreens (sunshades) are opaque, usually insoluble chemicals that produce their effect by reflecting and scattering light. Examples are titanium dioxide, talc, kaolin, zinc oxide, and bentonite. Occasionally these agents are used in combination with chemical sunscreens to complement the protection afforded by the latter. Even a cream vehicle will sometimes provide protection.

In this study the following experimental sunscreen products were studied:

- A. *Chemical Sunscreen:*
 - 7.0% *p*-aminobenzoic acid in a clear viscous solution
- B. *Physical Sunscreens:*
 - (1) Opaque cream base
 - (2) 10% talc in opaque cream base
- C. *Chemical Physical Sunscreen:*
 - 5% amyl *p*-dimethylaminobenzoate and 10% talc in opaque cream base

SOLUTION-DILUTION METHOD

The current laboratory method of evaluating sunscreens involves quantitatively dissolving the finished product containing the sunscreen in an ultraviolet-transparent solvent such as isopropanol, filtering the solution, preparing dilutions, and measuring transmission (6).

Figure 1 shows the transmission curves determined for the experi-

mental sunscreen products by this solution-dilution method on a Beckman Model DB spectrophotometer.

Disadvantages of this method are: (a) Only the chemical sunscreen and isopropanol-soluble components in the product are extracted from the finished product; the insoluble components of the base are left behind. (b) Since the physical sunscreen is usually insoluble in the solvents used, it is not extracted and therefore cannot be evaluated. Thus,

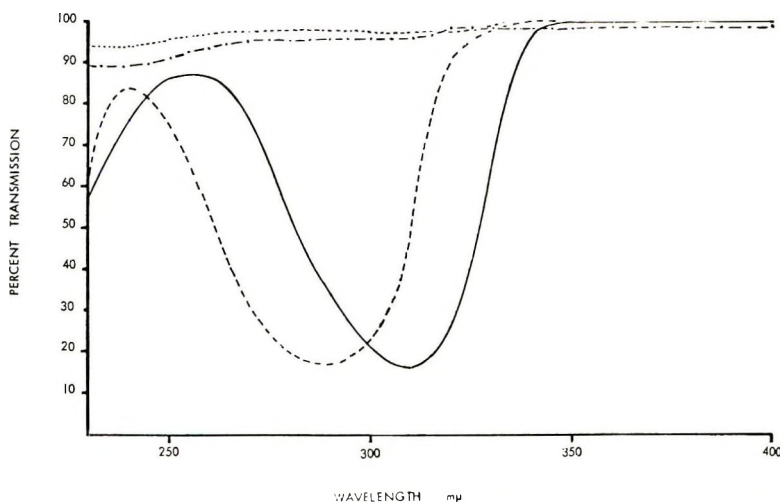


Figure 1. Ultraviolet transmission of sunscreen products using solution-dilution method and spectrophotometer. --- 7% *p*-aminobenzoic acid in clear viscous solution (0.069672 g/l of isopropanol); - - - 5% amyl-*p*-dimethylaminobenzoate + 10% talc in opaque cream base (0.12746 g/l of isopropanol); - · - · - 10% talc in opaque cream base (0.09929 g/l of isopropanol); and - - - opaque cream base (0.10914 g/l of isopropanol)

in case of a chemical/physical sunscreen combination, the total sun-screening capacity of the product cannot be determined. (c) The product is evaluated in a condition and concentration entirely different from those used on the skin. (d) The procedure is quantitative but involved and time consuming.

THIN FILM TECHNIQUE

This study, designed to avoid the disadvantages of the solution-dilution method, has resulted in the development of the Thin Film Technique. It consists essentially of sandwiching a thin film of the sunscreen product between two quartz slides separated by means of spacers of known thickness. The quartz slides* are ground and polished on

* Arthur H. Thomas Co., Philadelphia 5, Pa.

both sides, measure 2.54×5.08 cm, and have a thickness of 1 mm with a tolerance of 0.000–0.001 mm. The spacers consist of undistorted 0.0254 ± 0.00254 mm thick No. 1100-0 alloy aluminum foil* cut into strips 3.2 mm wide \times 25.4 mm long. The complete sunscreen product was used as such without any processing. A clean, quartz slide was placed on a hard, flat surface. Two strips of foil spacers were cut carefully to avoid distortion and placed on the slide at either end. A small amount of product was placed in the center of the slide. Another quartz slide was aligned and placed on this slide carefully, starting from the center and working away from it. Both ends of the slide were then firmly pressed simultaneously. A uniform film of the product showed in the center of the slide sandwich. While pressure was maintained, the slides were then taped† to one another at both ends above the foil locations. This thin film specimen was then ready for spectrophotometric, monochromatic, reflection, and other measurements. A blank was prepared in the same manner without the product. In the studies that follow, thin film specimens prepared as above were used.

Monochromator Studies

A high intensity xenon arc ultraviolet light monochromator system described by Sayre *et al.* (7) was used in conjunction with an RCA 935 photocell and specially constructed quartz cuvette/quartz slide holder for measurement of the transmission spectra of the sunscreen products. The technique utilized was to measure the output of the source from 250–400 $m\mu$ wavelength in 10 $m\mu$ increments, with blank quartz slides in the slide holder. Next, quartz slides with the thin film of the sunscreen product sandwiched between, as described above, replaced the blanks in the holder, and the light transmitted was measured by the photocell. The output of the source was again measured with the blank slides in the holder. Any significant change in the intensity of the source would thereby be detected; during the experiments the intensity, caused by wandering of the plasma in the arc, was found to vary up to 10%. The values of the light transmitted by the blank and sample are both expressed in volts; thus the ratio of sample to blank in volts gives the light transmitted by the sample. Transmission measurements were made for all the experimental sunscreen products. These are shown in Figs. 2 and 3 as "monochromator direct transmissions."

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† Scotch Brand, Minnesota Mining and Manufacturing Co., St. Paul, Minn.

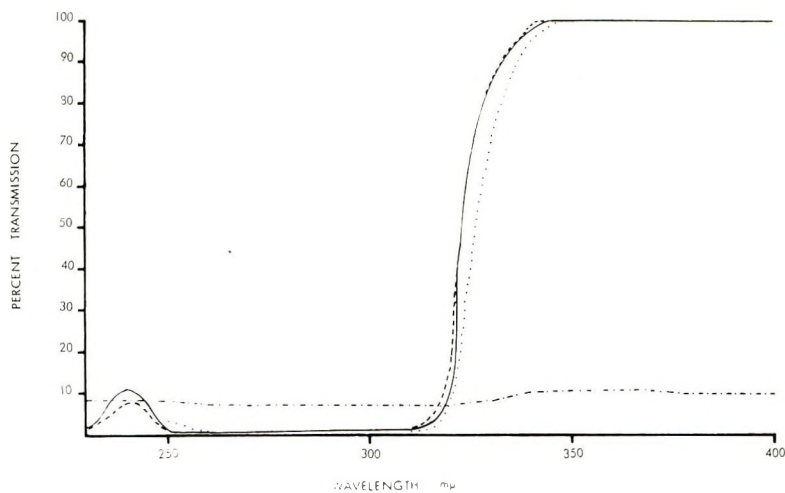


Figure 2. Ultraviolet transmission and reflection of 0.0254 mm thick film of 7% *p*-aminobenzoic acid in clear viscous solution. Using spectrophotometer with integrating spheres: — total transmission, - - - direct transmission, and - · - · - percent reflection. Using monochromator: · · · direct transmission

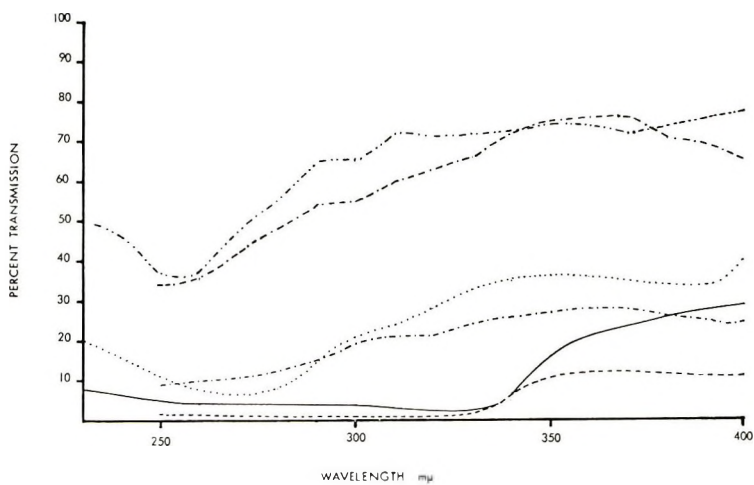


Figure 3. Comparison of two methods for determining transmission on 0.0254 mm thick films of three sunscreen products. Total transmission using spectrophotometer with integrating spheres; direct transmission using monochromator. 5% amyl-*p*-dimethylaminobenzoate + 10% talc in opaque cream base: — total transmission and - - - direct transmission. 10% talc in opaque cream base: · · · total transmission and - · - · - direct transmission. Opaque cream base: - · - · - · total transmission and - - - - - direct transmission

Studies with Integrating Spheres

While the use of the monochromator and photocell indicated that a considerable portion of the light was scattered in a forward direction by samples containing both chemical and physical sunscreens, the total amount of this forward scattered light could not be evaluated due to the geometry of the equipment. Since the phototube subtends an angle of 26 degrees with the sample, only light within 26 degrees normal to the sample was measured.

Integrating spheres (Cell Space Reflectance Accessories*) were employed to adapt the Cary Model 14 Spectrophotometer for measurement of the total transmission of light (8). In addition to measurement of the total quantity of light scattered in a forward direction

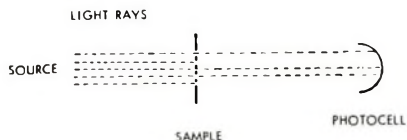


Figure 4. Direct transmission—spectrophotometer

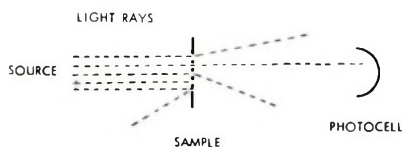


Figure 5. Direct transmission—spectrophotometer with sample that scatters and transmit light

through the specimen, this instrumentation eliminated several errors in measurements due to the monochromator and photocell combination. First, this is a double beam instrument; second, its optics were designed for exact spectrophotometric measurements; third, its readout is automatically recorded; and fourth, its spectral range permits greater information to be obtained about each specimen. In addition to being able to measure the forward scattered light, the reflected or backward scattered light could also be measured so that the optical properties of each sunscreen were more fully determined.

All the thin film specimens of the sunscreen products, used for the monochromator, were also tested by this method. Three measurements were made upon each thin film specimen. The first was the ordinary or

* Applied Physics Corp., Monrovia, Calif.

direct spectrophotometric transmission measurement. Figure 4 indicates the arrangement of the various components for this measurement. Only light passing straight through the thin film reaches the photocell and is evaluated as transmitted light. This light will be defined as direct transmitted light. Light scattered by the film as shown in Fig. 5 cannot be measured by this technique; moreover, measurements on substances that scatter light are likely to indicate erroneously that significantly less light is passing through the specimen.

The second measurement utilized the integrating spheres in conjunction with the spectrophotometer. An integrating sphere is essentially a hollow metal sphere, coated internally with barium sulfate, placed in the light beam of both the sample and reference compartment of the Cary spectrophotometer. The sample slide is placed directly in front of the integrating sphere. The light is reflected as it enters the sphere from a mirror to a standard reference surface in the sphere. The standard reference surface was prepared with magnesium oxide, having a reflectivity of essentially unity. Now the light is diffusely reflected about the interior of the sphere by its barium sulfate coated interior until it is reflected through the exit to the photocell. Differences between spheres and the greater amplification of light required are electronically compensated. The forward scattered light from the thin film is collected by the integrating sphere and evaluated by the photocell as the total transmission of the film. The arrangement of the components for these measurements is shown in Fig. 6.

By replacing the reference surface of the sample compartment sphere with the sample, the reflection or backward scattered light can be measured. Figure 7 illustrates the arrangement for these reflection measurements. All the light, no matter how it is reflected from the surface of the sample, is part of the flux in the sphere and is measured by the photocell.

Thus the optical properties of the thin film can be measured or calculated. Comparison of the first and second types of measurements (direct transmission and total transmission) indicated if the film behaves as just a chemical sunscreen (i.e., obeys the Lambert-Beer Law) or if it has both the chemical and physical sunscreen attributes. The forward scattering (FS) measurement indicates the percent total transmitted light. The backward scattering or percent reflection (BS) measurement determines the total light reflected by the specimen. The percent absorption (Abs.) of the sunscreen product can then be calculated: $\text{Abs.} = 100 - \text{BS} - \text{FS}$.

Measurements on Thin Film Specimens

Figure 3 illustrates the difference between total transmission and the monochromator direct transmission measurements on two physical sunscreen products and a chemical physical sunscreen combination product. Figure 2 compares the results for the total transmission, direct transmission, and monochromator direct transmission for a product with only a chemical absorber. All three measurements readily agree in this instance. Hence only the total transmission of the thin film need be

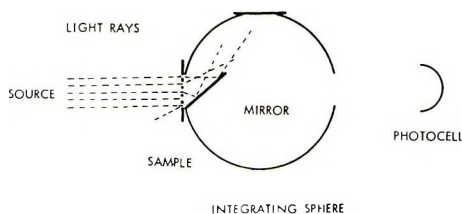


Figure 6. Diffuse scattering with integrating spheres

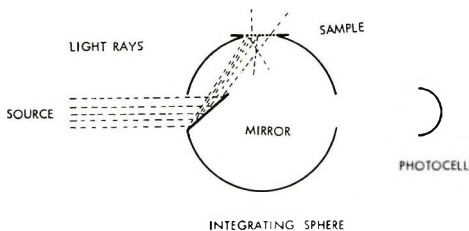


Figure 7. Diffuse reflectance with integrating spheres

determined, regardless of the type of protection offered by the film, since the major protective value of each sunscreen is determined by its total transmission in the sunburn region of the spectrum. Little variation was obtained in total transmission in a thin film after drying. Obviously, if the film separates mechanically, physically, or chemically upon drying, its optical characteristics will change.

In general, the total transmission values agree with the direct transmission values from the monochromator for each sample. Usually the monochromator values show less transmission than those from the integrating spheres total transmission values. This difference was anticipated since the monochromator with the photocell did not collect all available light from each sample. The most comparable values obtained were those in the region where the chemical absorber absorbed

most efficiently, and the greatest divergence from the values occurred where the physical particles exerted the greatest effect in the total absorption of light (i.e., near 360–700 $m\mu$ and 230–270 $m\mu$). The comparable runs without chemical absorbers and also without the physical sunscreen show even greater divergency between measurements, as shown in Fig. 3. Again this shows the loss of light due to scattering by physical particles rather than to normal absorption. The photocell-monochromator combination just could not detect as much of the scattered light as the integrating spheres.

Some difference in the shape of curves obtained by the different techniques is to be anticipated from the optical and mechanical inferiority of the monochromator as compared to the Cary spectrophotometer. Two important reasons are: (a) the intensity of the light source of the monochromator is unstable; it is not simultaneously in balance with a reference beam; (b) the monochromator optics are much poorer (wider slits, greater band width, etc.) since it was not designed for narrow-band spectrophotometric work.

Using films of different thicknesses, 0.0254, 0.0019, 0.0127, and 0.00635 mm \pm 10%, with physical and chemical/physical sunscreen products, it was found that for transmission measurements the physical sunscreens do not obey the Lambert-Beer Law.

The reflective characteristics of the thin films were also examined to determine the degree to which reflection contributes to the protective ability of sunscreen products. In Fig. 8 the reflection spectra of films with physical and chemical/physical sunscreen products may be seen. Figure 2 shows the reflection spectrum of a film with a chemical sunscreen. The most striking feature of the reflection spectrum is that it appears to follow the total transmission spectrum. If the sample transmits well it also reflects well. If it absorbs well in some spectral region, or transmits little light, it does not reflect well. For example, the opaque cream base reflects uniformly at all wavelengths slightly more light than the *p*-aminobenzoic acid (clear viscous) solution. However, addition of 10% talc to the opaque cream base doubles its reflective capacity in the 300 $m\mu$ to 700 $m\mu$ region. Addition of 5% amyl *p*-dimethylaminobenzoate to the talc product did not increase the reflection very much. On the contrary, because of the absorbing capacity of the amyl-*p*-dimethylaminobenzoate in the sunburn range, the reflection of the talc product was reduced to the level of the opaque cream base in the 230 $m\mu$ to 350 $m\mu$ range; from 350 $m\mu$ to 700 $m\mu$ the reflection was equal to or slightly higher than that from the talc product.

Comparison of monochromator or integrating spheres measurements of thin films with the measurements of the solution-dilution method reveal interesting and important observations: The transmission characteristics are qualitatively similar for the chemical sunscreen product as determined by both methods (Figs. 1 and 2); however, these are entirely different for the physical and the chemical/physical sunscreen products, particularly in the longer wavelengths (Figs. 2 and 3). The physical sunscreen, being insoluble in solvents used in the solution-dilution method, obviously cannot affect the transmission spectrum as it does in the

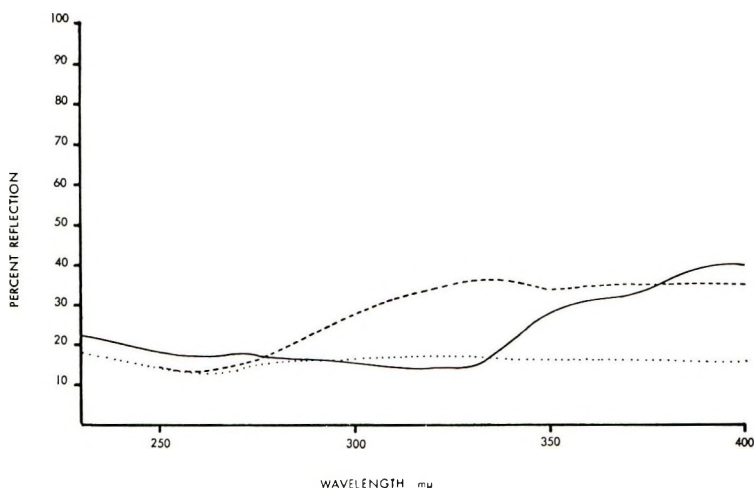


Figure 8. Percent reflection using spectrophotometer with integrating spheres of 0.0254 mm thick films of three sunscreen products. — 5% amy1-*p*-dimethylaminobenzoate + 10% talc in opaque cream base; - - - 10% talc in opaque cream base; and . . . opaque cream base

film specimens. This also is responsible for the lack of reflection in the solution-dilution method samples, although reflection is observed so well in the thin film specimens. Naturally, the lack of physical sunscreens in the extracted solution makes it impossible to determine the differences in transmission when one physical sunscreen is added to another.

In conclusion, measurements with the thin film technique are more representative of conditions actually occurring on the skin since the product, when applied on the skin, will form a film, and the thickness of this film can be reproduced by this technique. In addition, the product is optically measured quickly and conveniently in its original form without being laboriously altered to the solution form, as in the solution-dilution method.

IN VITRO STUDIES WITH HUMAN EPIDERMIS

Specimens of Caucasian human skin were surgically obtained at autopsy, and the intact epidermis was separated from the underlying tissues by the stretch technique (9). Histological examination of the epidermal skin revealed good, white, entire epidermis, with a total thickness of $40\ \mu$, comprised of stratum corneum ($16\ \mu$ thick), and stratum malpighian ($24\ \mu$ thick). The epidermal section was then mounted on a quartz slide for total transmission and reflection measurements. The same measurements were repeated after the physical/chemical sunscreen was applied to the epidermis. The total transmission of the epidermal

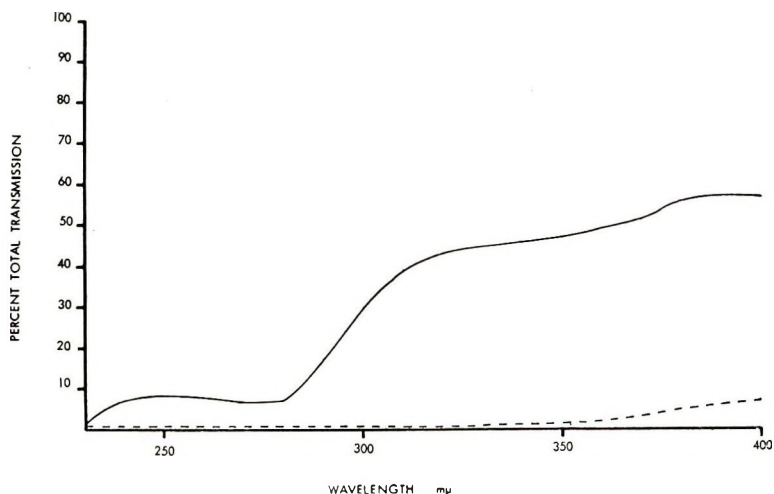


Figure 9. Total transmission of human epidermis (*in vitro*) using spectrophotometer with integrating spheres. — human epidermis and - - - human epidermis with sunscreen product, 5% amyl-*p*-dimethylaminobenzoate + 10% talc in an opaque cream base, applied

specimen was similar to that found by Everett *et al.* (10) using a similar technique of measurement. The application of the chemical/physical sunscreen to the epidermis considerably reduced the total transmission of both ultraviolet and visible light to less than 3% of the light penetrating the epidermis originally (Fig. 9). In the regions where the skin normally transmitted the greatest amount of light (320–700 $m\mu$) the physical sunscreen component (talc) of the combination sunscreen product lowered the total transmission markedly. In the sunburn region (280–320 $m\mu$) the total transmission was also markedly reduced because of the chemical sunscreen component.

A considerable quantity of the light in the sunburn and solar carcinogenic portion of the spectrum penetrates through normal epidermal layers to the dermis. The application of the sunscreen markedly reduces this penetration. The reduction in transmittance corresponds somewhat roughly to the observed erythema protection obtained with this chemical physical sunscreen product.

The initial reflection spectra obtained from these epidermal skin specimens (Fig. 10) were characteristic of those observed for other specimens by Everett *et al.* (10). The addition of a chemical physical

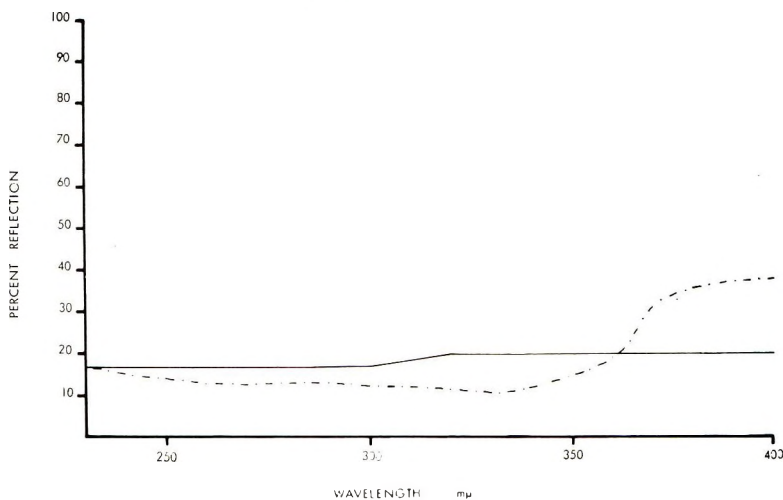


Figure 10. Percent reflection of human epidermis (*in vitro*) using spectrophotometer with integrating spheres. — human epidermis and - - - human epidermis with sunscreen product, 5% amyl-*p*-dimethylaminobenzoate + 10% talc in an opaque cream base, applied

sunscreen product altered the reflection spectra in the sunburn region only a little. In the visible (370–700 $m\mu$), the addition of the sunscreen markedly increased reflection to one and one-half times normal reflectance. In the region where the chemical sunscreen of the product as well as the skin absorbed the strongest (280–350 $m\mu$) the skin and sunscreen combination reflected less light than was reflected by the skin initially. Throughout the shorter portion of the spectrum (230–280 $m\mu$) the skin alone and skin with sunscreen reflected the same.

IN VIVO REFLECTION STUDIES WITH HUMAN SKIN

Based on the *in vitro* studies, *in vivo* studies were attempted, using the dorsal surface skin of subject's hand in place of the epidermis specimen. Three *in vivo* studies indicate very little difference between the

reflection of the skin *in vivo* with or without the protection of a sunscreen. This appears to be true in the visible as well as in the ultraviolet range. The slight differences between the reflectance spectra could be as easily attributed to differences in pressure or positioning of the subject's hand over the opening to the integrating sphere. The fact that in one experiment the skin with the sunscreen actually reflected less light than the skin without sunscreen, while in the other two experiments the reverse of this occurred, would tend to support the contention that positioning, pressure, and trauma from applying the sunscreen may be more influential in changing the reflection spectra than the actual physical additive. Further studies are in progress to obtain information as to how changes in the circulation and the trauma of application of the sunscreen affect the reflection of the human skin and how to modify the equipment to eliminate these drawbacks.

SUMMARY

Development of sun-protective preparations must continue to provide products giving a broad range of protection against the undesirable cutaneous responses to sunlight, such as sunburn, photosensitivity reactions, accelerated aging, and carcinogenesis. Most of the products available provide protection against sunburn and promote increased pigmentation. One of the drawbacks is a lack of a suitable laboratory method that will yield realistic information about the protective ability of the sunscreen product without the need for prolonged and strong exposure tests in humans to sunlight and other light sources with similar spectra. Use of the thin-film technique presented in this paper with a xenon arc monochromator has afforded such information. Modifying this method by using integrating spheres with a Cary spectrophotometer Model 14, in place of the monochromator, has made it possible to measure the total transmission or forward scattering and reflection or back scattering in addition to the direct transmission usually measured. The thin film technique makes it possible to determine rapidly these measurements on the sunscreen films simulating those applied on the skin without altering in any way the sunscreen product, as is commonly done with the classic solution-dilution method. The new method has, in addition, made it possible to evaluate not only the chemical sunscreens but also the physical sunscreens and chemical/physical sunscreen combinations. This has not been possible with previously published standard laboratory methods. The versatility of the thin film technique

makes it possible to measure the protective ability of sunscreen products on excised human epidermis as well as conduct similar measurements on human skin *in situ*.

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Hygroscopicity and Hardness of Hair Spray Resins at Varying Humidities

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Presented May 10, 1966, New York City

Synopsis—An apparatus is described for measuring moisture pick-up, drying rate, and Sward Hardness of various resin films at relative humidities of from 50 to 90%. Data are given on films laid down from anhydrous SDA #40 ethyl alcohol. Resins used include: Polyvinyl Pyrrolidone (PVP K-30), Vinylpyrrolidone/Vinyl Acetate Copolymer (PVP/VA 735), VEM[®] Resins, 90, 50, and 0% neutralized with 2-amino-2-methyl-1,3-propanediol (AMPD), and National Starch Resyn[®] 28-1310, 90, 70, and 0% neutralized with AMPD. One set of data used the unplasticized films, and a second set used the film plasticized with 3.5% DC-555 Silicone Oil. Although this apparatus has been useful for collecting data on resins used in hair sprays, the method and apparatus can be used for obtaining data with these and other resins for applications such as adhesives and coatings.

INTRODUCTION

There has been a noticeable lack of data comparing moisture pick-up and hardness of hair spray resin films at various humidities. What little data are available have been on work that was done only with one type of resin by the manufacturer using a method not fully described. One Technical Service Bulletin (1) shows the water absorption rate of Resyn[®]† 28-1310, neutralized 125, 90, and 0% with 2-amino-2-methyl-1,3-propanediol (AMPD), for periods of up to 160 hours. A patent (2) assigned to National Starch gives the Sward Hardness of Resyn 28-1310

* G. Barr Co., Niles, Ill. 60648.

† Resyn is a registered trademark of National Starch and Chemical Corp., Resin Division, New York, N. Y.

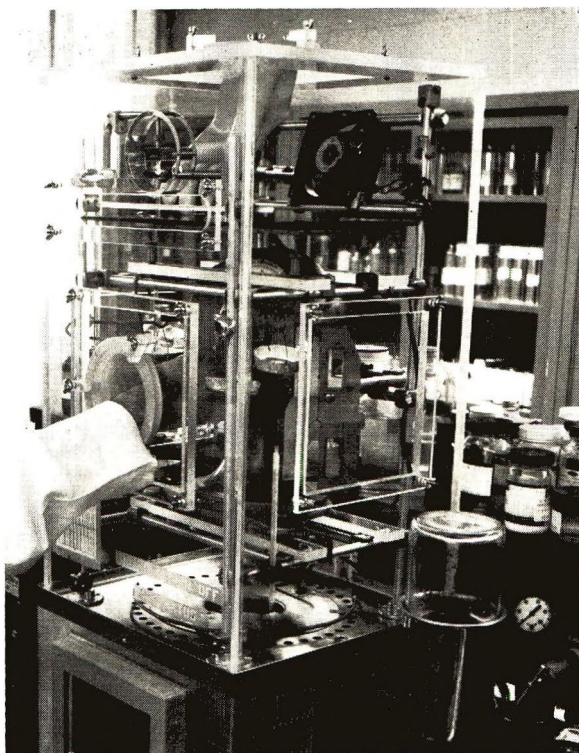


Figure 1. Controlled humidity chamber

neutralized to varying degrees with several different amino-alcohols. Sward Hardness values range from 44 down to 14, starting with un-neutralized resin and going up to 150% of the theoretical equimolar amount of copolymer reacted. Another Technical Service Bulletin (3) describes the hardness of Resyn 28-1310 at varying degrees of neutralization with four different amino-alcohols. This bulletin also shows the moisture content at equilibrium with 50 to 90% relative humidity of Resyn 28-1310 films neutralized to 100, 80, and 60% with AMPID. These films were cast from solutions in anhydrous SDA-40 ethyl alcohol. The hygroscopicity of PVP K-30* (4) and PVP/VA (5) has been reported at relative humidities ranging from 30 to 90%.

About the only work comparing various resins using the same method was done in Japan (6). VEM[®]† -640, -645, and -649, PVP K-30,

* PVP K-30 and PVP/VA, Antara Chemicals, Division of General Aniline and Film Corp., New York, N. Y.

† VEM is a registered trademark of G. Barr Co., Division of Pittsburgh Railways Co., Niles, Ill.

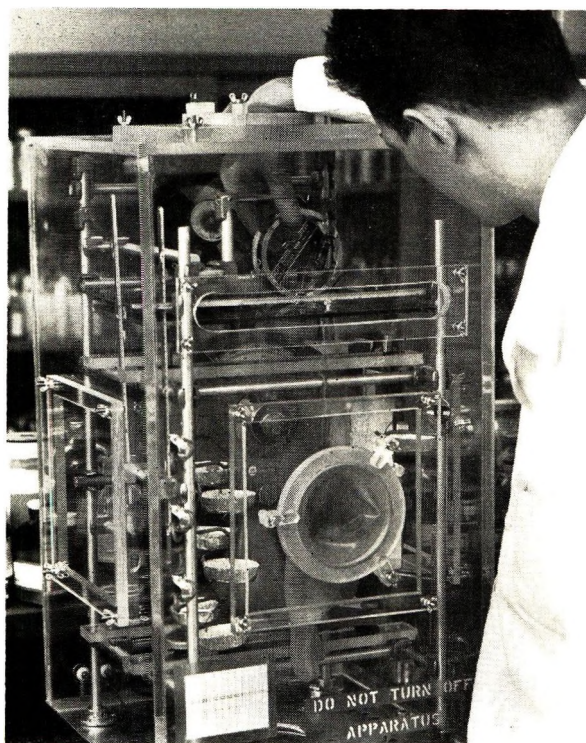


Figure 2. Chamber with Sward Hardness Rocker

PVP/VA, Nasuna A/B, shellac, and PVM/MA were tested for moisture pick-up at 70 and 90% R.H. Data for 50/50 mixtures of the above resins were also obtained. Unfortunately, this work does not include Resyn 28-1310.

EXPERIMENTAL

Apparatus

The Controlled Relative Humidity instrument (Model VP-100A*) was modified by replacing the original glass jar with a rectangular Plexiglass chamber (Fig. 1) which has openings for inserting rubber gloves. One additional opening in the top front side was provided for replacing the polished glass plate for Sward Hardness measurements. Because of the large size of the chamber, two additional fans were included for increasing air circulation. In the lower part of the chamber is installed a Sauter Balance† with a scale range from 0–1000 mg.

* Blue M Co., Blue Island, Ill.

† Sauter Co., Ehingen-Württ., Germany.



Figure 3. Sauter balance and weighing dishes

with 2 mg divisions and 1 mg sensitivity. In order to run hardness measurements simultaneously with moisture pick-up, a Sward Hardness* instrument was inserted into the top part of the chamber (Fig. 2). For cooling, tap water was used, which made it possible to adjust the relative humidity from 50% up to 90%. Lower relative humidities could have been obtained by the use of colder circulating water, but that range was of no interest in these experiments.

A constant temperature of 29.5°C was maintained in the chamber for all tests. It was possible to control the temperature in the chamber within $\pm 0.05^\circ\text{C}$ and the relative humidity within $\pm 0.5\%$.

The usual manner of obtaining relative humidity utilizes dry and wet bulb thermometers. In general, wet bulb readings depend on the air velocities over the wet wick. At lower air velocities, higher wet bulb temperature will result, and hence a higher humidity reading will be made. By the use of a Bendix Psychron Model 566† which is de-

* Gardner Laboratory, Inc., Bethesda, Md.

† The Bendix Corp., Baltimore, Md.

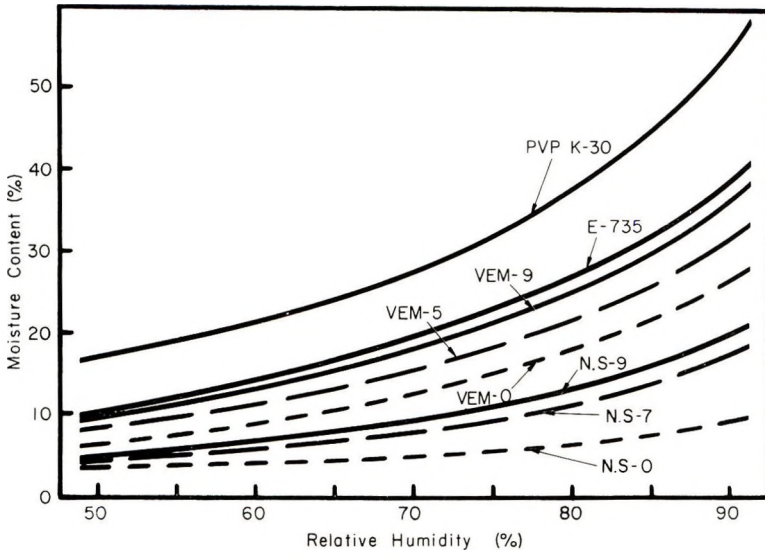


Figure 4. Moisture absorption of various resin films vs. relative humidity (30°C)

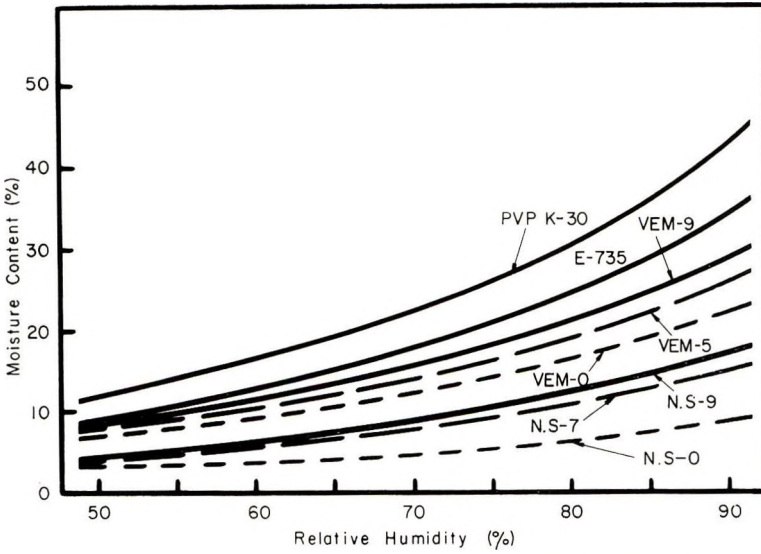


Figure 5. Moisture absorption of various films with 3.5% plasticizer (Silicone oil DC 555) vs. relative humidity (30°C)

signed to meet U. S. Weather Bureau specification and which uses an air velocity of 5 m/sec past the wet and dry bulb, it was found that the air velocity in the chamber was high enough to obtain correct relative humidity readings.

Method

Approximately 1.25 g of a 40% solution of resin in anhydrous ethanol was placed evenly in an aluminum dish (diameter 5.1 cm) provided with a hanger and dried at room temperature over a period of two days. At the end of this time a uniformly dried film formed on the bottom of the dish. Final drying of the film was done at 105 °C for a 2-hour period in a circulating oven. In this way it was possible to obtain a resinous film weighing 0.5 ± 0.02 g in each dish with practically no surface bubbles, which might increase the surface in contact with air and probably would change the rate of conditioning. This arrangement made it possible to place eight dishes on hangers in the chamber for one run. For the determination of moisture pick-up or loss the dishes were placed on the hook of the Sauter Balance (Fig. 3), weighed, and then returned to the hangers.

For Sward Hardness measurement three different films were cast on the polished glass plate from a 40% solution of the resin in anhydrous ethanol having a wet thickness of 3×10^{-3} in. (1.2×10^{-3} cm). These films were then dried at room temperature outside of the chamber for two days. In this way the air in the chamber was not contaminated with alcohol vapor. Then the glass plate with the films was placed in the chamber and conditioned for two days under the humidity conditions desired for determining the hardness. It was found that this time period was sufficient to obtain equilibrium conditions of the films. Duplicate hardness measurements were taken about six hours later to check the results.

RESULTS

Data shown include results obtained with four resins commercially used as hair fixatives: National Starch Resyn 28-1310, G. Barr Co. VEM Resin, Antara PVP/VA E-735, and Antara PVP K-30. The first two resins have carboxylic groups which, for hair spray purposes, usually are neutralized with an amino-alcohol. In the case of Resyn 28-1310, 90% (N.S-9) and 70% (N.S-7) neutralization with AMPD and also unneutralized resin (N.S-0) were used. The VEM resin was 90% neutralized (VEM-9) and 50% neutralized (VEM-5) with AMPD, as

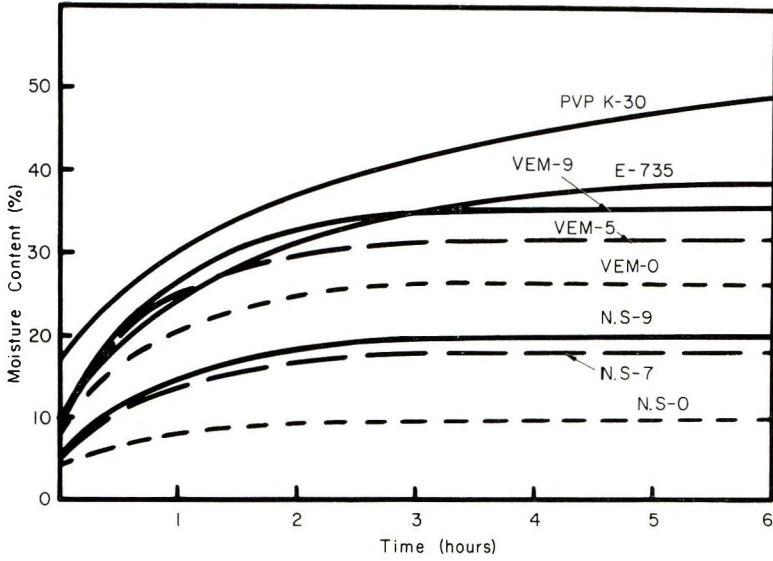


Figure 6. Moisture absorption of various resin films *vs.* time upon conditioning from 50 to 90% relative humidity (30°C)

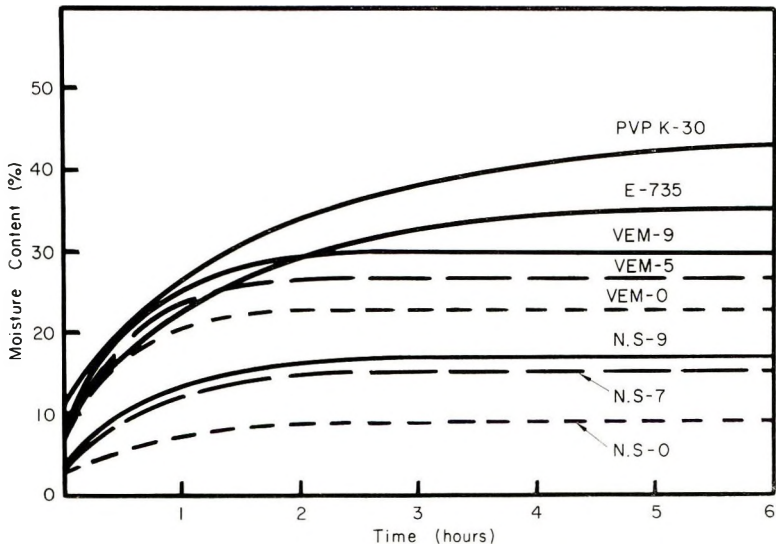


Figure 7. Moisture absorption of various resin films with 3.5% plasticizer (Silicone oil DC 555) *vs.* time upon conditioning from 50 to 90% relative humidity (30°C)

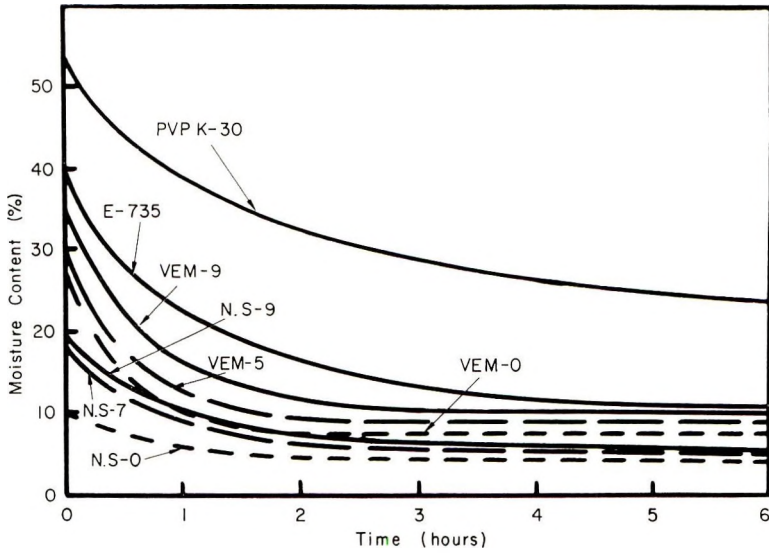


Figure 8. Drying of various resin films vs. time upon conditioning from 90 to 50% relative humidity (30°C)

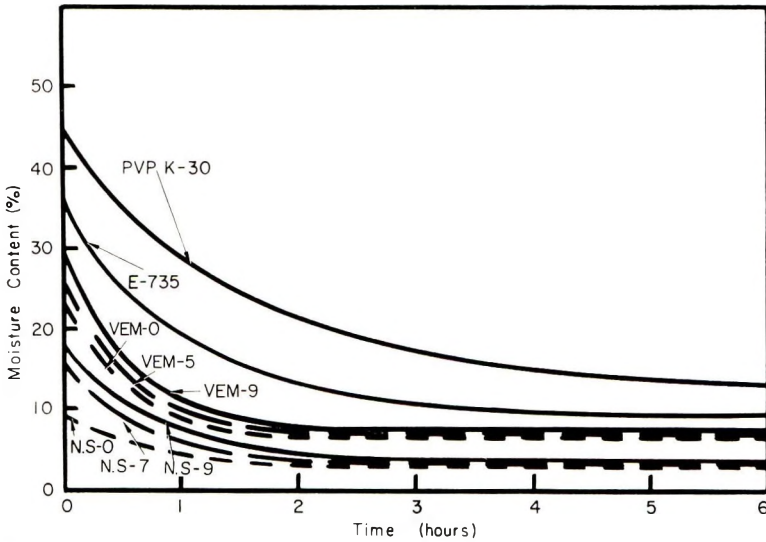


Figure 9. Drying of various resin films with 3.5% plasticizer (Silicone oil DC 555) vs. time upon conditioning from 90 to 50% relative humidity (30°C)

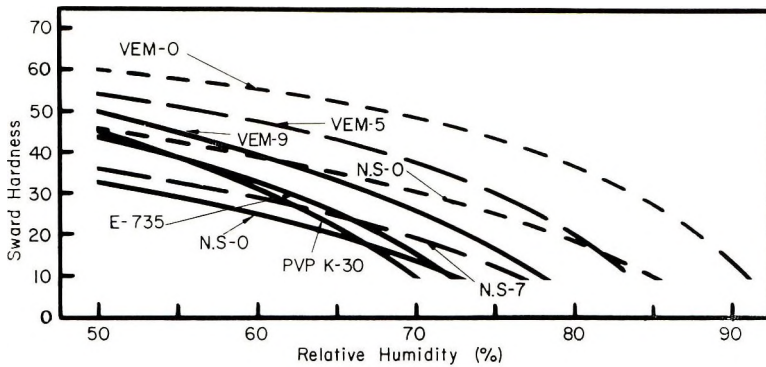


Figure 10. Sward hardness of various resin films vs. relative humidity (30°C)

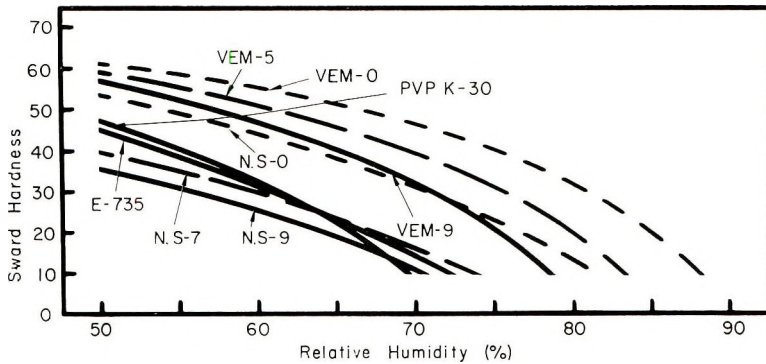


Figure 11. Sward hardness of various resin films with 3.5% plasticizer (Silicone oil DC 555) vs. relative humidity (30°C)

well as unneutralized (VEM-0). The percentage of water absorption is based on dry resin. All tests were made with films with and without plasticizer. Whenever a plasticizer was used, it was 3.5% Silicone Oil DC 555* based on dry resin.

Figure 4 shows the moisture absorption at different relative humidities of various resin films at equilibrium. PVP K-30 shows the greatest moisture absorption, followed by PVP/VA 735. VEM has lower humidity pick-up. As can be anticipated, unneutralized resin has lower moisture absorption than does neutralized resin because of its lower water solubility. Resyn 28-1310 has the lowest moisture absorption. Figure 5 shows the results with plasticized films. There is a decrease in the water absorption of the plasticized films in comparison with the nonplasticized films.

* Dow Corning Corp., Midland, Mich.

Figure 6 shows the moisture absorption rate of these films conditioned at 50% relative humidity and then exposed to 90% RH. Both carboxylic types of resin have a much faster conditioning rate than PVP/VA 735 and finally PVP K-30. Plasticized films have approximately the same conditioning rate (Fig. 7), except that the initial and final absorptions are lower than those of unplasticized films.

The drying rate of films conditioned at 90% RH and then exposed to 50% RH is shown in Fig. 8. Equilibrium is reached by all the films in two to four hours except PVP/VA 735 and PVP K-30. Figure 9 gives the results with films that have been plasticized. The drying rate curves of plasticized films are all displaced downward due to the hydrophobic character of the plasticizer.

Figure 10 shows the variation of Sward Hardness with relative humidities from 50 to 90% at equilibrium. It can be seen that the unneutralized VEM-0 and 50% neutralized VEM-5 give the hardest films. These are followed by 90% neutralized VEM-9, except beyond 61% RH where the hardness is surpassed by the unneutralized Resyn 28-1310. It can be seen that the hardness of both the PVP K-30 and PVP/VA 735 decrease more rapidly. Figure 11 gives similar data using plasticized films. Hardness obtained with plasticized films with silicone oil could, of course, be affected by at least three factors: (a) plasticizer decreases the moisture pick-up which results in harder film; (b) plasticizer itself decreases the hardness; and (c) plasticizer lubricates the surface between the film and the Sward Hardness rocker, thereby increasing the Sward Hardness reading. Each of these factors may produce different effects, even on the same plasticized film at different humidities.

CONCLUSIONS

In conclusion, it can be seen from the foregoing that the greater the degree of neutralization the greater the hygroscopicity of the films of carboxylic types of resins. PVP/VA 735 and PVP K-30 films are more hygroscopic than any of the films made with VEM or the Resyn 28-1310 resins. Although moisture pick-up by the VEM resin is greater than that of Resyn 28-1310, the hardness of the former at any given humidity is greater. Greater hardness at high humidity means less tack. A hard film on the hair will break more easily when the comb is passed through, whereas a soft, tacky film will put drag on the comb. Soft, tacky films will also result in hair snagging with resultant difficulty in combing. A certain degree of moisture pick-up is perhaps desirable

because it makes the films more flexible and prevents flaking since the absorbed water acts like a plasticizer.

It is to be understood from the data presented that several resins are available to give any desired hygroscopicity or hardness characteristics; the data should not be construed to show relative ratings. Soft and tacky films may be desirable for such formulations in which sticking together of hair after combing is of prime importance.

(Received June 8, 1966)

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Evaluation of Certain Factors Influencing Oil Deposition on Skin After Immersion in an Oil Bath

M. E. STOLAR, Ph.D.*

Presented May 10, 1966, New York City

Synopsis—Previously developed methods for determining quantitatively the amount of oil and recording the oil film deposited on the skin after immersion in oil baths were utilized to evaluate various oils of potential use in bath oil formulations. The effect of the presence of various surfactants in mineral oil on the amount and skin coverage of the deposited oil film was determined. The existence of a relationship between spreading coefficient values and the amount or skin coverage of the deposited oil film was investigated.

The use of bath oils in the treatment of dry scaly skin has gained favor in the last few years. Originally, dry skin was explained on the assumption that the surface lubricating lipid film was qualitatively or quantitatively modified or even missing (1). Blank (2) and Borota (3) demonstrated that cornified material obtained from callus remained soft and pliable as long as it retained moisture. To extend the duration of moisture retention, as indicated by softness and pliability, the tissues were coated with oil and placed on a wet surface.

Normal baths are frequently contraindicated due to their degreasing and drying effects on the skin (4). Pillsbury *et al.* (5) claimed that immersion of the skin in water followed by topical application of grease demonstrated beneficial effects. These may be attributed to hydration of the stratum corneum due to a decrease in the rate of moisture loss

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Table I

Effect of Grade of Mineral Oil (0.02%) Dispersed in Water (40°C) on Oil Deposition on Skin after 15 Minutes of Immersion

Grade of Oil	Composition in % ^a		Specific Gravity at 60°F	Saybolt Viscosity at 100°F	Amount of Oil Added to Bath (g)	Amount of Oil Eluted from Skin (g)	% of Oil Deposited on Skin
	Naphthenic	Paraffinic					
Mineral oil USP ^a	40.0	60.0	0.885	350	0.200	0.044 ^b	22.2
Mineral oil NF ^a	37.0	63.0	0.852	83	0.200	0.062 ^b	31.0
Mineral oil technical ^a	30.0	70.0	0.832	55	0.200	0.080 ^b	40.0

^a Supplied by Sonnenborn Chemical and Refining Company, New York, N. Y.

^b Average of six determinations.

Table II

Spreading Coefficient Values for Various Grades of Mineral Oil

Grade of Oil ^a	Specific Gravity at 60°F	Surface Tension at 25°C (dynes/cm)	Interfacial Tension at 25°C	Spreading Coefficient at 25°C
Mineral oil USP	0.885	32.2	44.6	-4.8
Mineral oil NF	0.852	33.4	41.9	-3.3
Mineral oil technical	0.832	31.4	41.5	0

^a Supplied by Sonnenborn Chemical and Refining Company, New York, N. Y.

from the skin without affecting the rate of moisture diffusion from the underlying tissues (1). Clinical studies attesting to the beneficial effects of bath oils have been reported in medical literature (6-9).

Commercially available bath oils may be classified as either spreading or dispersible types. The spreading type tends to form a surface film of oil on the water, while the dispersible type forms a dispersion of fine droplets of oil throughout the water. Both types are claimed to deposit an oil film on the immersed skin.

Knox (10) described the development of a water dispersible oil bath which is deposited on skin surfaces. Taylor (11) reported that about twice as much oil is deposited from aqueous dispersions of mineral oil as from vegetable oil. Similar conclusions were reached by Knox (12). Taylor's results are based on the difference between the initial amount of oil present in an immersion cylinder and the residual amount after immersion for a specified period of time. This difference is indicative of the amount of oil deposited on the treated skin surface.

Stolar (13) recently developed a method for determining the actual amount of oil deposited on the skin after immersion in an oil bath and



Figure 1a. Mineral oil U.S.P.



Figure 1b. Mineral oil N.F.



Figure 1c. Mineral oil technical

Figures 1a—1c. Effect of grade of mineral oil (0.02%) dispersed in water (40°C) on skin coverage of the deposited oil films

visually defined the area covered by the deposited film. This method was used to compare the affinity to skin of four commercial bath oils and to record the area covered by the deposited oil film.

This report is concerned with the evaluation of various oils of potential use in bath oil formulation for their affinity to skin and the determination of the coverage or continuity of the deposited films. Furthermore, it investigates the effect of some nonionic surfactants on the amount and continuity of the oil film deposited from aqueous dispersions of mineral oil.

Table III
Effect of Type of Oil (0.02%) in Aqueous Dispersion (40°C) on Oil Deposition on Skin after
15 Minutes Immersion (Average of Six Determinations)

Type of Oil	Amount of Oil Added to Bath (g)	Amount of Oil Eluted from Skin (g)	% Oil Deposited
Isopropyl esters of fatty acids			
Isopropyl myristate	0.200	0.078	39.0
Isopropyl laurate	0.200	0.079	39.5
Isopropyl palmitate	0.200	0.066	33.2
Isopropyl linoleate	0.200	0.074	37.2
Sebacic acid esters of alcohols			
Di-isopropyl sebacate	0.200	0.062	30.7
Di-butyl sebacate	0.200	0.072	36.2
Di-octyl sebacate	0.200	0.071	35.7
Triglyceride esters ^a			
Triglyceride of short chain fatty acids	0.200	0.073	36.5
Triglyceride of modified coconut oil	0.200	0.0612	30.6
Polyol esters ^a			
Polyol esters of fatty acids	0.200	0.011	5.7
Polyol diester of short chain fatty acids	0.200	0.087	40.0
Alcohol			
Hexadecyl alcohol	0.200	0.080	40.0
Lanolin derivatives			
A ^b	0.200	0.079	39.5
B ^b	0.200	0.095	47.5
C ^c	0.200	0.025	12.5
Vegetable oils			
Almond oil	0.200	0.018	9.0
Cottonseed oil	0.200	0.057	28.5
Corn oil	0.200	0.063	31.5

^a Supplied by Drew Chemical Corporation, Boonton, N. J.

^b Supplied by American Cholesterol Products, Inc., Edison, N. J.

^c Supplied by Malmstrom Chemical Corporation, Newark, N. J.

Table IV
Spreading Coefficient Values for Various Oils

Type of Oil	Surface Tension of Oil (dynes/cm)	Interfacial Tension at 25°C	Spreading Coefficient
Isopropyl esters of fatty acids			
Isopropyl myristate	31.3	16.7	20.3
Isopropyl laurate	29.8	23.8	19.5
Isopropyl palmitate	27.8	25.9	17.1
Isopropyl linoleate	31.6	19.8	18.7
Sebaic acid esters of alcohol			
Di-isopropyl sebacate	31.1	12.9	29.7
Di-butyl sebacate	33.0	22.1	19.5
Di-octyl sebacate	33.4	15.1	26.1
Triglyceride esters ^a			
Triglyceride of short chain fatty acids	32.3	12.7	28.9
Triglyceride of modified coconut oil	32.0	20.6	19.8
Polyol esters ^d			
Polyol esters of fatty acids	33.5	31.1	7.8
Polyol diesters of short chain fatty acids	31.0	21.2	22.4
Alcohols			
Hexadecyl alcohol	28.8	24.1	19.1
Lanolin derivatives			
A ^b	32.0	16.5	23.0
B ^b	31.8	11.2	36.0
C ^c	31.3	11.5	31.2
Vegetable oils			
Almond oil	31.0	45.8	-5.1
Cottonseed oil	34.9	48.0	-8.1
Corn oil	31.9	51.6	-10.6

^a Supplied by Drew Chemical Corporation, Boonton, N. J.

^b Supplied by American Cholesterol Products, Inc., Edison, N. J.

^c Supplied by Malmstrom Chemical Corporation, Newark, N. J.

EXPERIMENTAL

The experimental procedure and methods for determining the amount and continuity of the deposited oil film were previously described (13). Briefly, the procedure consisted of dispersing, by stirring for five minutes with a Lightnin mixer, 0.200 g of oil containing 4000 USP units of Vitamin A palmitate in one liter of tap water. The oil bath was maintained

Table V

Effect of Concentration of Various Surface Active Agents on Deposition of Mineral Oil on Skin After 15 Minutes Immersion (Average of Six Determinations)

Type and Concentration (%) of Surfactant in Mineral Oil (Technical)	Amount of Oil Added to Bath (g)	Amount of Oil Eluted from Skin (g)	% Oil Deposited
Polyethylene Glycol 400 Dilaurate			
0	0.200	0.080	40.0
4	0.200	0.082	40.0
6	0.200	0.056	28.0
8	0.200	0.039	19.7
Polyethylene Glycol 400 Dioleate			
0	0.200	0.081	40.0
4	0.200	0.080	40.0
6	0.200	0.065	32.5
8	0.200	0.040	20.0
Polyoxyethylene (5) Tallow Amine			
0	0.200	0.080	40.0
4	0.200	0.046	23.2
8	0.200	0.033	16.7
Polyoxyethylene (5) Soybean Amine			
0	0.200	0.080	40.0
4	0.200	0.081	40.5
8	0.200	0.035	17.8

at 40°C in a water bath. The subject immersed his hand so that the water level reached his wrist, exposing the same surface area at each immersion. At the end of 15 minutes, the hand was removed and allowed to air dry. The back of the hand was exposed to a source of ultraviolet irradiation in a dark room and the fluorescence photographed using Ektachrome-X film.

The deposited oil film was then eluted with mineral oil, filtered through a #1 Whatman filter paper, and the volume adjusted with mineral oil to 100 ml. The eluate was then assayed spectrophotometrically at 325 m μ .



Figure 2a. Isopropyl myristate



Figure 2b. Isopropyl palmitate



Figure 2c. Isopropyl linoleate



Figure 2d. Di-isopropyl sebacate



Figure 2e. Di-butyl sebacate



Figure 2f. Di-octyl sebacate



Figure 2g. Triglyceride of short chain fatty acids



Figure 2h. Triglyceride of modified coconut oil

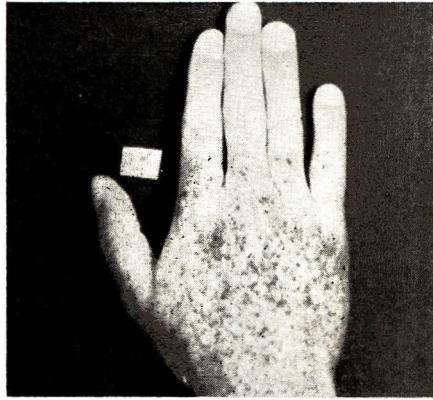


Figure 2i. Polyol esters of fatty acids

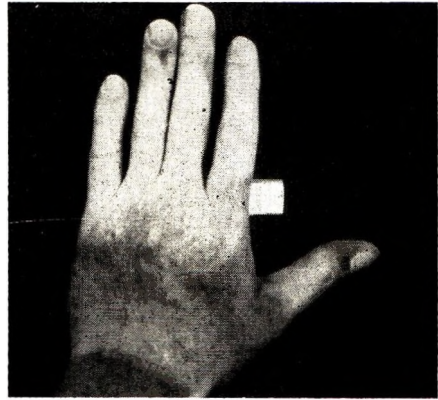


Figure 2j. Polyol di-esters of short chain fatty acids



Figure 2k. Hexadecyl alcohol



Figure 2l. Lanolin derivative "A"



Figure 2m. Lanolin derivative "B"

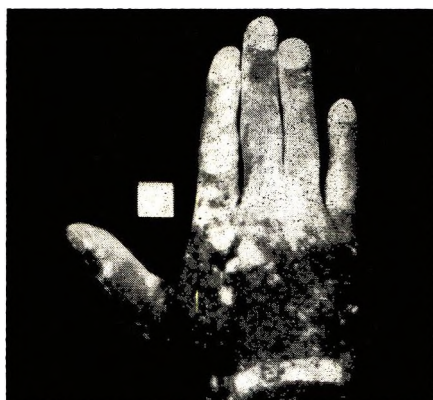


Figure 2n. Lanolin derivative "C"



Figure 2o. Cottonseed oil

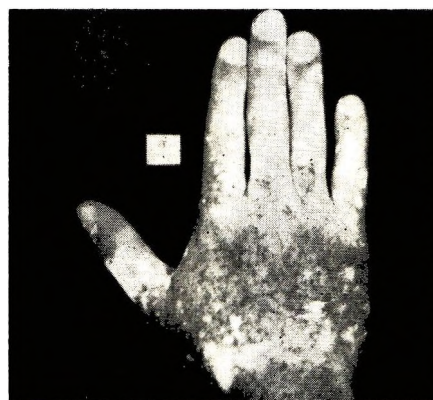


Figure 2p. Corn oil

Figures 2a-2p. Effect of various oily liquids in aqueous dispersions, maintained at 40°C, on skin coverage of deposited oil films

The spreading coefficient has been used as an indication of spreadability of one immiscible liquid over another (14). Spreading coefficient values were calculated from the relation:

$$\text{Spreading coefficient} = \text{ST (water)} - \text{ST (oil)} - \text{IFT (water-oil)}$$

The surface tension (ST) and interfacial tensions (IFT) were obtained with the DuNuoy tensiometer. The procedure consisted of measuring the surface tension of the aqueous phase which was then layered gently with the oil phase; the interfacial tension was then determined. The surface tension of the oil was determined separately and the spreading coefficient calculated from these values after corrections were made for density and ring size.

RESULTS AND DISCUSSION

The most extensively used ingredient in topical and cosmetic formulations is mineral oil. The affinity of mineral oil to hair from a shampoo formulation was demonstrated by Goldemberg (15). Taylor (11) reported that bath oils formulated with mineral oil were superior to bath oils prepared with vegetable oil. Neither researchers stated the type of grade of the mineral oil used in their investigation.

Mineral oils in given viscosity ranges may appear to be quite similar, yet can be significantly different in composition, physical properties, and behavior on the skin (16). These oils contain paraffinic (20–47%) and naphthenic (53–83%) fractions, and significant variations in relative percentages of these two fractions of hydrocarbons may exist in oils having similar viscosities.

Table I demonstrates the effect of the grade or composition of mineral oil in aqueous dispersion, maintained at 40°C, on oil deposition on the skin after immersion for 15 minutes. From these results it would appear that the amount of oil deposited is affected by the grade of mineral oil. With increasing viscosities there appears to be a decrease in the amount of oil deposited. Furthermore, the viscosities appear to be directly related to the naphthenic content. An increase in the naphthenic content of the mineral oil results in an increase in viscosity and an apparent decrease in affinity to skin. This is exhibited in Figs. 1a–1c.

No correlation appears to exist between spreading coefficient values obtained for the various grades of mineral oil and the amount or coverage of the deposited oil film (Table II).

Table III illustrates the effect of various oily liquids dispersed in water maintained at 40°C on oil deposition on skin after immersion for 15 minutes. Figures 2a–2p record the coverage of skin achieved by the deposited oil films from aqueous dispersions of the various oily liquids.

A direct correlation may be noted between the spreading coefficient values obtained for the isopropyl ester series and the amount of oil deposited on the skin (Table IV). With increasing spreading coefficient an increase may be noted in the amount and coverage of the skin by the deposited oil film.

In the sebacic acid esters series, significantly different spreading coefficient values were recorded for di-butyl and di-octyl esters. However, the amount of oil deposited appears to be the same. Figures 2e and 2f exhibit better coverage of skin from aqueous dispersions of di-butyl sebacate than from di-octyl sebacate. The best skin coverage was re-

corded from aqueous dispersions of di-isopropyl sebacate (Fig. 2d). However, the amount of di-isopropyl sebacate deposited on skin appears to be the least with the highest spreading coefficient value obtained in this series.

A relationship between spreading coefficient and the amount of oil deposited on the skin was recorded for triglyceride and the polyol

Table VI
Effect of Concentration of Various Surfactants on Spreading Coefficient of Mineral Oil (Technical)

Type and Concentration (%) of Surfactant in Mineral Oil	Surface Tension of Oil (dynes/cm) at 25°C	Interfacial Tension at 25°C	Spreading Coefficient
Polyethylene Glycol 400 Dilaurate			
0	31.4	41.5	0
4	31.4	0.8	42.9
8	30.9	1.2	42.8
Polyethylene Glycol 400 Dioleate			
0	31.4	41.5	0
4	30.1	1.8	42.0
8	28.8	1.0	42.3
Polyoxyethylene (5) Tallow Amine			
0	31.4	41.5	0
4	30.9	1.0	42.9
8	31.8	0.8	42.4
Polyoxyethylene (5) Soybean Amine			
0	31.4	41.5	0
4	31.2	1.3	42.5
8	30.9	1.0	42.8

esters of fatty acids. In both series, with increasing spreading coefficient values there appears to be an increase in the amount and coverage of the oil film deposited after immersion in an oil bath.

Hexadecyl alcohol demonstrates good deposition on skin after immersion in aqueous dispersion and a substantial spreading coefficient value. However, the coverage of the deposited film on the skin appears to be inadequate when compared to the film deposited by esters of similar spreading coefficient values.

The affinity to skin of three lanolin derivatives in aqueous dispersions is recorded in Table III. A relationship appears to exist between the spreading coefficient values obtained for two lanolin derivatives (A and B) and the amount of oil deposited on the skin. However, lanolin

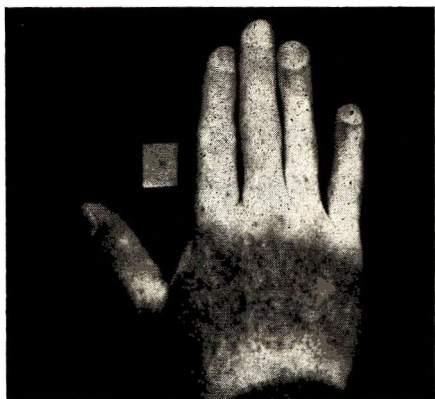


Figure 3a. 4% Polyethylene glycol 400 dilaurate in mineral oil



Figure 3b. 8% Polyethylene glycol 400 dilaurate in mineral oil



Figure 3c. 4% Polyethylene glycol 400 dioleate in mineral oil



Figure 3d. 8% Polyethylene glycol 400 dioleate in mineral oil



Figure 3e. 4% Polyoxyethylene (5) tallow amine in mineral oil



Figure 3f. 8% Polyoxyethylene (5) tallow amine in mineral oil



Figure 3g. 4% Polyoxyethylene (5) soybean amine in mineral oil



Figure 3h. 8% Polyoxyethylene (5) soybean amine in mineral oil

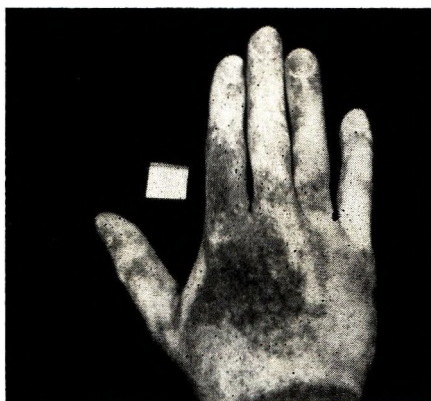


Figure 3i. Mineral oil (technical)

Figures 3a-3i. Effect of presence and concentration of various surfactants in mineral oil (technical) dispersed in water, maintained at 40°C, on skin coverage of deposited oil films

derivative A, with the lower spreading coefficient value, appears to achieve superior coverage of the skin than lanolin derivative B. A significantly higher spreading coefficient value was obtained for lanolin derivative C, which exhibited poor deposition and coverage of the skin (Table IV).

The deposition of oil on the skin from aqueous dispersions of various vegetable oils appears to be unsatisfactory, due to their spotty nature (Figs. 2o and 2p). Significant differences in the amount of oil deposited on the skin were recorded in order of decreasing affinity: corn oil > cottonseed oil > almond oil.

Table V illustrates the effect of the presence of various surfactants at different levels in mineral oil (technical grade) on oil deposition on the skin after immersion in aqueous dispersions. From the results, it would appear that increasing the concentration of surfactants above about 4% results in a decrease in the amount of oil deposited. The amount of oil deposited appears to be unaffected as long as the concentration of the surfactant remains below a certain level. This level appears to be between 4-6% for polyethylene glycol 400 dilaurate, polyethylene glycol 400 dioleate and polyoxyethylene (5) soybean amine. The critical concentration for polyoxyethylene (5) tallow amine appears to be between 0-4%.

The addition of 4% surfactant to mineral oil (technical grade) results in a dramatic increase in spreading coefficient values. Additional increases in surfactant concentration to mineral oil does not appear to affect significantly the spreading coefficient values (Table VI). Similar results were observed by Paruta *et al.* (17).

Figures 3a to 3i illustrate the effect of the presence of various surfactants at different levels in mineral oil (technical grade) on the skin coverage of the deposited oil film. These photographs demonstrate that the area covered by the deposited oil film remains the same as that of the original oil, regardless of the concentration of surfactant. However, with increasing concentrations of surfactants, the amount of oil deposited is reduced, as may be seen by the decrease in the intensity of fluorescence.

From the data presented, it appears that a relationship may exist between spreading coefficient values and the amount of oil deposited on the skin after immersion in an oil bath. No relationship could be observed between spreading coefficient values and the character of coverage achieved by the deposited oil film. The surface area covered by the deposited oil film appears to be characteristic for the specific oil, and addition of various concentrations of surfactants does not appear to affect the area covered but rather the amount of oil deposited.

SUMMARY

The affinity to skin of various oils of potential use in bath oil formulations was investigated.

The effect of the presence of various surfactants in mineral oil (technical grade) on oil deposition after immersion in an oil bath was determined.

A possible relationship between spreading coefficient values and the amount or skin coverage of the deposited oil film was investigated.

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Book Reviews

DYNAMICS OF CHROMATOGRAPHY, PART I: PRINCIPLES AND THEORY, by J. Calvin Giddings. Marcel Dekker, Inc., New York. 323 pages, illustrated and indexed. Price: \$11.50.

This is the first of three parts entitled, "Dynamics of Chromatography," by Dr. Giddings. It is almost entirely devoted to relating chromatographic processes to mathematics. New concepts, for example, the random walk treatment, are covered extensively for the first time. Essentially, the author is laying the theoretical basis for the more practical Parts 2 and 3.

Zone migration is called the cornerstone of chromatography. The first chapter brings the reader inside the chromatographic column where each molecule is in a stop-and-go game of leap frog, being alternately affixed and released from the stationary phase. Next, the importance of controlling migration rate differences is emphasized. A historical review of zone spreading concepts is presented with emphasis on the random walk. Molecular diffusion, sorption-desorption and flow phenomenon are de-

scribed with the aid of schematic illustrations for those of us who are less mathematically capable. Major emphasis is on the mathematical interpretation, however.

Chapters 3 and 4 deal with the nonequilibrium in chromatographic columns. Early paragraphs in each chapter adequately explain the phenomena, and lengthy interpretations follow.

Next comes the relationship between packing structure and flow dynamics. "Packing structure is so complicated that it has defied all efforts to come to grips with it mathematically." Dr. Giddings pretty much accepts his above dictum, and a very readable chapter follows. Flow pattern is discussed in terms of its relationship to porosity, interstitial channels, and the bending and coiling of a column. The flow processes of paper and thin-layer chromatography are compared. They depend on capillary action.

Finally, the author concludes his first volume with "achievement of separation." As this is the ultimate goal, he wishes for a set of rules to cover the diversity of chromatography. These rules are not yet in

sight, so the chromatographer must combine principles, theory, and intuition. Recognition of the latter came as a surprise to this reader in view of the mathematical nature of preceding chapters of the book. Optimum parameters for resolution are noted. He shows how factors such as sample size, dead volume, column length, flow velocity, and temperature affect resolution. How these can be adjusted for fast separation is next emphasized.

Part I will be of interest only to the most mathematically inclined practitioners of chromatography. It ends on a note of practical application, however. Perhaps Parts 2 and 3, concerned with gas chromatography and liquid chromatography, will be useful to the chemist in the laboratory.—JAMES W. JENKINS—Ph.D. Leeming/Pacquin Division, Chas. Pfizer & Co., Inc.

LEXICON OF DETERGENTS, COSMETICS AND TOILETRIES, by G. Carrière, Elsevier Publishing Company, New York, 1966. 203 pages. Price \$10.

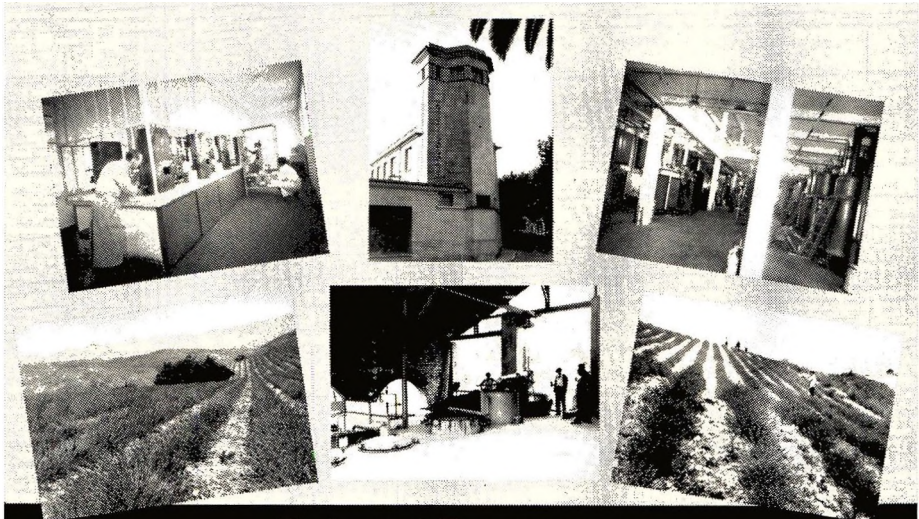
This booklet is one of the Elsevier series of Lexica in which specialized terms in many languages are assembled. This lexicon comprises two

parts: Part one is concerned primarily with detergents and includes translations of 257 terms into 19 languages; the second portion deals with cosmetics and toiletries and includes 266 terms in 8 languages. With the aid of a table of contents it is quite easy to find terms on soil, water, lipsticks, or nail preparations in a variety of languages.

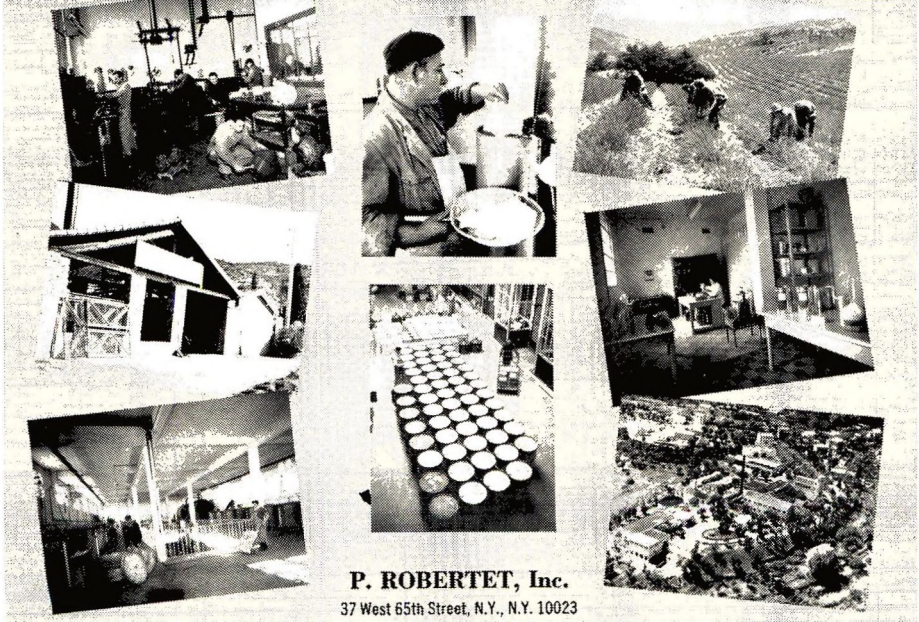
This volume has been edited by a well-known authority in the detergents and toiletries fields. In addition, the contributors to individual languages deserve credit for making a book of this type available. Despite the obviously careful editing, certain minor discrepancies have crept into the text. Typical of these is item #139 under detergents in which the "acid layer of the skin" is translated as "lo strato acido della pelle" into Italian, whereas the "acid mantle" is translated as "il tonico" in item #328 of the cosmetic section.

Minor defects of this type do not detract from the utility of this book. The primary value of a dictionary of this type is translation of technological jargon, something which an ordinary dictionary cannot cover.

Over all, this book should be of interest to those who deal with detergents and cosmetics on an international scope.—M. M. RIEGER, Warner-Lambert Research Institute.



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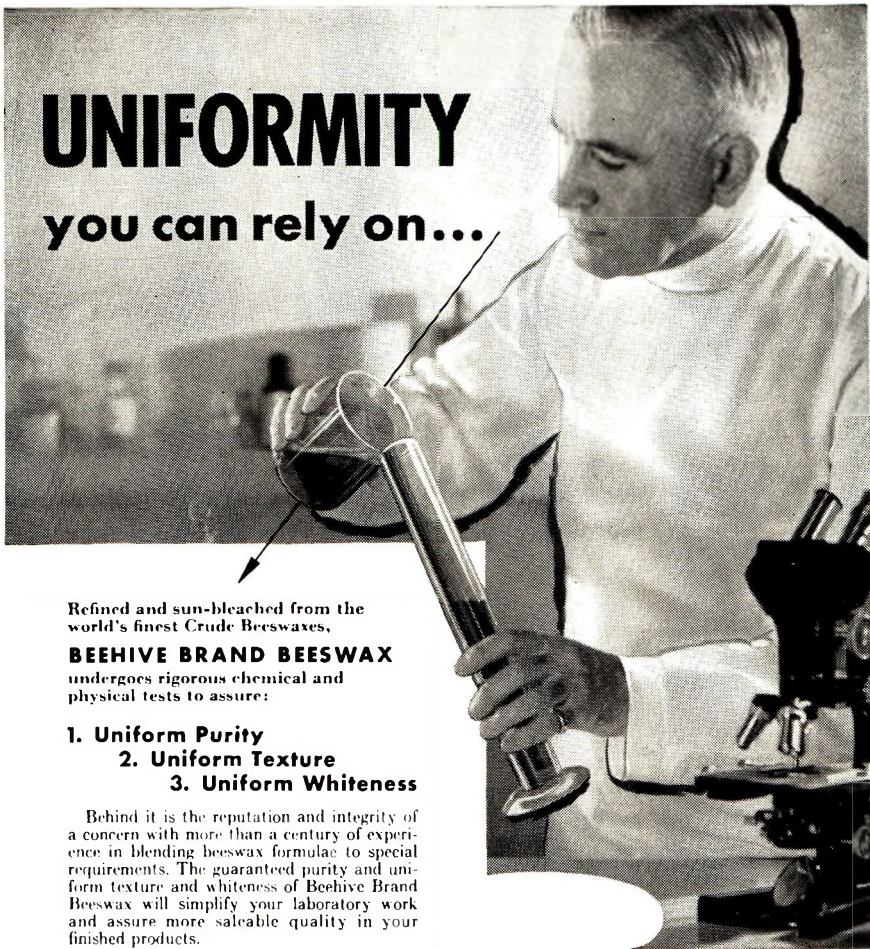
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