VOL XVII No 13

Journal of the Society of Cosmetic Chemists

Contents

	Page
SOCIETY NEWS	
I.F.F. Award 1965	747
New members	748
ORIGINAL PAPERS	
A new and realistic electronic approach to the evaluation of anti- perspirant activity <i>R. J. James</i>	749
Caucasian hair, Negro hair, and wool: similarities and differences J. Menkart, L. J. Wolfram, and Irene Mao	769
The action of antiperspirants Christopher M. Papa	789
Molecular complex formation in aerosol emulsions and foams Paul A. Sanders	801
DEPARTMENTS	
Synopses for card indexes	xxxv
Literature survey	xxxix
Book reviews	831
Index to Volume XVII	835
Index to advertisers	xxxiii

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Editor:	Dr. Martin M. Rieger, 170 Tabor Road, Morris Plains, N. J. 07950
Associate Editor:	Gabriel Barnett, 145 Paterson Ave, Little Falls, N. J. 07424
Business Manager:	George King, 505 Hamilton Road, Merion Station, Pa. 19066
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Literature Survey:	Joseph H. Kratochvil, 100 Jefferson Road, Parsippany, N. J. 07054
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INDEX TO ADVERTISERS

American Cholesterol Products, Inc	xxv
Biologic Consultants	xxvii
Colgate-Palmolive Co	xxvii
Cosmetic Laboratories, Inc	xxvii
Croda, Inc	viii
Dodge & Olcott, Inc	v
Enjay Chemical Co	xxiv
Evans Chemetics, Inc.	i
Firmenich, Inc	x-xi
Fleuroma	xix
Florasynth Laboratories, Inc	xii
Fluid Chemical Co., Inc	xvi
Fritzsche Brothers, Inc.	xvii
Givaudan CorpInside Front	Cover
Goldschmidt Chemical Corp	xxviii
Halby Products Co., Inc	iii
Hoffmann-La Roche, Inc	xv
International Flavors and Fragrances, Inc	xxvi
Lanaetex Products, Inc	xxix
Leberco Laboratories	xxvii
Malmstrom Chemical Corp	xxii
Miranol Chemical Co	xxxi
Parento, Compagnie, Inc	xxi
Pennsalt Chemicals Corp	vi-vii
Pennsylvania Refining Co	xxxiv
Reheis Chemical Co	iv
R.I.T.A. Chemical Corp	xxxii
Robeco Chemicals, Inc	XX
Robertet, P., IncInside Back	Cover
Robinson Wagner Co., Inc	xiii
Roure-DuPont, Inc	xxiii
Vanderbilt, R. T., Co., Inc.	XXX
Van Dyk and Co., Inc	xviii
Verley, Albert and Co	ix
Washine Chemical Corp	xiv
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The following synopses can be cut out and mounted on 3×5 in. index cards for reference, without mutilating the pages of the Journal.

A new and realistic electronic approach to the evaluation of antiperspirant activity: R. J. James. Journal of the Society of Cosmetic Chemists 17, 749 (1966)

Synopsis—A new method for the evaluation of antiperspirant activity eliminates many of the problems inherent in other proposed methods. The use of electronic equipment gives fast results. The use of phosphorus pentoxide cells minimizes the problem of temperature control. Simultaneous measurements are recorded on an X-Y recorder instead of the strip chart recorder usually used. The method described illustrates the measurement of the dynamic changes in rates of perspiration.

Caucasian hair, Negro hair, and wool: similarities and differences: J. Menkart, L. J. Wolfram, and Irene Mao. Journal of the Society of Cosmetic Chemists 17, 769 (1966)

Synopsis—Caucasian hair, Negro hair, and Lincoln wool are compared in terms of physical structure, mechanical properties, chemical composition, and rate of reaction with various reagents. Several significant differences are found between wool and the two hair samples. The principal one is the extent of crosslinking, which is greater in the hair, as indicated by higher sulfur and cystine contents, and a higher proportion of the material found in the γ -keratose fraction after oxidation. The heavier crosslinking is reflected in greater resistance to attack by hot acid and in slower reduction by thioglycolate or sulfite solutions. The main difference between Caucasian and Negro hair is the fiber geometry; the Caucasian fiber approximates a cylinder, the Negro a twisted oval rod. In their physical and chemical properties, the two fibers are very similar.

The action of antiperspirants: Christopher M. Papa. Journal of the Society of Cosmetic Chemists 17, 789 (1966)

Synopsis—Stripping experiments, methylene blue iontophoresis, and histological studies after induced sweating indicate that formaldehyde forms a physical plug in the sweat duct. On the other hand, similar experiments with aluminum chloride treated skin suggest that this antiperspirant acts by altering the permeability of the sweat duct.

Molecular complex formation in aerosol emulsions and foams: Paul A. Sanders. Journal of the Society of Cosmetic Chemists 17, 801 (1966)

Synopsis—Previous studies in nonaerosol systems have shown that certain detergents, such as sodium lauryl sulfate, form molecular complexes with long chain alcohols or acids at air/water or oil/water interfaces. In the present investigation it has been shown that molecular complexes are also formed in aerosol emulsion systems. It was found that, in many cases, the addition of a long-chain alcohol to an aerosol emulsion system prepared with the tricthanolamine salt of a fatty acid or sodium lauryl sulfate as the surfactant produced a marked increase in emulsion and foam stability and a decrease in foam drainage. In some cases foam viscosity was increased. These effects of the long-chain alcohols occur in nonaerosol and aerosol systems and are indicative of molecular complex formation.

LITERATURE SURVEY*

Analytical

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^{*} Prepared by Joseph H. Kratochvil and Joseph L. Rosenstreich.

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lii

I.F.F. Award 1965

The I.F.F. Award for the year 1965 was split between the authors of two papers in an unusual ceremony, during the Seminar luncheon on September 20th in New York.

The I.F.F. Award is made possible through the generosity of International Flavors & Fragrances, Inc., and is administered by the Editorial Committee of the JOURNAL OF THE SOCIETY OF COSMETIC CHEMISTS. This award is a means of recognizing the author(s) of the outstanding paper published in an American issue of the JOURNAL during the past calendar year.

This year the award was shared by Robert L. Goldemberg for his paper, "Use of Anti-irritants in Cosmetic Formulating," and by Messrs. Steven Carson, Myron S. Weinberg, and Richard Goldhamer for their paper, "An Improved Method for Testing the Safety of Hair Dye Preparations." These papers appeared in the JOURNAL OF THE SOCIETY OF COSMETIC CHEMISTS during the year 1965. The I.F.F. Award, which includes an honorarium of \$1000, will be made in future years to stimulate original and scientific contributions to the JOURNAL.

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- 510 N. Dearborn St., Chicago, Ill. 60610 Becker, Dr. Samuel W., Jr., 8631 S. Jeffery, Chicago, Ill. 60617
- Berube, Gene R., 2 Merr West Boylston, Mass. 01505 2 Merrimount St.,
- Bisceglie, Frank P., 529 Thurnau Dr., River Vale, N. J. 07675 Buffa, Charles W., 94 Hamilton Ave., Fairview, N. J. 07022
- Chhotalal, Miss Metha S., Max Factor & Co., 1655 N. McCadden Pl., Hollywood, Calif. 90028
- Deleamont, Pierre T., 25 Shore Dr., Plan-dome, N. Y. 11030
- Fontana, Jean Marie, 333 E. 79th St., Apt. 6N, New York, N. Y 10029
- Goldstein, Bernard R., 7147 Niles Ave., Niles, Ill. 60648
- Hanak, Joseph S., 91 Morehouse, Bridge-port, Conn. 06605
- Hays, Kenneth W., Fiber Products Div., The Kendall Co., West St., Walpole, Mass. 02081
- Jeffries, Charles T., 431-B S. 19th Ave., Maywood, Ill. 60153
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- Kingman, Lauren C. Jr., The Mearl Corp., 217 N. Highland Ave., Ossining, N. Y. 10562
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- Kligman, Dr. Albert M., Hospital of the Univ. of Pa., 34th & Spruce Sts., Philadelphia, Pa. 19104
- Knox, Dr. John M., Dept. of Dermatology. Baylor Univ. College of Medicine, 1200 Moursund Ave., Houston, Tex. 77025
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- Livingstone, Alexander, General Aniline & Film Corp., 457 Lincoln St., Easton, Pa. 18042
- Magid, Dr. Louis, 19 Clifton, N. J. 07013 199 Haddonfield Rd.,
- Massey, Rollin F., 31 E. Walnut St., Hinsdale, Ill. 60521 McKenzie, Walter L., 41 Woodland Rd.,
- Northboro, Mass. 01532
- Micchelli, Albert L., 21 Bayberry Lane, Middletown, N. J. 07748
- Miley, Gene A., Standard Pharmical, Inc., 1300 Abbott Dr., Elgin, Ill. 60120 Panzer, George W., Alcolac Chemical Corp.
- 3440 Fairfield Rd., Baltimore, Md. 21226
- Paolucci, Peter M., Shell Chemical Co., 923 Park Sq. Bldg., Boston, Mass. 02116
- Phelan, Matthew A., 2420 Garrett Rd., Drexel Hill, Pa. 19026
- Quasius, Alan R., The Toni Co., 456 Merchandise Mart, Chicago, 111. 60654
- Randa, Charles P., Avon Products, Inc., 6901 Golf Rd., Morton Grove, Ill. 60053
- Reilly, Raymond K., Stalfort, Inc., 2012 Hammonds Ferry Rd., Baltimore, Md. 21227
- Reynolds, Ralph, Pylam Products Co., Inc., 95-10 218th St., Queens Village, N. Y. 11429
- Rich, Gene B., Del Laboratories, 565 Broadhollow Rd., Farmingdale, N. Y. 11727
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- Siegel, Dr. Jerome J., Alberto-Culver Co., 2525 W. Armitage Ave., Melrose Park, Ill. 60164
- Snyder, J. MacLean, American Medical Assoc., 535 N. Dearborn St., Chicago, Ill. 60610
- Soyster, Stuart, 24 Benjamin Rd., Tenafly, N. J. 07670 Thabit, Richard J., Mona Industries, Inc.,
- 65 East 23rd St., Paterson, N. J. 07514 Van Buskirk, Walter L., 28 Nelson Rd.,
- Melrose, Mass. 02176
- Vierling, Dr. Richard A., P. O. Box 44, Tonawanda, N. Y. 14150 Wolkoff, Schering Corp., 86 Orange St.,
- Bloomfield, N. J. 07003
- , Bennett, Avon Products, Inc., & Division Sts., Suffern, N. Y. Woods, Bennett, Avon Fair 10901
- Woods, Dr. John C., ICI-Organics-Inc., Main St., Dighton, Mass. 02715

A New and Realistic Electronic Approach to the Evaluation of Antiperspirant Activity

R. J. JAMES, B.S.*

Presented May 10, 1966, New York City

Synopsis—A new method for the evaluation of antiperspirant activity eliminates many of the problems inherent in other proposed methods. The use of electronic equipment gives fast results. The use of phosphorus pentoxide cells minimizes the problem of temperature control. Simultaneous measurements are recorded on an X-V recorder instead of the strip chart recorder usually used. The method described illustrates the measurement of the dynamic changes in rates of perspiration.

INTRODUCTION

Many systems have been devised to measure perspiration in order to evaluate antiperspirant materials. Generally, they can be divided into three classifications. The simplest of these detect perspiration by visual observation of color effects with dyes, starch iodide paper, or specially prepared plastic films (1-4). The next are gravimetric in nature and require the collection of sufficient perspiration to be weighed (5-7). These may either absorb perspiration in pre-weighed pads and absorption cups or freeze it from a gas stream into convenient traps. Finally, there are those methods which sense and record perspiration electronically (8–14). Only these are suitable for continuously recording the changes in perspiration rates during a test period. The method to be described is of the last type.

^{*} Shulton, Inc., Clifton, N. J. 07015.

The manner in which data are handled, either as they are obtained by the equipment or later treated by the investigator, is very important. To date, all of the methods devised have erred in one fundamental detail. Those methods which rely on the gravimetric determination of perspiration automatically carry the error into the data. Where electronic recording has been used, the error has been unavoidably, though unknowingly, included in the results. The error is essentially the averag-



Figure 1. Idealized plot of the perspiration output of area A vs. area B. Without product (line 1) and with product applied to area A only (line 2)

ing of test results at various perspiration rates to produce a *single point* ratio for a series of determinations made on a control area as compared to those made on a test area. This situation has occurred because too much time would be required for a single product evaluation if the other methods were to produce a sufficient number of points to assure the *constant nature* of the ratio for different rates of perspiration. The method described in this paper resolves the problem by continually recording the ratio itself. Before describing the procedure, the principles of obtaining perspiration ratios must be reviewed.

MATHEMATICAL CONSIDERATIONS

When one wishes to compare the effectiveness of antiperspirant materials, it is obvious that tests must be performed directly on human subjects. However, people perspire for many reasons other than too high a temperature or humidity. Many other factors, such as emotion, temperament, and other psychological stimuli, state of health, variation of diet, etc., also influence the degree of perspiration (15). Control of all these variables simultaneously and from test to test is totally impossible. For this reason a single x to x' comparison, in which the output of per-



Figure 2. Plots of perspiration output of area A vs. area B for different types of control tests

spiration, x, from one area, A, of the body is compared to x' from the same area of the body after a product has been applied, cannot be made with accuracy. There is no way of knowing how much of an influence these variables may have had on the change in the value of x to x' as opposed to the influence of the test product coincidentally applied.

In order to obtain a value for x' which represents an antiperspirant effect free of these variables, it has become standard practice to measure the output from another area, B, simultaneously with that from area A, as originally proposed by Fredell and Read (5). The ratio of perspiration between the two areas may be defined as x/y, where x is the value

obtained from area A and y is the value from area B. This test is done once with no product applied to either area. The ratio itself becomes the control.

In a second test an antiperspirant material is applied to area A, leaving area B untreated. If the material is effective, a lower perspiration rate will be obtained from area A, to which a new value x' is as-



Figure 3. Practical plot of the output of perspiration from area A & B under control and test conditions. The dotted lines represent the corrected test lines for a valid comparison

signed. The output from area B may increase or decrease, but rarely will it be the same as that of the first test, because of the influence of the uncontrollable variables. The value y' is assigned to it.

The result of the second test is a new ratio of perspiration from the two areas for the condition where a product is applied to one side only. It is expressed as x'/y'. However, x' includes both the effects of the product and those of the uncontrollable factors which caused y to change to y'. Thus the following inequality results:

$$\frac{x'}{y'} \neq \frac{x}{y}$$

A new value of x from area A, which does not include the effects of the product when y' is the value for area B, is now required. It may be cal-

culated as x'' from the previously determined values x, y, and y' in the following expression:

$$\frac{x''}{y'} = \frac{x}{y} \text{ or } x'' = \frac{xy'}{y}$$
(1)

If the uncontrollable factors cause y' to be less than y in the control area, then x'' will be less than x. If the converse is the result, then x'' will be larger than x (see Fig. 1).

If the antiperspirant applied has been effective, the calculated value x'' from area A, representing the condition of *no product applied*, will always be larger than x', the value determined from area A representing the condition of *a product applied*. The following calculation will give the *per cent depression* of perspiration caused by the product:

$$\frac{x'' - x'}{x''} \cdot 100 = \text{ per cent depression}$$
(2)

This is the mathematics used in previous work to obtain the desired information from antiperspirant test data. However, these calculations were based on the tacit assumption that the ratio of the rates of perspiration from two areas on a human body remained constant at *all levels of perspiration*. Previous work has taken for granted that this assumption is valid; however, no direct proof has yet been offered. It is known that human subjects perspire in erratic ways and from many causes other than exercise, heat, and humidity (15). Thus, the constant nature of this ratio becomes of prime importance. If proven to be false, then all previous work becomes quite inconclusive.

In part this paper will *prove* the *validity* of the original assumption and that previous work, based on the calculations just discussed, has contained unexpected errors which have compromised the results. A graphic representation of these calculations compared to the graphic treatment of data by the proposed procedure will clarify these errors and demonstrate a means for separating them from the desired information.

The graph in Fig. 1 presents the relationship of the perspiration values obtained from area A as plotted against those from area B. Point a is the specific ratio, x/y, obtained for the values x and y derived from the simultaneous test of areas A and B respectively. Because the perspiration rates from the right and left axilla of a given individual are rarely equal, these values will seldom be the same. Line 1 drawn through point a to the origin of the graph represents, as assumed, a set of equal ratios for the values obtained from areas A and B for various rates of perspiration in the condition where no product is applied to either area.



Figure 4. Diagram of equipment organization

Point b, the ratio x'/y', represents the new ratio obtained from a second test in the condition where a *product is applied* to area A only. Similarly, line 2 through point b to the origin of the graph is a new set of equal ratios, but in the second condition. The value x'' is obtained by extending a horizontal line from point y' through b to the intersection c on line 1 and dropping a perpendicular from c to the x-axis. Now two perspiration rates, x' with product and x'' without product, have been established for area A while the value for area B is unchanged, i.e., equal to y'. These two values of x may now be used to calculate the per cent depression of perspiration as described previously (eq. 2).

This treatment of data, whether graphic or calculated, is valid only if the ratios in each set are constant at different perspiration rates, i.e., the lines on the graph representing them pass through the origin. Assume that point b had fallen instead on a line such as 3, not passing through the origin. With this *single point* there is no way of knowing which way the ratio line is directed. As a result, the value calculated for x'' would be incorrect, because it would be based on one of the many ratios found on line 3, and the ratios along line 3 vary.

In their effort to save time, or to produce a practical number of tests economically, previous methods have erred by relying on a single point

754



Figure 5. Gas supply and regulating system

determination of the ratios concerned. In a sense they have been forced to do this by the time required to collect sufficient perspiration for an accurate weight to be made or sufficient data to produce the single point by averaging. The constancy of the ratios cannot be determined by this type of data treatment. The ratio line or constancy of the ratio can be determined conveniently only by the continuous recording of the ratio itself rather than its components.

The procedure described in this paper continuously records the ratio changes which result directly in a ratio line rather than a single point. A consequence of this precision is the discovery of several forms of error or deviations from the ideal ratio line. The analysis of these deviations and the understanding of their operation has led to the determination and application of the necessary corrections to obtain valid results.

Four types of tests are represented by the lines in Fig. 2. Line 4, *passing through the origin*, is the ideal test along which the ratios of the



Figure 6. Absorption cup. The outer concentric space uses a vacuum to hold the cup to the subject. Moisture is picked up in the center section

values from area A to those of area B are equal. This test, however, is quite rare. Line 5, not passing through the origin, represents the more common form of test. It may be displaced to one side or the other of the ideal line. The ratios along this line are not equal. Line 6, another form of test, has the same properties as line 5 but a starting point also above the origin. Again the ratios are not constant along this line. Line 7 is quite rare and represents the only specific case of an individual with different perspiration ratios at different rates. It can also be displaced from the origin.

The important observation to be made here is that the perspiration ratios of most subjects change along a linear function as illustrated by lines 5 and 6. This fact has been repeatedly demonstrated by the procedure to be described. The fact that the ratios along these lines are not constant is due to an additive factor, the source of which will be explained later. If this additive factor is eliminated, the lines will pass through the origin and become ideal lines. Consequently, the original assumption, that two areas on an individual would retain a constant ratio at different perspiration rates, has been proven correct. The problem has remained with the elimination of experimental errors.

756

The device used to refine these displacements or errors from the desired information is based on the realization that the *slopes* of the ratio lines obtained from the tests are the factors to be compared. The slope of the line is independent of its placement on the graph.

Figure 3 illustrates how this comparison may be accomplished graphically. One draws new lines respectively parallel to the test lines and



Figure 7. Absorption cup in place

passing through the origin. The change in ratio from point a on the parallel to the control ratio line to that of point b on the parallel to the test ratio line may immediately be read. Perspiration rate x'' has been reduced to x' by the application of a product to site A. Values x'' and x' taken from Fig. 3 may now be used to calculate the absolute reduction of perspiration, as in equation 2.

The causes of these additive shifts in position of perspiration ratio lines are not easily defined. Tentative data suggest that variations in

JOURNAL OF THE SOCIETY OF COSMETIC CHEMISTS

758

the initial wetness of the skin during the test may be the cause. It is surprising how long it takes to dry wet skin even with a very dry gas stream. Capillary creepage of liquid moisture from the wet side to the dry side under the edge of the separation in the cup may be another cause. If this is so, then any method utilizing absorption cups will be subject to this error. Applying grease to the cup can possibly eliminate



Figure 8 Heating chamber

this creepage, but great care must be used not to smear the test area with it or the results will be compromised.

The additive error becomes considerably more serious as the test period is reduced or as the rate of perspiration utilized for the test is decreased. However, if the test period is lengthened, one loses the ability to follow the relative change in product effectiveness with time. If heavy perspiration rates are used, a washing away of the active material renders the test inaccurate. Since the procedure to be described can separate the error from the desired data, a short test period does not impair the results. Consequently, this procedure can both follow a product effect with time and measure, if any, a wash-out effect as well.



Figure 9. Temperature controller

EXPERIMENTAL PROCEDURE

The block diagram in Fig. 4 illustrates the test equipment assembled for this procedure. All gas connections are of 3.2 mm Teflon tubing to minimize the permeation of moisture through the walls of these connections.

The gas supply system is shown in Fig. 5. Dry nitrogen from the cylinder flows through a sensitive regulator to reduce the pressure to approximately 1000-1500 mm. The wet and dry gas values may be balanced against each other to adjust the delivery of moisture to the main gas stream and to calibrate the operation of the system. The manometer need not be accurate. It is used only to insure that the pressure from test to test remains the same or as a cue to a leaking cup. The mixing chamber assures the thorough distribution of added moisture before the gas stream is diverted to the rest of the system.

Clear plastic is used in fabricating the absorption cups (Fig. 6). The importance of relocating them precisely in the identical area for a test



Figure 10. Moisture monitor assembly

series requires sufficient visibility for locating placement marks exactly. Note the concentric sections. Dry air in the center one sweeps away the moisture for measurement while a vacuum in the outer section holds the cup in place. A vacuum of more than 130 mm should never be used, as this may injure the subject. Preferably it should remain between 50-75 mm.

The cups are placed into the shaved axillae of the subject after he has been rolled into the heating chamber (Fig. 7) and is comfortable on his back. The gas stream is turned on at about 1200 cc per minute, and the whole system is allowed to dry down. The subject should not be perspiring at this point.

The heating chamber (Fig. 8) consists essentially of a large inverted box on legs into which a subject, lying on his back, may be rolled. His holding the trapeze-like accessory both positions and limits the motion of his arms which may disturb the cups. With the side doors closed the temperature is raised by a circulating hot air blower and controlled by an electronic temperature sensing device. A clear plastic plate (6 mm) has been used throughout its construction to forestall claustrophobic tendencies of the subjects by allowing them to see out on all sides. The temperature controller (Fig. 9) senses the temperature in the air duct rather than in the chamber and is set for a temperature higher than that actually applied to the subject. However, it is calibrated to produce the desired exposure temperatures, which are read on an auxiliary thermometer. The rapid cycling of the heater that results from this arrangement maintains extremely stable exposure temperatures

The level of temperature to which each subject is exposed depends



Figure 11. Phosphorous pentoxide cell

upon his particular nature and to outside relative humidity. Each individual has a different and critical temperature-humidity ratio above which active perspiration ensues. The relative humidity tends to remain fairly stable during tests; thus the critical temperature of the subject is of greater concern and is determined at the start of a test for each. During a test the subject is heated to his critical temperature or slightly below. His perspiration is then brought about by having him work his legs as if riding a bicycle. To decrease the perspiration he is allowed to



Figure 12. x-y recorder

rest. In this way, the variation in perspiration will cause the *x*-*y* recorder to trace a sloped pattern on the graph.

It has been found that when a subject lies still in the warm atmosphere he may be lulled to sleep by the hypnotic drone of the equipment. When asleep, his perspiration can become quite erratic. An actual alternating pulse of one axilla followed by the other has been observed.

The moisture monitors (Fig. 10) are placed side by side so that their meters may be viewed simultaneously. The two flow meters on the top right of each instrument measure the total flow of gas and the quantity that is sampled for moisture content. The system is calibrated to read in μ l/l (parts per million) of moisture when the sample flow is set for 10 ml/min. Since the measurements required are all relative, the exactness of this calibration is unimportant. However, the gas flows should not change during a test.

Several scales may be selected by the attenuator switch on the left front of each instrument. Its position will depend on the relative output of perspiration of the subject under test.

762

The large black knob on the right front of each instrument encloses the moisture sensing cell (Fig. 11). It consists of a capillary U-tube containing two separate spiral electrodes. The whole assembly is en-



Figure 13. Results of a series of tests as outlined in Table 1: 13a is the control; 13b is Test 1; 13c is Test 2; and 13d is Test 3 (note changing slopes)

capsulated in epoxy resin. The electrodes are activated with phosphoric acid which, when subjected to an electric current, is converted to the nonconducting phosphorus pentoxide by the electrolytic destruction of the water present. As a result, the conductivity of the cell is reduced essentially to zero. When the cell senses moisture, its conductivity will rise accordingly. The meter reads this conductivity and will balance when the input of moisture equals its destruction.



Figure 14. Graphic comparison of the test results obtained for five products, each tested on five subjects

This type of sensor was specifically chosen because it records all of the moisture present in the stream. It is insensitive to relative humidity variables in the gas stream caused by temperature changes. The slight changes which may occur in the gas velocity due to small temperature changes are negligible.

Finally, the electric signals representing changing moisture levels in the two gas streams are delivered to an x-y recorder (Fig. 12). This machine automatically plots the values received from each area being measured against one another. The simultaneous variations in perspiration rates represented by these values appear as a line pattern with a

764

distinct slope representing the desired ratio. Retraces should be run for a period of time, generally 15 to 30 min, which is sufficient to produce a suitable pattern from which an accurate slope may be determined. After each test a blank run is made to determine the normal slope the machine will produce with both cups sensing equal moisture levels. This may be done by first sealing the sensing part of the cups with plastic caps. Then, manual manipulation of the wet gas valve will cause this calibration line to be drawn. Deviations in the slopes of these lines appearing in a set of tests may be utilized to correct the test associated with them. It has become standard practice to correct all charts to a calibration with a slope of 1. All charts may then be compared directly.

RESULTS

A series of four charts (Fig. 13) illustrates the information developed from a test designed to obtain the maximum perspiration reduction in four hours. The testing procedure is as outlined in Table I.

In Fig. 13a the pattern labelled "control" is the recording obtained from an individual with no product applied to either side. The pattern labeled "calibration" represents what the system will record when both cups are sensing equal or nearly equal moisture levels. This graph represents the type of initial control information obtained from each test subject and demonstrates the normal ratio for the subject under test before a product is applied.

Figure 13b shows the new ratio (test 1) obtained, after the first application (See Table I) has depressed the perspiration of the left side. The dotted line lobeled "control" has been drawn in to show the relationship of the original ratio to the new ratio labeled test 1. A reduction of 75% of the original perspiration rate may be calculated from this chart.

Figures 13c and d show the further change in the ratios measured as more product is applied in accordance with the schedule in Table I. When compared to the original control line, the pattern, labeled test 2 in Fig. 13c, is lower than the one for test 1 and represents a depression in the perspiration rate of 96.5% in Fig. 13d. The pattern for Test 3 is even lower and represent a 98.5% reduction of the perspiration rate.

Data showing the effect of a product on five subjects which was obtained in the manner described above are compiled in Table II. One can see how the effect of a product can vary from individual to individual, particularly on the initial application. Similar data comparing one experimental and four commercial products in Fig. 14 show a wide

	Product		
Time	Test	Application"	
0	V		
.5		N	
1			
1.5	N		
2		1	
2.5	\checkmark	N	
3		1	
3.5	V		
4			

Table I									
Dutline	of	the	Testing	Procedure					

" Product applied before test.

 Table II

 Typical Results Obtained for a Test of One Product on Five Subjects

Product: A	Test No. 1	Test No. 2	Test No. 3	Average	Dev.	Dev. ²
W. V.	89.0	80.0	94.5	87.8	+ 5.6	36
J. M.	80.0	98.5	98.5	92.7	+10.5	121
L.M.	95.0	96.5	95.0	95.5	+13.3	169
F. S.	22.5	73.5	82.5	59.5	-22.7	529
R. G.	41.0	98.0	96.0	78.3	-3.9	16
		*				
Average	65.4	89.3	92.0	82.7		S.D. ± 14.7

All values are per cent depression of perspiration.

variety of results. Considering the implication of the claims made for the commercial products B, C, D, and E, much investigation is still needed before these implications of high efficiency may be stated as fact.

SUMMARY

In summarizing, a new electronic system of measuring anti-perspirancy has been described. Since it continuously records the ratio of perspiration rates between two areas it has illuminated several facts:

1. The ratio of perspiration rates between two areas on the same human body remains constant over a considerable range of perspiration. The original assumption that this was true has been proven to be correct.

2. Previous methods which developed the ratio from the averages of recorded data have had an unnoticed additive error built into their calculations.
3. The new method has demonstrated a means of eliminating this error in order to obtain more accurate information about the effect of antiperspirants on human subjects.

4. The method produces results about as rapidly as the subject in test can generate perspiration.

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Caucasian Hair, Negro Hair, and Wool: Similarities and Differences

J. MENKART, Ph.D., L. J. WOLFRAM, Ph.D., and IRENE MAO, M.D.*

Presented May 10, 1966, New York City

Synopsis—Caucasian hair, Negro hair, and Lincoln wool are compared in terms of physical structure, mechanical properties, chemical composition, and rate of reaction with various reagents.

Several significant differences are found between wool and the two hair samples. The principal one is the extent of crosslinking, which is greater in the hair, as indicated by higher sulfur and cystine contents, and a higher proportion of the material found in the γ -keratose fraction after oxidation. The heavier crosslinking is reflected in greater resistance to attack by hot acid and in slower reduction by thioglycolate or sulfite solutions.

The main difference between Caucasian and Negro hair is in fiber geometry; the Caucasian fiber approximates a cylinder, the Negro a twisted oval rod. In their physical and chemical properties, the two fibers are very similar.

I. INTRODUCTION

Human hair, the wool of the sheep, and the hairs of the various goat species of textile importance (angora, cashmere) are closely related to each other morphologically, chemically, and physically. The similarities between them are, in most respects, so close that fiber investigators at one time developed the habit of treating them as almost interchangeable. It became customary to employ human hair for studies of fiber mechanical properties directed to the understanding of wool; the following quotation (1) is typical of numerous papers of the 30's and early 40's: "Human hair was selected for use in preference to wool

^{*}Harris Research Laboratories, Inc., Gillette Research Institute, Washington, D. C. 20007.

because of the greater uniformity of cross-sectional area of the fiber along its length."

The practice came to be used as the realization grew that the various animal fibers differ significantly from each other in some important respects and that consequently caution needs to be exercised in applying results obtained on one fiber type to other keratin fibers. In Section II of this paper, differences between wool and hair are discussed; published data are the main source of the survey.

A narrower fiber comparison of interest to the cosmetic chemist is that between human scalp hair of different races. In this field, literature information is scanty except for data on fiber diameter and cross-sectional shape gathered in anthropological studies (2–5). A comparison of some chemical, physical, and mechanical properties of Caucasian and American Negro hair was therefore undertaken; a standard wool (Lincoln) was examined at the same time. The results are reported in Section III.

II. WOOL AND HAIR

In evaluating published comparisons of wool and hair, the problem of intraspecies differences comes into play. Most of the literature data are based on Caucasian hair, but in the case of wool many breeds and crossbreeds have been employed. The interbreed differences in most properties are probably minor, but in the review which follows the breed is specified wherever known.

1. Morphological

Both wool and hair fibers have the same main morphological components—cuticle, cortex, and medulla. The cuticle, which consists of overlapping flat scale cells about $0.5 \ \mu$ in diameter, surrounds the fiber. In wool, the cuticle layer is 1–2 scale-cells thick; in hair, 5–6 cells (6). The cuticle is more resistant to diffusion of reagents than the cortex (7) and more resistant to attack by chemical reagents, such as sodium sulfide (8). It thus acts as a protective barrier to the body of the fiber against chemical as well as mechanical degradation. The greater resistance of human hair to chemical attack by many reagents (see below) is at least partly due to the thickness of the cuticular layer.

The cortex of keratin fibers consists of spindle-shaped cells, oriented along the axis of the fibers. In wool, the cortex is divided into two components, differing from each other in their structure and reactivity:*

^{*} They are named (9) ortho- (the more reactive component) and para-cortex (the less reactive one).

HAIR AND WOOL

in Merino wool fibers, the two portions lie side by side, each comprising about half the cortex, and are wound around each other in phase with the fiber crimp (10, 11); in crimpless wools, the *ortho* component occupies the core of the fiber, and the *para* is located around it in the form of an annulus (12). No such differentiation is noted in Caucasian hair fibers; their cortex appears homogeneous, and it resembles the *para* component of the wool fiber cortex in composition and properties (13).

The protein within the cortical cells is organized in the form of parallel microfibrils, embedded in a matrix. The microfibrils, which represent the crystalline component of the structure, contain little or no sul-



Figure 1. The adsorption regain isotherms of wool (19) and hair (20)

fur; the matrix is sulfur-rich and amorphous (14). In the *para*-portion of the fine wool cortex and in the cortex of hair, the microfibrils are packed in a hexagonal array. In the *ortho*-cortex of wool or mohair, the microfibrils are arranged in the form of whorls or spirals; the contrast between the microfibrils and the matrix, as revealed by staining with osmium, is much lower (14).

The innermost morphological component, the medulla, is not invariably present either in wool or in hair, though it is more common in hair. In any case, it is believed to make little or no contribution to the chemical and mechanical properties of the fiber.

2. Chemical Composition

A keratin fiber does not constitute a single chemical entity. As already indicated, a complex substructure is present even within the individual morphological components. Thus, the whole fiber is clearly



Figure 2. The swelling of wool (21) and hair (23) in aqueons HCl

a mixture of proteins, and a total analysis represents an average value.

The cuticle contains more cystine than the cortex (15), and there are differences in other amino acids, also (16); the *para*-cortex of wool contains more sulfur than the *ortho*-cortex (13). It is therefore to be expected that hair, with a higher ratio of cuticle to cortex and a cortex which is wholly *para* in nature, should differ significantly in its amino

Tensile Proper	ties of Merine	o Wool and Ha	ir at 21°C	
	At 65% R.H. (24)		In pH7 Buffer (25)	
Property	Hair	Wool	Hair	Wool
Initial modulus, mg cm ⁻²	55	31	21	12
Stress at yield point, mg cm ⁻²	1.16	0.62		
Stress at 20% extn., mg cm ⁻²			0.52	0.45
Extension to post-yield, %			27	29
Post-yield modulus, mg cm ⁻²		* + *	5.3	3.4
Extension at break, %	37	20	51	41
Stress at break, mg cm ⁻²	2.0	1.1	1.7	0.8

 Table I

 Tensile Properties of Merino Wool and Hair at 21°C

Table II Dye Uptake of Different Fibers (7)

Fiber	Half-Dycing Time (Hr)	Diffusion Coefficient (Arbitrary Units)
Human hair A	56	7.5
Human hair B	-1-1	7.8
Human hair M	47	8.5
Human hair N	54	8.7
80's Merino wool	1.8	10.7
56's Down wool	2.0	17.4

HAIR AND WOOL

acid composition from wool. A comparison of a human hair sample with those from several breeds of wool indeed shows the hair to be richer in cystine and proline and poorer in alanine, leucine, tyrosine, phenylalanine, glutamic and aspartic acids, lysine and arginine. Real, but smaller, differences are seen among the various wool samples (17).

3. Acid Binding, Moisture Regain and Swelling

The acid-binding of hair, which is an index of the number of basic groups present, is slightly lower for hair than for wool (18); this is in accord with the lower basic amino acid content of hair, noted above.

Wool and hair have practically identical moisture-adsorption isotherms at relative humidities up to 95%. Here, the regain* curves



Figure 3. A typical keratin fiber stress-strain curve

diverge (Fig. 1); in saturated water vapor, wool had a regain of 33-34% (19) and hair of about 30% (20).

The difference in saturation swelling is probably a function of the extent of restraint imposed by crosslinking. The effect is shown still more strikingly (Fig. 2) when the additional swelling caused by transfer from water to a strongly acidic medium is examined: Cotswold wool (21) and 64's Merino wool (22) exhibit a volume swelling of about 6% when transferred from water to HCl at pH 1, whereas human hair swells about $3\frac{1}{2}\%$ under the same conditions (23).

4. Mechanical Properties

The fiber tensile properties vary within an animal species and even from one fiber to another within a lock. One may nevertheless state

^{* &}quot;Regain" is the moisture content based on the dry weight of the material.

broadly that hair is somewhat stiffer, stronger, and more extensible than wool, both in air and wet. Typical data are given in Table I.

The lower elastic modulus of wool indicated by such extensometric data is due partly to the perturbing effect of fiber crimp; a greater rate of stress relaxation, made possible by the lower degree of crosslinking, may also play a part. The dynamic moduli of hair and Lincoln wool, as determined by sonic measurement, are closely similar (26).

Another mechanical parameter exhibiting a difference between wool and hair is the post-yield turnover point. When wool fibers are extended in water at different temperatures, the location of this point on the strain axis (Fig. 3) is constant up to a characteristic temperature and then shifts to higher strains; this effect has been ascribed to a secondorder transition in the keratin (27). As shown in Table I, the extension at which the post-yield slope begins is similar for wool and hair at room temperature. However, the transition temperature is 72 °C for B. A. fleece wool (1), 70 °C for Merino wool, and 85 °C for hair examined under the same conditions (28). It appears that a higher temperature

	Fiber					
	Human Hair		64's Wool			
Property	Intact	Acid- Treated*	Intact	Acid- Treated*		
Stress at 20% extension, mg cm ⁻²	0.49	0.23	0.44	0.13		
Stress at break, mg cm ⁻²	1.28	0.46	1.18	0.19		
Energy to break, g cm ^{-2} \times 10 ^{-1} †	30	11	30	4		
Extension to break, 70	58	51	51	30		

Table III	
Fiber Mechanical Properties in pH 9.2 Buffer-Effect of Acid (34)

* 0.04 N H₂SO₄, 16 hours at the boil.

† Per unit volume.

	Residue Weight, %		
Fiber	6 N HCl	$6 N H_2 SO$	
Lincoln wool	0.06	0.06	
Caucasian hair			
Brown	2.5	2.2	
White	1.0	0.7	
Negro hair	2.7	2.3	

Table IVAcid Hydrolysis: 0.5 g Fiber in 25 ml Solution, 105°C, 18 Hours

is required in hair before disulfide-sulfhydryl interchange reaches sufficient proportions to have a significant effect on the mobility of the protein chains.

5. Diffusion of Reagents

Human hair fibers have a larger diameter than most wools; consequently, the penetration of a reagent to the core of the fiber would take longer even if its diffusion through the keratin substance were the same. In fact, for most large molecules, such as dyes, the diffusion is slower in hair.

Data derived from measurement of uptake of an acid dyestuff at 60 °C by several fibers are shown in Table II (7). The half-dyeing time, which is affected by both the fiber diameter and the diffusion coefficient, is 25 times longer for hair than for wool.

Type of Side Chain and Amino Acid	Lincoln Wool	Caucasian Hair	Negro Hair
Aliphatic	2830	2350	2470
Glycine	590	539	-541
Alanine	601	471	509
Valine	570	538	568
Leucine	740	554	570
Isoleucine	333	250	277
Aliphatic Hydroxyl	1020	1520	1290
Serine	541	870	672
Threonine	483	653	615
Aromatic	540	260	380
Tyrosine	266	132	202
Phenylalanine	273	130	179
Acidic	1400	1330	1350
Aspartic Acid	575	455	436
Glutamic Acid	828	871	915
Basic	1040	790	800
Lysine	310	213	231
Arginine	662	512	482
Histidine	71	63	84
Sulfur Containing	750	1440	1380
Half-Cystine	745	1380	1:37()
Cysteic Acid	6	55	10
Methionine	0	0	0
Heterocyclic			
Proline	490	672	662
Ammonia	1030	780	985

Table V Amino Acid Contents (Micromoles/g)



Figure 4. The rate of crosslinking of keratin by mercury, as indicated by the increase in work to stretch (29)



Figure 5. Alkali insolubility as a function of time in boiling $0.04 N H_2SO_3$ (10)

All four specimens of human hair have very similar diffusion coefficients. Those of both wools are higher: one slightly so, the other substantially.

The phenomenon has also been examined for the action of mercuric acetate on human hair and Lincoln wool, as measured by the increase in work required to stretch the fibers as shown in Fig. 4 (29). The

slopes of the lines are in the ratio of 3.9:1; correcting for the difference in mean fiber diameter between the two substances, the rates of diffusion are in the ratio of 2.2:1. (The staining of cross-sections with hydrogen sulfide showed that reagent diffusion was indeed the rate-controlling factor.) The "half-reaction times" are 5 hours for the wool and 75 hours for the hair—a fifteen-fold difference.

6. Rates of Reaction

Differences in reaction rates among the keratin fibers are well demonstrated in the two standard solubility tests, in urea-bisulfite (30)



Figure 6. Rate of development of supercontraction stress (24)

and in alkali (31). The urea-bisulfite test distinguishes quite sharply between wool and hair, the former being soluble to the extent of about 50%, the latter about 15% (32).

In the alkali-solubility test, intact keratin fibers all exhibit fairly small weight losses (4-6%) for hair, 10-12% for wool). The weight loss is increased by prior exposure to oxidizing agents or acids; this increase is much greater for wool (and mohair) than for human hair (10). The effect of exposure to boiling 0.04 N sulfuric acid is indicated in Fig. 5, where alkali *insolubility* (the weight fraction remaining after the test) is plotted, on a logarithmic scale, against the time of treatment with acid. The differences in ease of degradation are evident in microscopic examination: wool fibers, pretreated with acid and then examined in alkali, swell and lose their birefringence very rapidly, whereas hair is quite resistant (13). They are also reflected in the mechanical properties of the fiber, as indicated by the data of Table III (33).

		Fractionation		
		Keratose	: (%)	
Fiber Type	α	β	γ	Total*
Merino wool (34)	56	10	25	91
Caucasian hair	43	15	33	91
Negro hair	43	14	33	90
		Sulfur	Content (%)	
	α	β	γ	Whole Fiber†
Merino wool (34)	1.88	2.13	5.84	3.38(2.72)
Caucasian hair	2.56	3.69	6.04	5.45(3.65)
Negro hair	2.38	4.00	6.60	5.40(3.77)

Table	VΙ
raction	ation

* The totals do not sum to 100%, due to incomplete recovery; the deficit is believed to reside mainly in the γ -fraction.

† The figure in parentheses represents the sum of the sulfur contents of the three fractions.

Table VII Moisture Regain at 22°C

Fiber Type	Desorption Regain at 65% R.H.	Absorption Regain at 87% R.H.
Lincoln wool	15.4	23.4
Merino wool	14.8	
Caucasian hair		
White	15.2	23.3
Brown	16.2	24.1
Negro hair	16.0	24.4

Table VIII Tensile Properties

Fiber Type	Denier	Yield Stress (g/denier)	Breaking Stress (g/denier)	Extension to Break (%)
65% R.H.				
Lincoln wool	20	1.10	2.04	43
Caucasian hair	46	0.93	1.68	11
Negro hair	37	1,11	1.81	40
pH 7 Buffer				
Lincoln wool	20	0.35	1.70	65
Caucasian hair	42	0.42	1.41	47
Negro hair	38	0.46	1.24	48

HAIR AND WOOL

7. Supercontraction and Set

Different animal fibers exhibit a wide range of supercontraction values after an hour's boiling in several media (35). The kinetics of the process for different fibers have been examined in 9 M LiCl (24) and in cuprammonium hydroxide (36) with similar results: hair has a much longer initiation period before the onset of supercontraction and a slower rate of supercontraction (or generation of tension); the level of contraction or maximum force attained is sometimes also lower. Typical data are shown in Fig. 6.

In examining the acceptance of set by stretched keratin fibers in boiling water or borax, Mitchell and Feughelman (37) found that the amount of set diminished as the fiber diameter increased. On the other



Figure 7. Rate of set generation as a function of time of setting, at 40% extension (one hour's release in the same medium)

hand, Whewell (38) reports some specific differences: in setting fibers in boiling water for 2 hours, he obtained 12% set in hair and values ranging from 6 to 21% for four samples of wool.

Data from these laboratories show that Lincoln wool accepts set more rapidly than hair. The curves of Fig. 7 illustrate the results obtained with fibers set at 40% extension in boiling pH 7 phosphate buffer for the time indicated and released for one hour in the same medium.

The amount of set is affected by holding the fiber under strain prior to exposure to the setting process; this fact was noted at an early stage of studies in this field (27). The authors find that the extent of super-



Figure 8. Set as a function of time extended, in cold water, prior to setting

contraction obtained after 2 minutes' setting at 40% extension, followed by 30 minutes' relaxation (both in boiling water), diminishes linearly with the time the fiber is held stretched prior to setting (Fig. 8); here again, hair has less supercontraction at any given time than Lincoln wool, but the decrease in supercontraction with time under tension runs in parallel for the two fiber types.

III. CAUCASIAN AND NEGRO HAIR

A. Materials

1. *Caucasian Hair*—Brown European hair, from mixed women's combings, purchased from a New York hair dealer, was employed. The hair was about 28 cm long, in the "remis" state (bundled, root-to-tip oriented).

In one experiment (Table IV), white hair from the same source was used.

2. Negro Hair—A blended batch of barber's clippings from a Chicago barber shop, 3–6 mm long, from some thirty male heads* was the material used in all the work other than the tensile tests (Table VIII); these were carried out on fibers from one female head, purported to have had no hot combing or chemical treatment.

3. Lincoln Wool—This was a sample (SW 296) of one of the "Standard Wools" derived from pen-reared sheep maintained in the CSIRO

^{*} Kindly provided by J. L. Underwood, The Toni Co., Chicago, Ill.



Figure 9. Cross-sections of Caucasian and Negro hair

Ian Clunies Ross Laboratory, Prospect, N.S.W., Australia (39).* This wool is essentially nonmedullated, of 36's quality (mean diameter about 40 μ).

4. Merino Wool-Australian, of 64's quality.

5. Fiber Preparation—The fibers were Soxhlet-extracted with methylene chloride for 4 hours and then with absolute ethanol for 2 hours. They were then rinsed three times with deionized water, soaked in 0.01 N HCl overnight, and washed in repeated changes of deionized water until the rinse water has a pH of 5. The fibers were then dried by exposure to an atmosphere of 65% R.H. and 70 °F, in which they were stored throughout the work.

6. *Reagents*—All the chemical reagents were of purified or higher grade.

B. Results and Discussion

1. *Morphological*—Two features distinguishes Negro and Caucasian hair at the morphological level: the irregular crimp of the Negro fiber, presumably developed by the same mechanism which is responsible for the formation of crimp in wool (40), and the more oval shape of its cross-section[†] which twists irregularly as it progresses along the fiber axis;

^{*} Kindly supplied by J. F. P. James, CSIRO Division of Textile Physics, Ryde, N.S.W.

 $[\]dagger$ The average ellipticity indices (ratio minor axis/major axis) for the two hair specimens of this study are: Caucasian 0.71, Negro 0.56.

fibers of irregular section are occasionally evident (Fig. 9). In other respects—thickness of cuticle, shape and size of scale and cortical cells, for example—there is no discernible difference. Mercer (11) reports finding evidence of a bilateral structure (see II, 1) in the Negro hair cortex. Observations here suggest that, if there is any such differentiation, its magnitude is much smaller than that found in wool.

Negro hair is typically heavily pigmented (Fig. 9). However, using the residue of hydrolysis in 6 N HCl as a measure of the melanin content (41), it appears that brown Caucasian hair and black Negro hair are very much alike (Table IV). A significant weight of insoluble material is obtained when white Caucasian hair is subjected to this test; microscopic examination of this residue shows it to consist largely of cell

	Solubility, %		
Fiber	Urca-Bisulfite	Alkali	
Lincoln wool	52.5	10.4	
Caucasian hair	27.0	5.0	
Negro hair	37.2	4.1	

, í	Tabl	ψIX	
Urca-Bisulfite	and	Alkali	Solubilities

Table N Cysteine and Cystine Contents, Ce										
	Original Fiber		Alkali Sol. Residue		UB Sol. Residue					
Fiber	CySH	CySSCy	CySH	CySSCy	CySH	CySSCy				
Lincoln wool	0.31	9.8	0.43	0.77	0.91	4.42				
Caucasian hair	0.78	17.2	0.46	2.23	1.66	11.4				
Negro hair	0.66	17.8	().46	2.57	0.96	11.0				

Table XI Acid Hydrolysis, 0.04 N H₂SO₄, Reflux

	Weight Loss (C After)					
			Alkali Solubility Test, Following			
Fiber	4 Hours	18 Hours	4 Hours*	18 Hours*		
Lincoln wool	0.4	21.1	82.7	97_6		
Merino wool	10.00	21.4	+ * +			
Caucasian hair	1.8	6.0	13.8	74.4		
Negro hair	0.2	8.0	10.5	57.4		

* Based on original weight, prior to acid exposure.

membrane fragments. The values of the last column in the Table show that $6 N H_2SO_4$ leaves a similar amount of residue.

One racial difference in the pigment has been reported, though on the basis of a very limited sample. Using the electron microscope, Swift (42) had measured the size of isolated melanin granules and found those from Negro hair to be larger than those from Caucasian (and also Chinese) hair.

2. Chemical Composition—Table V lists the results of amino acid analyses, performed by column chromatography on the three fiber samples.* Acid hydrolysates were used; tryptophan, if present, was thus destroyed. Methionine was not detected in any of the samples.

The results for the Lincoln wool and Caucasian hair are in general agreement with those reported by others (17). The main interest resides in the Caucasian-Negro hair comparison. Here, the only notable differences are the deficiency of serine and threenine and the excess of tyrosine, phenylalanine, and ammonia in the Negro hair. One cannot place any meaningful interpretation on these data at present.

Another indication of the chemical structure is given by fractionation into "keratoses," using the method developed by Alexander (43), which is based on oxidation with peracetic acid and dissolution in aqueous ammonia. The fraction insoluble in the ammonia, which is termed β -keratose, is believed to consist of cell membranes and similar material. The fraction precipitated by the adjustment of pH down to 4, called α -keratose, is thought to originate from the crystalline portion of the protein and the residue, γ -keratose, from the amorphous protein of high sulfur content.

The values for the two hair samples were determined by the procedure of Corfield, Robson, and Skinner (34); the sulfur contents were determined by the oxygen combustion method of Parisot (44). The results are given in Table VI, along with those reported by Corfield *et al.* for Merino wool.

The two hair samples fractionate almost identically. Compared with the wool, they are higher in β - and γ - and lower in α -keratose, which indicates that the hair contains a higher proportion of amorphous, highsulfur material. The sulfur contents of all three fractions are higher in the hairs, which is indirect evidence to the effect that the fractions are themselves complex mixtures of proteins and protein degradation products. Interestingly, the proportion of the sulfur content of the

^{*} We are indebted to Dr. E. Gross of the NIAMD, NIH, Bethesda, Md., for these data.



Figure 10. Rate of reduction in alkaline thioglycolate

Figure 11. Rate of reduction in sulfite at pH 6



Figure 12. The data of Figure 10 replotted in terms of fraction of disulfide bonds ruptured

wool recovered in the three fractions is lower in hair (68-70%) than in the wool (78%). The proportion of material unrecovered is the same; this must, therefore, have a higher sulfur content in the case of the hair.

3. Acid Binding and Moisture Regain—The acid binding of Caucasian and Negro hair has been compared by Sagal (45); no difference was found. The absence of pigment has no influence on the result in either kind of hair.

HAIR AND WOOL

Data for the moisture regain, determined under desorption conditions at 65% R.H. and under absorption conditions at 87% R.H., are given in Table VII. Only slight differences among the various fibers are seen.

4. *Mechanical Properties*—The data of Table VIII were obtained in single cycle force-extension tests to break on an Instron tensile tester, 2.54 cm fiber specimens being extended at 2.54 cm/min, the broken fibers being subsequently weighed on a torsion microbalance to establish their linear density. (As noted in Section III, A, 2, the Negro hair fibers are a different material from that used in the rest of the study.)

The two hair samples behave very similarly; the wool is stronger and more extensible—a finding in direct contrast to that in Table I. This strikingly illustrates the variability resulting from differences between samples and test procedures.

5. Diffusion of Reagents—Diffusion appears to be the rate-determining step in the reduction of keratin by mercaptans (46) and by sulfites (47). Thus, the rate of reduction may be used to compare the diffusion of the reagents in different fibers.

The results of such measurements at $36 \,^{\circ}$ C, using ammonium thioglycolate at pH 9.3 and ammonium sulfite at pH 6.0, are given in Figs. 10 and 11. The two types of hair give practically identical curves, except for the slightly lower reduction level of Caucasian hair in the later stages of the reaction with thioglycolate; the wool is reduced much more rapidly. When the data are considered in terms of the fraction of the disulfide bonds ruptured, the equilibrium is found to be somewhat higher for wool in the case of thioglycolate (Fig. 12) and identical for all three fibers in the sulfite experiment.

6. *Rates of Reaction*—The solubilities of the three fibers in the ureabisulfite and the alkali tests are recorded in Table IX.

As expected, the hair is more resistant to attack than wool in both media. In the UB test, a difference between the two hair types appears, the reasons for which are not clear.

When the cystine and cysteine contents of the residues from the tests were determined by the Zahn-Traumann method (48), the two hairs again behaved very similarly to each other and differently from the wool (Table X).

The response to boiling dilute acid was measured by determining the weight change (a) in the acid exposure itself and (b) in subsequent alkali solubility test. The results, which are summarized in Table XI, again show a large difference between the hair and wool, with the two hair samples quite closely matched.

IV. CONCLUSIONS

A comparison of the morphological structure, chemical composition, diffusion of reagents, rate of reaction with various agents, and setting and supercontraction behavior reveals some significant differences between wool and hair; the two hair types, Caucasian and Negro, appear very similar in all the features examined.

In acid binding and moisture regain, all the fibers are similar. The fiber mechanical properties exhibit considerable variations even within a fiber type.

The slower rate of diffusion of reagents into hair and its greater resistance to chemical modification are due to structural differences at both the morphological and molecular level; specifically, the important factors are the thicker cuticle and the more extensive covalent crosslinking of the hair keratin.

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The Action of Antiperspirants

CHRISTOPHER M. PAPA, M.D.*

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Synopsis—Stripping experiments, methylene blue iontophoresis, and histological studies after induced sweating indicate that formaldehyde forms a physical plug in the sweat duct. On the other hand, similar experiments with aluminum chloride treated skin suggest that this antiperspirant acts by altering the permeability of the sweat duct.

Our fastidious population indulges in a significant amount of concern about those usually private recesses, the axillae. Women require the area be denuded of hair, and both sexes apparently wish the underarms dry and odor free. While cold steel swiftly decides the fate of the useless pelage, the chemical attack on axillary perspiration remains unsure. If the compound to be applied has bacteriostatic properties, it will succeed as a deodorant since it retards the microbial growth which produces the offensive odor (1). Usefulness in sweat suppression, however, is a more vexing problem.

The eccrine sweat glands contribute the major portion of secretory products in the axilla. Therefore, interest has centered on chemicals which might produce anhidrosis, interference with the production or delivery of sweat to the skin surface. Both formalin and aluminum salts have been employed for this purpose for some time, without sure knowledge of their modes of action. The popular concept is that both

^{*} Department of Dermatology, University of Pennsylvania, School of Medicine, Philadelphia, Pa. 19104.

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Figure 1. Anhidrotic square of skin following treatment with 10^{c}_{c} formalin solution. The lower half of the area has been stripped to remove stratum corneum using cellophane tape. Following thermal stimulation sweat appears in the stripped area only as the superficial obstruction is relieved. The skin has been painted with a starchcastor oil suspension, and the perspiration shows as white droplets

produce some type of plug at or near the skin surface, occluding the eccrine ostium and preventing the exit of perspiration (2, 3). Formalin introduced into the skin by iontophoresis has also been reported to inhibit glandular secretion directly (4). Unable to find evidence for plugging, Sulzberger, *et al.* suggested that aluminum worked in an entirely different manner. An inflammatory infiltrate, found about the intraepidermal and high dermal portions of the duct, was thought to interfere with transport of the sweat (5). This action was said to result from "chemotaxis." By the use of sensitive *in vitro* studies, however, Blank, *et al.* concluded that aluminum did not penetrate into dermal tissue (6). It was with the hope of resolving these contradictory views that the following study was initiated.

EXPERIMENTAL

Production of Anhidrosis

Twenty healthy men served as subjects. Square gauze pads (5 cm), with either 10% formalin, 20% aluminum chloride (hexahydrate), or distilled water, were placed on the volar surfaces of forearms under an occlusive dressing of Saran Wrap^{**} and Clear Tape^{*†} overnight for 18 hours. Following removal of these patches, the men were exposed to

ACTION OF ANTIPERSPIRANTS



Figure 2. Area of volar forearm treated with aluminum chloride solution remains anhidrotic even though the lower half has been stripped to remove the horny layer. It would thus be unlikely that a superficial block is operating in this instance. The few sweat puncta appearing in the stripped site reflect the unmasking of the anhidrosis produced by the aqueous vehicle

 $38 \,^{\circ}$ C and 90% relative humidity in a controlled heat chamber. Sweating was demonstrated by painting a starch-castor oil mixture over the skin; droplets of perspiration are observed as white puncta on the surface (7). Initially all treated areas are anhidrotic, but within an hour the water site gradually recovers sweating as the skin dries. All subsequent test procedures in the formalin and aluminum areas, therefore, were carried out only after allowing time for the effects of simple hydration to disappear. When undisturbed, the aluminum and formalin anhidrosis was complete for 3 to 4 days, slowly returning to normal within 7 to 14 days. The following experimental maneuvers were then carried out on the anhidrotic sites to determine how each agent had brought about sweat suppression.

Horny Layer Removal

Half of each anhidrotic test site was stripped with Scotch Tape^{**} until the major portion of the stratum corneum was removed and the "glistening layer" was reached. Coupled with the visual estimation of

^{*} Dow Chemical Co., Midland, Mich.

[†] Johnson & Johnson, New Brunswick, N. J.

[‡] Minnesota Mining Co., St. Paul, Minn.



Figure 3. Methylene blue pattern produced by iontophoresis into normal skin. This is the typical, specific pattern normally obtained. Each punctum represents passage of dye down the duct and diffusion into the surrounding epidermal tissue

sweating, this technique immediately demonstrates whether a superficial plug is actually present. Removal of the horny layer to relieve such blockage would allow prompt return of normal sweating.

Stripping of both the water and formalin treated sites brings perspiration back to the previously dry skin (Fig. 1). The aluminum-salt treated area of anhidrosis, on the other hand, showed little except a few droplets, representing no more than reversal of residual hydration effect (Fig. 2). The tentative conclusion was that formalin and water did actually produce some type of block within the stratum corneum, while the cause of aluminum anhidrosis was deeper in the skin.

Methylene Blue Iontophoresis

When methylene blue dye is introduced into the skin by iontophoresis, a typical punctate pattern develops which is entirely localized about the eccrine ducts (Fig. 3). This staining represents transport of the dye down the eccrine ducts and diffusion through the duct wall into the



Figure 4. Methylene blue iontophoretic pattern in aluminum salt treated area. The coarse, darker puncta are produced by greater transductal permeation of the dye and more extensive epidermal staining

surrounding epidermal tissue (8). To accomplish this, the sweat glands must be active and their lumina patent. To test for these conditions, an attempt was made to produce pore patterns in the formalin and aluminum sites of anhidrosis. Thus, after the obligate one hour delay to negate the effects of hydration, a 0.1% methylene blue solution was iontophorized into the treated areas using 0.5-0.75 ma/cm² for 5 minutes.

It was not possible to produce an eccrine pore pattern in the formalin treated skin. Since the stripping experiment already demonstrated superficial blockage, passage of the dye into the ducts was not expected. In the aluminum-salt treated site, however, not only was the characteristic blue pattern present, but the puncta were sharper and larger (Fig. 4). It was thus ascertained that the ducts were patent and the glands functioning. As regards the augmented pattern, it was shown that this was due to increased transductal permeation of the methylene blue and wider diffusion into the surrounding epidermis. It now remained for histologic studies to verify these gross observations.



Figure 5. Biopsy of formalin treated skin before forced sweating. Amorphous material is seen plugging the uppermost coils of the duct within the stratum corneum. These masses are removed by the stripping procedure and allow sweat to flow onto the skin surface again. Note that the duct is not dilated. (H. & E. $250 \times$)



Figure 6. Formalin treated site after sweat stress, demonstrating distension of the sweat duet and accumulation of intraluminal masses of PAS positive diastase resistant material. These serial sections show the depth and extent of the duetal dilatation resulting from sweating into the plugged ecerine unit. Note the absence of periductal inflammation as seen in aluminum anhidrosis. (PAS with diastase digestion $\times 100$)

Effect of Forced Sweating in Anhidrotic Sites

Biopsy samples from each treated, unstripped, anhidrotic site and control volar forearm skin were obtained from five subjects immediately after removal of the occlusive patches. The men were then sweated for one hour and repeat specimens taken from sites adjacent to the prior biopsy. Tissues were fixed overnight in 10% neutral buffered formalin and sections stained with hematoxylin and cosin (H. & E.) and periodic Acid-Schiff (PAS), with and without diastase digestion.

The purpose of the sweat stress imposed on the subjects between the biopsy procedures was to assess the functional state of the secretory glands and to observe the effect of forcing perspiration into a duct which in the case of formalin treatment was presumed occluded, but more susceptible to transductal permeation in the case of aluminum salt induced anhidrosis.

A. Formalin—Pre-sweat samples in the formalin treated areas were characterized by the presence of amorphous intraluminal masses of eosinophilic material plugging the superficial coils of the eccrine duct (Fig. 5). These intraluminal masses were not PAS staining. The dermal and intraepidermal portion of the ducts appeared patent and normal. A good complement of PAS positive (diastase digestible) glycogen was contained in the secretory coil.

Following the heat stress, biopsies from the same areas showed a marked change in the microscopic picture. Ducts were widely dilated



Figure 7. Periductal inflammatiom in aluminum salt treated skin after one hour sweating. The eccrine unit is otherwise normal without ductal dilatation. The "plug-like" appearance of the horny layer is an artifact of sectioning. Pre-sweat samples of this skin are without inflammation, leading to the conclusion that perspiration pouring into the dermis invites the reaction. (H. & E. $\times 100$)

throughout their entire epidermal course and occasionally deep into the dermis. Intraluminal masses of PAS positive (diastase resistant) material, not seen in the pre-sweat samples, now were present along the length of the duct (Fig. 6). In the past this material was thought to play a role in the development of sweat retention and miliarial lesions (9), but it is more likely a secondary event due to the superficial obstruction present. Granules of the PAS positive (diastase resistant) substance are normally secreted by the sweat glands (10), and since the material cannot escape onto the skin surface when the duct is blocked it tends to accumulate intraluminally.



Figure 8. Skin biopsy from area of aluminum salt induced anhidrosis. Ductal rupture and leakage of sweat within the epidermis produce the acute inflammatory infiltrate. Note that the superficial coils above the lesion are patent, and the duct is not dilated. Clinically these lesions resembled the prickly heat rash (H. & $E. \times 250$)

Both the dilatation of the ducts and the appearance of the PAS positive nonglycogen material speak for continuing glandular function in the anhidrotic skin. Further evidence is seen by the disappearance of glycogen, the PAS positive diastase digested substance, from the secretory cells. In contrast to the pre-sweat samples, those specimens taken after the forced sweating show depletion of this material.

It is appropriate to comment that both the pre- and post-sweat samples taken from the water treated skin sites were identical in histologic appearance to those obtained from the formalin areas, with but one important exception. No discernible physical plug was present in the hydrated skin which could explain the high level blockage. This is not an unexpected result since poral closure by water is a temporary, functional event due to swelling of the horny layer cells at or near the eccrine ostium (11, 12).

B. Aluminum—Skin obtained from the areas of aluminum salt induced anhidrosis before the subjects were sweated was normal. No plugs or casts were seen, the ducts were not dilated and there was no evidence of inflammation. The secretory coil contained abundant glycogen. Following the hour of forced sweating, a striking picture had developed. A well formed infiltrate of polymorphonuclear and lymphocytic leukocytes localized in the periductal tissue about the epidermal-dermal function, where the eccrine duct traverses the rete peg (Fig. 7). Occasionally the infiltrate involved the epidermis and duct wall, coincident with spongiotic changes. This represented incipient miliarial lesions (see below). Glycogen disappeared from the secretory coil after the thermal stress, verifying normal glandular secretion.

The above sequence of histologic events are interpreted as demonstrating that aluminum chloride produces anhidrosis by altering the permeability or resorptive function of the epidermal portion of the eccrine duct. Under conditions of forced sweating the perspiration pours into the dermis faster than it can be cleared, inciting the periductal inflammatory reaction. This concept is supported by the demonstration of increased transductal permeation of iontophoresed methylene blue. It also explains why the stripping maneuver is ineffectual in restoring perspiration in aluminum anhidrosis.

Finally, it is to be reported that 3 of the 20 subjects developed scattered erythematous papules in aluminum salt treated areas only. These lesions erupted beneath the patch and closely resembled prickly heat or miliaria rubra. This diagnosis was confirmed on biopsy, which showed intraepidermal vesicles about the sweat duct, with an acute inflammatory reaction (Fig. 8). Finding miliaria only in aluminum chloride treated skin also speaks for a mechanism whereby damage to the intraepidermal duct, either anatomic or physiologic, permits transductal escape of sweat.

DISCUSSION

Superficial obstruction of the eccrine outlet has long been favored as an explanation of how spontaneous sweat disorders develop as well as how antiperspirants produce anhidrosis. The same mechanism has been indicted in sweat retention complication in the chronic dermatoses such as psoriasis and atopic dermatitis (13, 14). Such high level blockage, regardless of the cause, may be demonstrated by the following procedures: *i*, removal of the stratum corneum relieves the obstruction, bringing about an immediate return of sweating; *ii*, occluded eccrine ostia prevent JOURNAL OF THE SOCIETY OF COSMETIC CHEMISTS

development of the eccrine pore pattern produced by the iontophoresis of methylene blue; and *iii*, forced sweating into the obstructed ducts causes wide dilatation and the accumulation of PAS positive diastase resistant material. The actual nature of the occlusion is quite variable; in the chronic dermatoses it is a hyperkeratotic or parakeratotic plug, in formalin produced anhidrosis an intraluminal mass resulting from the protein precipitant properties of the chemical. In the sweat suppression by hydration, a functional poral closure alone is operative.

Aluminum anhidrosis meets none of the requirements that demonstrate a high level blockage. Removal of the horny layer does not reverse the sweat suppression and, rather than preventing formation of the methylene blue pattern, actually facilitates the staining. Forced sweating, rather than producing ductal dilatation, leads to periductal inflammation and, in extreme instances, ductal disruption as in miliaria This total picture suggests that aluminum salts alter the epirubra. dermal duct and permit large amounts of sweat to pour into the surrounding tissue. Perspiration will not reach the skin surface, in the same way that a multi-punctured garden hose, regardless of the head of pressure, prevents water from reaching the nozzle. Interestingly, Sulzberger (5) had described the same histologic findings in his studies on aluminum salt anhidrosis. Without employing the sequential biopsy technique and forced sweating as above, he concluded that the leukocytic infiltrate resulted from the "chemotaxis" of aluminum or its products and that the inflammation then interfered with ductal transport of sweat.

Finding miliarial lesions in several subjects parallels Shelley's experience with aluminum salt anhidrosis (3). This does not, however, mean that one should regularly expect to find rashes produced by underarm deodorants. As a matter of fact, these agents, primarily composed of aluminum salts, rarely cause such effects in practice. The explanation for this paradox rests in the fact that experimenters wisely choose glabrous skin such as the forearm or back, not the axilla, to study eccrine anhidrosis. While aluminum efficiently prevents perspiration in these areas, its effects in the underarm area are reduced. Miliaria probably does not develop because the chemical does not exert its full effect. The reason for the failure is not clear. The forchead is another resistant area (15), and it is interesting that both these regions (as well as the palmar surface) also do not permit easy development of eccrine pore patterns with methylene blue iontophoresis (8). It is necessary to state that these areas share a common difference which distinguishes them from the remainder of the body: The eccrine glands of the axilla, forehead, and

palm respond primarily to emotional rather than thermal stimuli. There is no evidence for anatomical differences between the eccrine units of the body. Therefore another yet unidentified variable is to be sought.

SUMMARY

Local eccrine anhidrosis was produced in human volunteers by application of 20% aluminum chloride, 10% formalin solutions, and water itself, under occlusive patches. It can be demonstrated that the sweat suppression of simple hydration and formalin is due to a high level obstruction of the eccrine duct. Removal of the stratum corneum relieves this type of anhidrosis. Methylene blue iontophoresis fails to produce a sweat pore pattern since the dye cannot enter the ducts, and there is histologic evidence of ductal dilatation following sweating into the closed system. A physical plug can be seen in the case of formalin induced anhidrosis, while the water effect is temporary and due to a functional closure of the eccrine ostium.

In aluminum salt produced anhidrosis, stripping off the stratum corneum does not bring about return of sweating, and an accentuated eccrine pore pattern results from the iontophoresis of methylene blue. Only after sweating is there an inflammatory infiltrate localized in the periductal tissue surrounding the eccrine duct at the epidermal-dermal junction. It is concluded that aluminum chloride increases transductal absorption of sweat.

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Molecular Complex Formation in Aerosol Emulsions and Foams

PAUL A. SANDERS, Ph.D.*

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Synopsis—Previous studies in nonaerosol systems have shown that certain detergents, such as sodium lauryl sulfate, form molecular complexes with long chain alcohols or acids at air/ water or oil/water interfaces. In the present investigation it has been shown that molecular complexes are also formed in aerosol emulsion systems. It was found that, in many cases, the addition of a long-chain alcohol to an aerosol emulsion system prepared with the triethanolamine salt of a fatty acid or sodium lauryl sulfate as the surfactant produced a marked increase in emulsion and foam stability and a decrease in foam drainage. In some cases foam viscosity was increased. These effects of the long-chain alcohols occur in nonaerosol and aerosol systems and are indicative of molecular complex formation.

INTRODUCTION

It has been known for a long time that combinations of certain surfactants produce unexpected effects in emulsion systems. The reason for this remained obscure for many years, and in many cases the causes are still unknown. However, the effect of combinations of surfactants and long-chain polar compounds, such as the fatty alcohols, has been shown to be due to the formation of molecular complexes between the surfactants and the alcohols. The study of these molecular complexes has clarified much of the phenomena observed with these combinations.

The investigation of molecular complexes was initiated in 1937 by Schulman and Rideal (1) with the discovery that sodium cetyl sulfate and cholesterol formed stable complexes at air/water interfaces. These complexes formed because of the attraction between the polar groups of

^{* &}quot;Freon" Products Laboratory, E. I. du Pont de Nemours and Co., Wilmington, Del.

the two compounds and the association between the hydrophobic portions of the molecules which resulted from the Van der Waal's forces of attraction. The strength of the complexes was found to be dependent upon the stereochemical configurations of the two interacting molecules (2). Sodium cetyl sulfate and cholesterol, cetyl alcohol, or elaidyl alcohol formed strong complexes. However, sodium cetyl sulfate and oleyl alcohol formed weak complexes because the *cis*-configuration of oleyl alcohol prevented a close association between the two molecules.

The effects of the complexes at oil water interfaces in emulsion systems were found by Schulman and Cockbain (3) to be similar to those observed previously at air water interfaces. Sodium cetyl sulfate alone or with oleyl alcohol gave poor emulsions of mineral oil in water, while combinations of sodium cetyl sulfate with cetyl alcohol or cholesterol gave good emulsions.

Studies of molecular complexes have also been carried out in connection with an investigation of the drainage rates of liquids through foams and the drainage properties of films from aqueous solutions. Miles *et al.* (4, 5) observed that the drainage rates of liquids through sodium lauryl sulfate foams were markedly decreased by the presence of lauryl alcohol. It was also noted that aqueous solutions containing combinations of sodium alkyl sulfates and cetyl alcohol gave slow draining films, while those with sodium alkyl sulfates alone or with oleyl alcohol gave fast draining films.

The slow draining property of the foams and films was attributed to a high surface viscosity which resulted from complex formation between the alkyl sulfates and alcohols. When the temperature of the slow draining films was increased, the film ultimately became fast draining at a specific temperature, called the film drainage transition temperature. The transition from slow to fast draining of the foams and films occurred at a temperature at which the molecules in the complex had sufficient thermal energy to overcome the bonding energies (6). Similar experiments were carried out on nonionic ethoxylated lauryl alcohols with lauryl or cetyl alcohol by Becher and Del Vecchio (7). These combinations also were found to give slow draining films.

The compositions of a number of molecular complexes have been determined. Epstein *et al.* (8) reported crystalline intermolecular products in which the sodium alkyl-sulfates and long-chain alcohols were combined in a mol ratio of 2:1. Kung and Goddard (9, 10) have also investigated the composition of complexes. Lithium, potassium, ammonium, and magnesium lauryl sulfates formed complexes in which the
COMPLEX FORMATION IN AEROSOLS

sulfate/alcohol ratio was 1:1, while calcium and tetramethyl ammonium salts did not form complexes. Complexes between stearic acid and sodium stearate with mol ratios of acid to salt of 1:1, 3:2, and 2:1, respectively, were reported by Ryer (11), while other acid/soap complexes with ratios of 1:2 were found by John and McBain (12).

The investigation of molecular complexes in aerosol emulsions and foams appeared to be a promising field of study from both a fundamental and practical aspect. Aerosol systems offered a unique opportunity to study complex formation in both an emulsion system and a foam derived from the same emulsion system. Aerosol emulsions consist of dispersions of liquefied propellants in an aqueous phase. When these products are discharged, the liquefied propellants vaporize and produce a foam. From a practical point of view, the use of emulsion systems for formulating aerosol products is now well established, and the number of aerosol products formulated as emulsion systems is increasing each year. Aerosol foams are particularly useful for cosmetic and pharmaceutical applications. These applications vary widely, and so do the properties desired for the aerosol products. Some basic information on aerosol emulsions and foams is already available (13-22), but the present knowledge is relatively small compared to what remains to be learned about these systems. If molecular complexes formed in aerosol emulsions and foams, this could provide an additional method for varying the properties of the systems. This in turn could lead to new and more effective applications for aerosol products.

Experimental

Complex formation was judged by the effect of the alcohols on the properties of the aerosol emulsions and foams. The properties that were determined were as follows:

- 1. Emulsion viscosity
- 2. Emulsion stability
- 3. Foam stiffness
- 4. Foam drainage

All of the properties, except foam stability, are summarized in tables. As a result of the difficulty of attempting to assign a numerical value to foam stability, the foam stability properties of the systems are discussed in the body of the report and are not present in the tables.

The reasons for selecting the preceding properties and the methods used to determine them were as follows:

- 5. Foam stability
- 6. Foam density
- 7. Type of discharge

Emulsion Viscosity

An increase in the viscosity of the bulk phase when an alcohol is added to an aqueous surfactant solution is generally considered an indication of complex formation between the surfactant and alcohol (4). In the present study, samples were prepared in glass bottles for visual observation. The approximate viscosity of the emulsions was estimated by inverting the bottles slowly and noting the flow characteristics of the emulsions. The viscosity was rated "low" if it was close to that of water and "high" if the emulsion was very thick. Products with viscosities in between these two extremes were rated medium.

Emulsion Stability

Emulsion stability was judged visually by the time required for phase separation to occur after the samples had been hand shaken. The samples were emulsified by a modification of the Briggs intermittent method of emulsification (23). The samples were shaken 20 times by hand, allowed to stand overnight, and reshaken 20 times immediately before stability determination. Phase separation, or creaming, is a common method for judging emulsion stability, even though it does not necessarily indicate coalescence of the droplets and breaking of the emulsions (24). Becher (25) has pointed out that, although creaming does not represent actual breaking of the emulsion, creaming is favored by large droplet size, and this may be an indication of a process which will lead to demulsification. Schulman considered phase separation to be sufficiently valid for comparison purposes when all the samples were prepared in the same way (3). As mentioned previously, Schulman found that complex formation could cause a considerable increase in emulsion stability.

Foam Stiffness

Foam stiffness was determined with a Cherry-Burrell Curd Tension Meter as described in Reference 16. Foam stiffness values are reported in grams and indicate the relative resistance of a foam to deformation resulting from the downward penetration of a curd knife. Although it is difficult to place an exact physical interpretation upon foam stiffness, it is assumed to be an aspect of foam viscosity. Complex formation has been reported to increase the surface viscosity of foams, and in the present case an increase in foam stiffness resulting from the addition of a unionized polar compound was interpreted as an indication of complex formation.

Stiffness values below about 30 indicate that the foams have low viscosity and are quite thin. Typical aerosol shaving lather foams have stiffness values ranging from about 50 to over 100.

Foam Drainage Rates

One of the most common indications of complex formation is the decrease in the rate of foam drainage that occurs with the addition of a polar compound to a surfactant solution. This is considered to be due to an increase in the viscosity of the liquid foam films, or laminae, that occurs with complex formation. Foam drainage rates were determined in the present work by discharging a known quantity of foam into a funnel and determining the amount of liquid that drained from the foam at various time intervals. With most series of foams, there were certain time intervals after discharge when the differences in drainage rates between the various foams were the most distinct. The comparative foam drainages that showed the greatest differences in the foams were selected. The time required for this varied with the different series of foams and is given in the tables.

Drainage from foams results from gravitational forces on the liquid films, which causes thinning of the foam laminae without rupture, or from rupture and collapse of the foam structure itself (26). Thus, the liquid which drains from a foam can have two different origins.

Foam Stability

Foam stability has two aspects, foam drainage and persistence of the foam. Thus, a foam may drain and become quite thin but may retain its structure. Foams also may collapse as a result of rupture of the foam structure. In the present study, comparative foam stabilities were judged visually by discharging a series of foams onto paper towels and noting the appearance of the foams after various time intervals. The rapidity with which the foam wet paper was an indication of the drainage, and the rate at which the foam collapsed was an indication of the stability of the foam structure. It was possible to line up a series of foams in order of their relative stabilities by this procedure, but it was not feasible to place any numerical value upon the stability.

Foam Density

Foam density was determined by weighing a known volume of the foam as soon after discharge as possible. The volume of the vessel used for density determinations was 350 cc.

Type of Discharge

The type of discharge of the products is indicated in the tables. Some of the products had a quiet discharge, while others had a noisy discharge. Some samples also discharged as liquids which subsequently expanded into foams. These products are described as having a liquid discharge. Others discharged a mixture of liquid and foam which also expanded into a foam. These are described as having a semiliquid discharge. The remaining products gave an immediate foam during discharge.

Microscopic Examination

Observations of the foam structures were made using a Bausch and Lomb Sterozoom microscope, Model BVB-73, equipped with a $10 \times$ paired widefield eyepiece and a power pod magnification of $3 \times$. Approximate bubble sizes in the foams were determined using a micrometer disc (#31-16-08) which measured intervals of 0.001 in.

The fact must be stressed that the bubble sizes reported are approximate. Some of the cells appeared to be fairly round, while others were very irregular in shape. The dimensions given in the report indicate the largest dimension of a specific bubble. All of the foams had bubbles which varied considerably in size. The sizes of the smallest and the largest bubbles were determined in order to show the range of sizes. No attempt was made to determine the frequency of any particular size.

Microphotographs were obtained with a Spencer triocular singlestage microscope, manufactured by the American Optical Company. It was equipped with a $15\times$ eyepiece and a 3.5 objective lens. The camera was focused through $10\times$ eyepieces with 3.5 objectives. The pictures were taken with a MP-3 Polaroid Multipurpose Industrial View camera with a 4×5 film adapter, using surface illumination and a one second exposure.

Preparation of the Aerosol Emulsions

The anionic surfactants, sodium lauryl sulfate and the triethanolamine salts of lauric, myristic, palmitic, and stearic acids were used in the present studies. Although sodium lauryl sulfate is not suitable for packaging in metal aerosol containers as a result of its corrosive effect, it has been widely used in complex formation studies in nonaerosol systems; and much information was already available about combinations of sodium lauryl sulfate and the long-chain alcohols. The triethanolamine salts of the fatty acids are used extensively in aerosol products,

and an investigation of systems with these surfactants was particularly desirable from a practical viewpoint.

Emulsions containing the triethanolamine soaps were prepared as follows: The fatty acid was heated until it melted (about 60-70 °C). An equimolar quantity of triethanolamine was dissolved in the water, and the aqueous solution was heated to about the same temperature as that of the melted fatty acid. The hot aqueous solution was then added slowly with stirring to the fatty acid. After the addition was complete, the mixture was removed from the heat and allowed to cool to room temperature with stirring. When alcohols were included in the formulation, they were melted together with the fatty acid.

Aqueous solutions of sodium lauryl sulfate were prepared by dissolving the surfactant in water. When alcohols were included, they were melted, and the aqueous solution of sodium lauryl sulfate, heated to the same temperature as that of the alcohols, was added with stirring to the alcohols.

Concentrations of the Surfactants

The concentrations of the surfactants in the aqueous phase were chosen so that the stability of the emulsions and foams from the surfactants alone would be low. Thus, any effect of the alcohols upon the system would be noticeable. Sodium lauryl sulfate, triethanolamine laurate, and triethanolamine myristate were normally used at concentrations of 0.10 m in the aqueous phase. On a weight percent basis, this amounted to about a 3.5% solution for these compounds. The addition of sufficient alcohol to produce a 1:1 molar ratio with the surfactant increased the weight percent to about 4.5%, depending upon the molecular weight of the alcohol. When other concentrations are used, this is noted in the tables.

Triethanolamine palmitate and stearate normally were used at concentrations of 0.025 m in the aqueous phase. This gave solutions of slightly over 1% on a weight basis. The addition of sufficient alcohol to give a 1:1 molar ratio with the alcohol increased the weight percent of the surfactant-alcohol complex to slightly over 2%.

Composition of the Aerosol Emulsions

Unless otherwise indicated, all aerosol emulsions were prepared with a composition of 90% aqueous phase and 10% Freon-12/Freon-114* (40/60) propellant. In some cases other propellants were tested, and this is indicated in the tables.

* Freon is a registered trademark of E. I. du Pont de Nemours and Co., Wilmington, Del.

Materials

The materials used in the present study were obtained from the following sources:

Fatty Acids

Lauric acid, myristic acid, palmitic acid and stearic acid were obtained from the E. F. Drew Chemical Corp. These acids are marketed under the trade names of "Wecoline" 1295, "Wecoline" 1495, "Wecoline" 1695, and "Wecoline" 1892, respectively.

Alcohols

Lauryl alcohol ("Lorol" 11), cetyl alcohol ("Lorol" 24), and stearyl alcohol ("Lorol" 28) were obtained from the E. I. du Pont de Nemours and Co. Olevl alcohol was supplied by Croda, Inc., myristyl alcohol by the Eastman Chemical Corp., and cholesterol by American Cholesterol Products.

Sodium Lauryl Sulfate

Sodium lauryl sulfate (Duponol* C) was obtained from the E. I. du Pont de Nemours and Co.

EXPERIMENTAL RESULTS

Sodium Lauryl Sulfate Systems

Variation in Alcohols

The addition of lauryl, myristyl, or cetyl alcohols to sodium lauryl sulfate systems increased emulsion viscosity and stability and decreased foam drainage. This is shown in Table I. Lauryl and myristyl alco-

* Duponol is a registered trademark of E. I. du Pont de Nemours & Co., Wilmington, Del.

	Variation in	Alcohols-Soc	lium Laury	l Sulfate S	ystems ^a	
			Foa	um Propert	ies	
	Emulsion Pro	operties	Drainage	Stiffness	Density	Type of
Alcohol ^b	Viscosity	Stability	(60 min)	(g)	(g/cc)	Discharge
None	Low	<1 min	82	11	0.055	Quiet
Lauryl	Medium	>5 hr	0	40	0.064	Slightly noisy
Myristyl	Medium	>5 hr	0	38	0.068	Noisy
Cetyl	Low to medium	>5 hr	2	14	0.062	Quiet
Stearyl	Low	<5 min	71	12	0.071	Quiet
Oleyl	High	30-60 min	84	10	0.067	Quiet
Cholesterol	Low to medium	1-5 min	84	13	0.061	Quiet

Table I

^a Sodium lauryl sulfate concentration = 0.10 M.

^b Sodium lauryl sulfate/alcohol ratio (molar) = 1:1.



Figure 1. Photomicrograph of a sodium lauryl sulfate foam



Figure 2. Photomicrograph of a sodium lauryl sulfate/lauryl alcohol foam

hols increased foam stiffness. Judging by these results, sodium lauryl sulfate forms complexes with these alcohols in the aerosol systems. Stearyl alcohol, oleyl alcohol, and cholesterol had little effect.

The effect of the alcohols upon foam stability was quite marked. Wetting results correlated well with drainage tests. Foams from sodium lauryl sulfate alone, or in combination with oleyl alcohol and cholesterol, caused immediate wetting, followed by that with stearyl alcohol. Foams with lauryl, myristyl, or cetyl alcohol did not wet for at least an hour.

The effect upon foam persistence was similar. The foam from sodium lauryl sulfate alone disappeared completely in 15 minutes, followed shortly thereafter by that with oleyl alcohol and cholesterol. The foam with stearyl alcohol was partially collapsed after one hour, while the remaining foams showed slight thinning but very little other change.

The effect of the alcohols upon wettability and foam persistence can be summarized as follows:

Foam wettability (in order of increasing wettability)

Lauryl alcohol	None
Myristyl alcohol < Stearyl alcohol	< Oleyl alcohol
Cetyl alcohol	Cholesterol

Foam persistence (in order of decreasing foam persistence)

Lauryl alcohol Myristyl alcohol > Stearyl alcohol > Oleyl alcohol > None Cetyl alcohol Cholesterol

Microscopic observation of the sodium lauryl sulfate foams showed bubble sizes ranging from about 0.001 to 0.01 in., with laminae thicknesses of about 0.001 to 0.003 in. The foams with lauryl alcohol present had a smaller bubble size and thinner laminae. The differences between the two foams are illustrated very effectively by the microphotographs in Figs. 1 and 2. These pictures indicate that complex formation between sodium lauryl sulfate and lauryl alcohol decreases bubble size considerably.

Variation in Concentration of Lauryl Alcohol

Increasing the concentration of lauryl alcohol in sodium lauryl sulfate systems increased emulsion stability and decreased foam drainage, as shown in Table II. The samples that were used to obtain the data in Table II were also photographed four hours after they had been shaken

Sulfate"/		÷	Fo	am Properties	5 ^b
Ratio (molar)	Viscosity	Stability	Drainage (60 min)	Stiffness (g)	Density (g/cc)
1:0	Low	<1 min	93	12	0.056
$1:\frac{1}{4}$	Low	$<1 \min$	33	34	0.059
1:1/2	Low	15-20 min	24	32	0.062
1:34	Low	30-60 min	4	38	0.063
1:1	Medium	>16 hr	0	34	0.065

1	n	1. 1		т	r
	1.21	n	e		
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Variation in Concentration of Lauryl Alcohol in Sodium Lauryl Sulfate Systems

^{*a*} Sodium lauryl sulfate concentration = 0.10 M.

^b All systems had a quiet discharge.

so that the effect upon emulsion stability could be illustrated visually. This is shown in Fig. 3. The sample on the left in Fig. 3 contained no lauryl alcohol and showed complete phase separation. The remaining samples had increasing concentrations of lauryl alcohol, corresponding to the compositions given in Table II. The sample on the extreme right, which had the highest sulfate/alcohol ratio (1:1), showed no phase separation.



Figure 3. Comparative emulsion stabilities of sodium lauryl sulfate systems with increasing concentrations of lauryl alcohol (from left to right). The picture was taken four hours after the samples were shaken

An increase in foam stiffness occurred with the first addition of lauryl alcohol, but subsequent additions caused little change. There appeared to be a slight tendency towards increasing density with increasing lauryl alcohol concentrations.

Foams with sulfate/alcohol ratios up to $1:\frac{1}{2}$ wet paper immediately, but the other foams with higher concentrations of alcohol did not wet

for an hour. The foam with no lauryl alcohol also collapsed soon after discharge, but the remainder of the foams retained their shapes for at least an hour. However, those that wet paper became quite thin on aging as a result of loss of liquid from drainage. In this series, as in many of the series to be discussed later, it is possible to select foams which wet and collapse, or which wet and retain their structure, or are quite stable.

Variation in Propellants

The effect of different propellants upon the properties of sodium lauryl sulfate/lauryl alcohol systems is shown in Table III. Freon-12 and Freon-12/Freon-114 (40/60) gave foams with the lowest drainage rates, and Propellant 152a and Propellant 142b gave foams with the highest. The former two propellants gave the stiffest foams and the latter two the lowest density foams.

Wettability data generally correlated with the foam drainage results. The foams with Propellant 152a and Propellant 142b wet paper within five minutes after discharge. The foams with the other propellants did not wet during at least one hour after discharge. The foam with Propellant 152a collapsed within 15 minutes and that with Propellant 142b collapsed within one hour. The other foams retained their shape.

Microscopic examination did not reveal any significant differences between foams formulated with Freon-12, Freon-12/Freon-114 (40/60), Freon-114, or Freon-12/Freon-11 (50/50). However, the foams with Propellant 142b or Propellant 152a had much larger laminae that

			For	im Propert	ies	
Propellant	Emulsion	Properties Stability	Drainage	Stiffness	Density	Type of Discharge
Tropenant	Viscosity	Stability	(2 111)	(g)	(g/cc)	Discharge
Freon-12	High	>5 hr	20	28	0.061	Slightly noisy
Freon-12/Freon-1	14					
(40/60)	High	>5 hr	0	44	0.062	Quiet
Freon-114	High	>5 hr	0	48	0.073	Quiet
Freon-12/Freon-1	1					
(50/50)	High	$\geq 5 hr$	2	16	0.059	Noisy
Propellant 142b	High	>5 hr	41	16	0.043	Slightly noisy
Propellant 152a	Medium	>5 hr	84	14	0.039	Slightly noisy

	Table III					
Variation in	Propellant-Sodium	Lauryl Sulfate"/Lauryl	Alcohol	Systen		

^a Sodium lauryl sulfate concentration = 0.10 M.

^{*h*} Sulfate/alcohol ratio (molar) = 1:1.



Figure 4. Photomicrograph of a sodium lauryl sulfate/lauryl alcohol foam with Freen-12/Freen-114 (40/60) propellant



 $\label{eq:Figure 5.} Figure 5. Photomicrograph of a sodium lauryl sulfate/lauryl alcohol foam with fluorocarbon 142b as the propellant$

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ranged in thickness from about 0.003 to 0.008 in., and the entire system appeared to be very fluid. The foams with the other propellants had a closer packed bubble structure with laminae approximating 0.001 in. in thickness. Microphotographs of the foams prepared with Freon-12/ Freon-114 (40/60) and Propellant 142b are illustrated in Figs. 4 and 5. The photographs show the increase in foam bubble size that occurs with Propellant 142b.

The effect of the propellants in systems with sodium lauryl sulfate alone was also investigated. However, in all cases, the foams were very unstable, and no differences due to a variation in propellants were detected.

Triethanolamine-Fatty Acid Systems

Variation in Alcohols

The effect of various alcohols upon the properties of triethanolaminefatty acid systems is shown in Tables IV, V, VI, and VII. Cholesterol was evaluated separately, and the effect of cholesterol, in comparison with that of the fatty alcohol corresponding to the fatty acid used, is shown in Table VIII. In all of the systems, certain alcohols caused a pronounced increase in emulsion viscosity and stability and a decrease in foam drainage. In these cases, molecular complex formation between the alcohols and the triethanolamine salts was judged to have taken place as a result of the effect of the alcohols upon the properties of the systems.

In each triethanolamine-fatty acid system, certain alcohols were more effective than others as far as complex formation with the triethanolamine salt was concerned. The data indicate that there is a slight tendency for the alcohols having about the same number of carbon atoms as that of the fatty acids involved to be the most effective for that particular system. Thus, lauryl alcohol was fairly effective in forming complexes with triethanolamine laurate but not with triethanolamine stearate. Likewise, stearyl alcohol forms strong complexes with triethanolamine stearate but not with triethanolamine laurate.

The over-all effects of the alcohols upon the various properties of the triethanolamine-fatty acid systems are summarized in Table IX. This summary should aid in the selection of a triethanolamine-fatty acid system with specific emulsion and foam properties. The effects of the various alcohols upon foam stability varied with the particular system involved, and each system will be discussed separately.

	Emulsion Properties Viscosity Stability		Foam Properties ^{c, d}		
Alcohol [»]			Drainage (30 min)	Stiffness (g)	
None	Low	<1 min	82	8	
Lauryl	Low to medium	>5 hr	2	20	
Myristyl	Low to medium	>5 hr	0	33	
Cetyl	Medium	>5 hr	0	18	
Stearyl	Low to medium	>5 hr	67	10	
Oleyl	Low	15-30 min	2	12	

Table IV
Variation in Alcohols-Triethanolamine Laurate ^a System

^{*a*} Triethanolamine laurate concentration = 0.10 M.

^b Soap/alcohol ratio (molar) = 1:1.

° All discharges are quiet.

^d Foam densities range from 0.062 to 0.067 g/cc.

Table V	
Variation in Alcohols—Triethanolamine Myristate ^a	Systems

			Foam Pr	operties ^c	
	Emulsion Pro	perties	Drainage	Stiffness	Type of
Alcohol ^b	Viscosity	Stability	(2 hr)	(g)	Discharge
None	Low	1-5 min	47	36	Quiet
Lauryl	Medium to high	>5 hr	0	48	Slightly noisy
Myristyl	Medium to high	>5 hr	0	48	Slightly noisy
Cetyl	Medium to high	>5 hr	0	28	Slightly noisy
Stearyl	Medium to high	>5 hr	4	26	Slightly noisy
Oleyl	Medium	>5 hr	3	22	Quiet

^{*a*} Triethanolamine myristate concentration = 0.10 M.

^b Myristate/alcohol ratio (molar) = 1:1.

 $^{\rm c}$ Foam densities ranged from 0.067 to 0.073 g/cc.

Ta	ble	VI	

			Fo	am Propert	ies	
	Emulsion	Properties	Drainage	Stiffness	Density	Type of
Alcohol [*]	Viscosity	Stability	(60 min)	(g)	(g/cc)	Discharge
None	Low	<1 min	33	25	0.09	Slightly noisy— liquid
Lauryl	Low	<1 min	27	20	0.14	Noisy-semiliquid
Myristyl	Medium	>24 hr	1	32	0.15	Noisy—liquid
Cetyl	Medium	>24 hr	5	34	0.18	Noisy—liquid
Stearyl	Medium	$>\!24~{ m hr}$	2	29	0.15	Noisy-liquid

Variation in Alcohols-Triethanolamine Palmitate" Systems

^a Triethanolamine Palmitate concentration = 0.025 M.

^b Palmitate/alcohol ratio (molar) = 1:1.

			Foa	m Properti	ies	
	Emulsion I	Properties	Drainage	Stiffness	Density	Type of
$Alcohol^b$	Viscosity	Stability	(2 hr)	(g)	(g/cc)	Discharge
None	Low	<1 min	53	14	0.19	Noisy—liquid
Lauryl	Low	5-15 min	52	8	0.21	Noisy—semi- liquid
Myristyl	Low to medium	$>\!24$ hr	0.5	32	0.17	Noisy—liquid
Cetyl	Medium	>24 hr	2	21	0.27	Noisy-liquid
Stearyl	Medium	$>\!\!24~\mathrm{hr}$	4	15	0.33	Noisy-liquid

 Table VII

 Variation in Alcohols—Triethanolamine Stearate^a Systems

^a Triethanolamine Stearate concentration = 0.025 M.

^{*b*} Stearate/alcohol ratio (molar) = 1:1.

				Foa			
Fatty	Alcohol ^b	Emulsion 2	Properties Stability	Drainage	Stiffness	Density	Type of
Acid	AICOHOI	VISCOSILY	Stability	(00 mm)	(g)	(g/cc)	Discharge
Lauric	None	Low	1-5 min	84	6	0.06	Quiet
Lauric	Lauryl	Low to medium	>24 hr	11	20	0.06	Quiet
Lauric	Cholesterol	Low to medium	>24 hr	36	3	0.08	Noisy
Myristic	None	Low	1-5 min	34	34	0.06	Quiet
Myristic	Myristyl	High	>24 hr	0	50	0.07	Noisy
Myristic	Cholesterol	Medium	>24 hr	5	17	0.10	Noisy
Palmitic	None	Low to medium	5-15 min	0	39	0.06	Quiet
Palmitic	Cetyl	High	>24 hr	0	42	0.09	Slightly noisy
Palmitic	Cholesterol	Medium	>24 hr	2	12	0.10	Slightly noisy
Stearic	None	Low to medium	5–15 min	0	30	0.11	Noisy—semi- liquid
Stearic	Stearyl	High	>24 hr	0	88	0.23	Noisy
Stearic	Cholesterol	Medium to high	>24 hr	2	16	0.12	Slightly noisy

Table VIII Effect of Cholesterol in Triethanolamine—Fatty Acid Systems

^{*a*} Triethanolamine-fatty acid concentration = 0.10 M.

^{*b*} Soap/alcohol ratio (molar) = 1:1.

Triethanolamine Laurate Systems—Systems which caused the most rapid wetting generally had the highest drainage rates. In the triethanolamine laurate series, the foams from triethanolamine laurate or those with oleyl or stearyl alcohols wetted paper almost immediately after discharge. These foams also had the highest drainage rates. Foams with lauryl, myristyl, or cetyl alcohols did not wet paper for over an hour.

The effect of the alcohols upon foam persistence followed a somewhat similar pattern. The foams from triethanolamine laurate alone, or in combination with oleyl or stearyl alcohols, started to collapse within 30 minutes after discharge. Foams with lauryl, myristyl, or cetyl alcohols retained their structure for over an hour.

Triethanolamine Myristate Systems—All of the alcohols increased the stability of triethanolamine myristate foams. The foam from triethanol amine myristate wetted paper immediately, but none of the foams containing lauryl, myristyl, cetyl, stearyl, or oleyl alcohol wetted paper during one hour after discharge. These results correlate with the foam drainage results.

Although the foam from triethanolamine myristate alone showed rapid drainage, it maintained its structure for over an hour but became progressively thinner. The foams with lauryl and oleyl alcohols showed some thinning after 30 minutes. The most stable foams were obtained with myristyl, cetyl, and stearyl alcohols.

Triethanolamine Palmitate and Stearate Systems—Foams from triethanolamine palmitate or stearate also wetted paper immediately, followed by those that contained lauryl alcohol. It must be remembered that the triethanolamine palmitate and stearate systems have a soap concentration only one-fourth that of the laurate and myristate systems. The foams with myristyl, cetyl, or stearyl alcohols did not wet paper for at least two hours. Those wetting results again correlate well with the foam drainage data.

The foams from triethanolamine palmitate or stearate alone collapsed after about one hour; those with lauryl alcohol became increasingly thinner as a result of drainage but maintained their structures for at least two hours. Foams with myristyl, cetyl, or stearyl alcohols developed slight surface crazing during two hours but otherwise showed little change.

One of the most interesting effects of the alcohols in the triethanolamine palmitate or stearate series was upon the product discharge. All of the systems gave a liquid or semiliquid discharge which subsequently expanded into a foam. However, the rate at which the product expanded into a foam after it had been discharged was a function of the chain length of the alcohol. For example, the discharge from triethanolamine stearate alone expanded almost immediately into a foam, and was followed by that with lauryl alcohol. The discharge

		Emulsion	Properties	Foam Properties			
Fatty Acid	Alcohol	Viscosity	Stability	Stability	Drainage	Stiffness	
Laurie	Lauryl	х	XX	XX		X	
	Myristyl	х	XX	XX		XX	
	Cetyl	XX	хx	XX		Х	
	Stearyl	Х	XX	0	0	0	
	Oleyl	0	0	0		0	
	Cholesterol	х	XX	-	-	0	
Myristic	Lauryl	XX	XX	х		0	
	Myristyl	хx	XX	XX		0	
	Cetyl	XX	XX	XX		0	
	Stearyl	х	X X	х	-	~	
	Oleyl	х	XX	х	0	-	
	Cholesterol	XX	XX	-	-	-	
Palmitic	Lauryl	0	0	х	ο	0	
	Myristyl	XX	XX	XX		0	
	Cetyl	XX	XX	XX		0	
	Stearyl	XX	XX	XX		0	
	Cholesterol	х	XX	-	0	-	
Stearic	Lauryl	0	0	0	0	0	
	Myristyl	х	XX	XX		5	
	Cetyl	XX	XX	XX		0	
	Stearyl	XX	XX	XX		0	
	Cholesterol	XX	XX	-	0	-	

 Table IX

 Effect of Various Alcohols upon Properties of Triethanolamine–Fatty Acid Systems

x = Positive effect.

xx = Pronounced positive effect.

o = Essentially no effect.

- = Negative effect.

- - = Pronounced negative effect.

from the system containing myristyl alcohol required still more time to expand into a foam, and that with stearyl alcohol required almost five minutes for expansion into a foam. The rate at which the liquid discharges expand into a foam may be an indication of the relative strength of the molecular complexes. The strongest complexes might be expected to show the most resistance to expansion during vaporization of the propellant after the product had been discharged.

The foam density also increased in the triethanolamine stearate/

alcohol series as the molecular weight of the alcohol increased. Again, this effect may be related to the strength of the molecular complex.

Microscopic Examination—Microscopic examination of the triethanolamine laurate and triethanolamine laurate/lauryl alcohol foams did not reveal any marked differences, although the presence of lauryl alcohol appeared to cause a slight decrease in bubble size. However, the addition of cetyl alcohol to triethanolamine palmitate systems or the addition of stearyl alcohol to triethanolamine stearate foams caused a very noticeable decrease in bubble size. This effect of the molecular complexes upon the bubble size of the foams is similar to that noted with the sodium lauryl sulfate foams. The effect of stearyl alcohol in decreasing the bubble size of triethanolamine stearate foams is illustrated by the microphotographs in Figs. 6 and 7. These foams had the compositions described in Table VII.

The effect of cholesterol upon the bubble size of foams was opposite to that observed with the long-chain alcohols. Cholesterol caused a noticeable increase in bubble size. This is illustrated by the microphotographs in Figs. 8 and 9 of triethanolamine palmitate systems with and without cholesterol. The data in Table VIII also show that cholesterol decreased foam stiffness, which again is an effect opposite to that found with long-chain alcohols. These two effects, the increase in bubble size and the decrease in foam stiffness, suggest that the triethanolamine-fatty acid/cholesterol complex is very fluid, and during discharge the vaporization of the propellant into a gas expands the film more than when cholesterol is not present. This would explain both the increase in bubble size and the decrease in foam stiffness.

Variation in Alcohol Concentration

The effect of increasing concentrations of alcohol with triethanolamine laurate/lauryl alcohol and triethanolamine myristate/myristyl alcohol systems is shown in Tables X and XI. In the triethanolamine laurate system, increasing concentrations of lauryl alcohol caused an increase in emulsion stability and a decrease in foam drainage. There was a slight tendency toward increasing foam stiffness.

Increasing lauryl alcohol concentration also increased foam stability. The foams with no lauryl alcohol or with a soap/lauryl alcohol ratio of $1:\frac{1}{4}$ wetted paper immediately after discharge. That with a soap/ alcohol ratio of $1:\frac{1}{2}$ wetted paper in about 25 minutes, and the foam with a soap/alcohol ratio of $1:\frac{3}{4}$ wetted in about an hour. These results correlated essentially with the foam drainage results.

T			Foam Properties ^b		
Alcohol Ratio	Emulsion P	Drainage	Stiffness		
(Mols)	Viscosity	Stability	(60 min)	(g)	
1:0	Low	1–5 min	86.0	8	
$1:\frac{1}{4}$	Low	30-60 min	83.0	10	
$1:\frac{1}{2}$	Low	>60 min	65.0	16	
$1:\frac{3}{4}$	Low	>16 hr	40.0	18	
1:1	Low to medium	>16 hr	11.0	22	

Table X
Variation in Lauryl Alcohol Concentration-Triethanolamine Laurate Systems

^a Triethanolamine laurate concentration = 0.10 M.

^h All discharges were quiet.

 $^\circ$ All foam densities ranged from 0.062 to 0.063 g/cc.

Table XI	
Variation in Myristyl Alcohol Concentration-Triethanolamine Myristate Sys	tems

Muriatuto#/			Foam Pr	operties ^b	
Alcohol Ratio	Emulsion	Properties	Drainage	Stiffness	Type of
(Mols)	Viscosity	Stability	(3 hr)	(g)	Discharge
1:0	Low	15-30 min	56	42	Quiet
1:1/4	Low	>5 hr	.2	45	Quiet
$1:\frac{1}{2}$	High	>5 hr	1	42	Slightly noisy
$1:3/_{4}$	High	>5 hr	0	52	Noisy
1:1	High	>5 hr	0	56	Noisy

" Tricthanolamine myristate concentration = 0.10 M.

^h Foam densities ranged from 0.064 to 0.069 g/cc.

			Foa			
	Emulsion P	roperties	Drainage	Stiffness	Density	Type of
Propellant	Viscosity	Stability	(60 min)	(g)	(g/cc)	Discharge
Freon-12	Low to medium	>5 hr	4	18	0.052	Quiet
Freon-12/Freon-114 (40/60)	Low to medium	>5 hr	4	21	0.065	Quiet
Freon-114	Low to medium	>5 hr	2	26	0.067	Quiet—semi- liquid
Freon-12/Freon-11 (50/50)	Low to medium	>5 hr	44	8	0.053	Quiet
Propellant 142b	Low	>5 hr	65	12	0.042	Noisy
Propellant 152a	Low to medium	>5 hr	3	14	0.036	Noisy

Table XII



Figure 6. Photomicrograph of a triethanolamine stearate foam



Figure 7. Photomicrograph of a triethanolamine stearate/stearyl alcohol foam

The same order occurred with foam persistence. The foam with no alcohol disappeared in about 30 minutes, and that with a soap/alcohol ratio of $1:\frac{1}{4}$ disappeared in one hour. The remainder became thin but retained most of their structure for over an hour.

In the triethanolamine myristate/myristyl alcohol series, the first increment of myristyl alcohol had a pronounced effect upon emulsion stability, foam drainage, and foam stability but subsequent additions of myristyl alcohol had much less effect. The foam with no myristyl alcohol wetted paper in about 25 minutes, while those with myristyl alcohol did not wet paper in an hour.

After two hours, the triethanolamine myristate foam had disappeared; but the remaining foams maintained most of their structure although the foams with the lower concentrations of alcohol became very thin.

Variation in Propellant

The effect of variation in propellants upon the properties of triethanolamine laurate/lauryl alcohol systems is shown in Table XII. All of the emulsions were quite stable, and differences in emulsion stability due to propellant variations were not detected. Freon-12/Freon-11 (50/50) and Propellant 142b gave the most rapidly draining foams, and Propellant 142b and Propellant 152a gave foams with the lowest density.

The foams with Freon-12/Freon-11 (50/50) propellant and Propellant 142b wet paper immediately after discharge, while foams with the other propellants did not wet during an hour after discharge. These results therefore correlate with the foam drainage data.

The foams with Freon-12/Freon-11 (50/50), Propellant 142b and Propellant 152a collapsed almost completely within 30 minutes after discharge. Those with Freon-12, Freon-12/Freon-114 (40/60), or Freon-114, retained their structure for over an hour after discharge, although they showed surface decomposition and thinning.

Variation in Soap/Alcohol Concentration

The properties of triethanolamine laurate/lauryl alcohol and triethanolamine myristate/myristyl alcohol systems at concentrations of 0.025 and $0.100 \ M$ in the aqueous phase are shown in Table XIII. Increasing the soap/alcohol concentration in the triethanolamine laurate/ lauryl alcohol system increased emulsion viscosity and emulsion stability but decreased the rate of foam drainage. Foams at both concentrations showed some thinning after two hours but retained their shapes.



Figure 8. Photomicrograph of a tricthanolamine palmitate foam



Figure 9. Photomicrograph of a triethauolamine palmitate/cholesterol foam

824 JOURNAL OF THE SOCIETY OF COSMETIC CHEMISTS

Increasing the concentration of the triethanolamine myristate/ myristyl alcohol complex increased emulsion viscosity, emulsion stability, and foam stiffness. The foams with either soap/alcohol concentration were fairly stable and, except for a slight surface crazing, changed very little in two hours.

Variation in Fatty Acid/Triethanolamine Ratio

Free fatty acids are normally present in solutions of the salts of fatty acids as a result of hydrolysis. These free fatty acids have been reported to form molecular complexes with the salts under certain circumstances. As previously mentioned, sodium stearate and stearic acid have been shown to form such complexes. The triethanolamine salts of the fatty acids, which are salts of weak bases and weak acids, would also hydrolyze to form free fatty acids. Complexes between the free fatty acids and the triethanolamine salts might be formed in the aerosol systems.

It was hoped to obtain evidence for the existence of such complexes in the triethanolamine-fatty acid systems by changing the concentration of free fatty acid. Samples were prepared in which the ratios of fatty acid to triethanolamine were 1:1.5 and 1.5:1. In the first system, there was an excess of the triethanolamine, and in the second system there was an excess of the fatty acid. If the aerosol system containing the higher concentration of fatty acid exhibited the usual properties

	Salt Concentratio	n in Aqueous Phase
	0.025 M	0.100 M
Triethanolamine Laurate/Lauryl Alco	ohol	
Emulsion viscosity	Low	Low to medium
Emulsion stability	$<1 \min$	>5 hr
Foam drainage (60 min)	20	6
Foam stiffness (g)	20	20
Type of discharge	Quiet	Quiet
Density (g/cc)	0.06	0.06
Triethanolamine Myristate/Myristyl	Alcohol	
Emulsion viscosity	Low	High
Emulsion stability	1–5 min	>5 hr
Foam drainage (60 min)	<u>·2</u>	0
Foam stiffness (g)	24	56
Type of discharge	Liquid-noisy	Noisy
Density (g/cc)	0.13	0.07

Effect	of	Variations	in	Soan	Alcohol	Concentrations'

" Soap/alcohol ratio (molar) = 1:1.

associated with complex formation, this would be considered evidence for the formation of the complexes.

The data obtained with lauric, myristic, and palmitic acids in combination with triethanolmine are given in Table XIV. In the triethanolamine laurate system, the addition of excess lauric acid had essentially no effect upon the properties of the system. There also was little effect upon foam stability. Foams from both mixtures wetted paper almost immediately after discharge and started to collapse. These results indicated that molecular complex formaticn was negligible in the triethanolamine laurate systems.

The addition of excess myristic acid to the triethanolamine myristate system had a noticeable effect upon the properties, however. Emulsion viscosity, emulsion stability, and foam stiffness increased, and the rate of drainage decreased. Foam stability was affected similarly. The foam with an excess of triethanolamine started to wet paper within 10 minutes, became very thin, and started to collapse. The foam with an excess of myristic acid showed little change after an hour. This effect of myristic acid upon the properties of the triethanolamine myristate system are typical of complex formation.

In the triethanolamine palmitate system, excess fatty acid resulted in an increase in emulsion stability and in foam stiffness. The rate of drainage was low regardless of the acid base ratio, and the foam stability was high. Foams with both acid base ratios showed little change after two hours. It seems likely that strong molecular complexes between palmitic acid and triethanolamine palmitate were formed, regardless of the acid/base ratio.

				Foam Pro	operties ^{c.d}
	Acid/Base	Emulsion Pro	Drainage	Stiffness	
Acid	Ratio (Mols)	Viscosity	Stability	(60 min)	(g)
Lauric	$1:1\frac{1}{2}^{n}$	Low	<1 min	85	9
	$1:1\frac{1}{2^{b}}$	Low	$<1 \min$	86	10
Myristic	$1:1\frac{1}{2}^{a}$	Low	<1 min	71	21
	11/2:10	Low to medium	>1 hr	5	37
Palmitic	$1:1\frac{1}{2}''$	Low to medium	15-30 min	0	23
	11/2:14	Low to medium	>1 hr	0	42

 Table XIV

 Variation in Fatty Acid/Triethanolamine Ratio

^a 1:1¹/₂ ratio = 0.10 M acid/0.15 M base.

 b 1¹/₂:1 ratio = 0.15 M acid/0.10 M base.

· All discharges are quiet.

^d All densities range from 0.060 to 0.069 g/cc.

DISCUSSION OF RESULTS

The addition of many of the long-chain alcohols to aerosol emulsions based either on sodium lauryl sulfate or the triethanolamine salts of the fatty acids has a pronounced effect upon such properties as emulsion viscosity and stability and foam drainage, stiffness, and stability. These effects are similar to those observed previously in nonaerosol systems and indicate that complex formation occurs between the surfactants and the alcohols in the aerosol systems. The aerosol systems used for the study were relatively simple. In aerosol products, where there may be many more ingredients, the effects of the added alcohols might be modified by the other components present, particularly if they were surface active.

The molecular complexes generally had an observable effect on the properties of both the aerosol emulsions and the resulting foams. Complex formation, therefore, occurred initially at the propellant/water interface in the aerosol emulsions and subsequently influenced the properties of the foams when the emulsion was discharged. Complex formation probably occurred in the bulk phase of the emulsion by solubilization of some of the alcohol molecules in the surfactant micelles as well as at the propellant/water interface.

The wetting properties of the foams on paper correlated well with the drainage rates and, in many cases, with foam persistence. The effect of the complexes in decreasing foam drainage was very marked and was consistent with the results reported on the effect of molecular complexes in nonaerosol foams. The effect of the complexes in decreasing foam drainage is considered to result from an increase in surface viscosity. In the present work, there were many cases in which an obvious decrease in foam drainage and an increase in foam stiffness could be interpreted on the basis of complex formation. In other cases, a decrease in foam drainage occurred, but there was no apparent increase in foam stiffness. It is probable that the foam stiffness measurements are much less sensitive than drainage tests and do not show increases in foam viscosity unless the increase is fairly large.

Cholesterol had little effect upon the properties of sodium lauryl sulfate systems and apparently did not form strong complexes with sodium lauryl sulfate in aerosol systems. Previous work indicated that cholesterol did form complexes with sodium alkyl sulfates in nonaerosol systems.

In triethanolamine palmitate and stearate systems, cholesterol increased foam drainage slightly and decreased foam stiffness. It also increased the bubble size of the foams. It is possible that these effects, which are opposite those of the long-chain alcohols, are due to the formation of liquid complexes with cholesterol. Previous work showed that cholesterol formed liquid complexes with sodium cetyl sulfate, while cetyl alcohol formed solid complexes (3). The addition of cholesterol to the triethanolamine-fatty acid systems may have resulted in the formation of a film which had a lower viscosity than that of the triethanolamine salt-fatty acid complex. This would produce a foam with lower stiffness. The increased bubble size would also account for the slight increase in the drainage. Miles and his co-workers showed that the flow of liquids through foams decreased with a decrease in bubble size in the foam (4). The increased bubble size resulting from the presence of cholesterol might be accounted for by the increased fluidity of the liquid films. Such films would be expected to expand more during discharge of the product and subsequent vaporization of the propellant than more solid films. The greater expansion should produce large bubble sizes.

Another interesting effect that occurred with the addition of many alcohols was the change from a quiet discharge to a noisy, sputtery discharge. There probably are a number of factors involved in this effect. If combinations of alcohols and surfactants form strong, solid complexes at the propellant/water interface in the emulsion, these complexes might resist expansion when the product was discharged and the propellant vaporized into a gas. The fact that triethanolamine palmitate or stearate emulsions also gave noisy discharges without any alcohols present is an indication that fairly strong complexes between the free fatty acid and the triethanolamine soap are formed in these systems.

Another factor may be the droplet size of the dispersed propellant in the aerosol emulsion or possibly the uniformity of the droplet size. Emulsions containing large-size droplets might produce a noisier discharge than those with smaller droplets. There is no direct evidence for this, but a larger droplet size might explain why some of the cholesterol emulsions gave noisy discharges and also why some propellants gave a product with a noisier discharge than other propellants.

SUMMARY AND CONCLUSIONS

The effect of various alcohols upon the properties of aerosol emulsions and foams was studied to determine if the alcohols formed molecular complexes with the surfactants. In many cases, the addition of an alcohol had a marked effect upon the aerosol system. Emulsion viscosity and stability were increased, foam drainage was decreased, and foam stability was increased. Foam stiffness was increased in some systems. On the basis of these results, it was concluded that molecular complexes were formed in aerosol systems. The effect of these complexes upon the properties of the systems was similar in many respects to that previously reported with nonaerosol systems.

The use of molecular complexes is an effective method for varying the properties of aerosol foams. By the proper choice of surfactant, alcohol, and propellant, foams may be obtained which wet immediately after discharge and then collapse or which wet immediately but retain their foam structure. Foams may also be obtained which are quite stable and show no wetting or collapse for extended periods. Aerosol emulsions can be formulated to give an immediate foam discharge or a liquid discharge which subsequently expands into a foam.

The aerosol emulsions were prepared with sodium lauryl sulfate or the triethanolamine salts of lauric, myristic, palmitic, and stearic acid as the surfactants and fluorinated hydrocarbon propellants as the dispersed phase. The effect of alcohols upon the emulsions and foams was studied with lauryl, myristyl, cetyl, stearyl and oleyl alcohols and cholesterol.

The extent to which any alcohol affected the properties of a specific aerosol emulsion or foam depended upon such factors as the type and concentration of the alcohol, the surfactant, and the propellant. The saturated fatty alcohols formed complexes in both sodium lauryl sulfate and triethanolamine-fatty acid systems. Microscopic observation showed that complex formation usually reduced the bubble size of the foams. In some instances, the addition of an alcohol resulted in a product which had a noisy or sputtery discharge. This was attributed to the formation of a solid molecular complex which resisted expansion when the liquefied propellant vaporized during discharge.

Cholesterol had little effect in sodium lauryl sulfate systems but formed fluid complexes in the triethanolamine-fatty acid systems. These complexes expanded easily during discharge, and this increased bubble size and decreased foam stiffness. Oleyl alcohol likewise had little effect in sodium lauryl sulfate systems but appeared to form weak complexes in some of the triethanolamine-fatty acid emulsions.

The type of propellant had a considerable influence on the properties of surfactant/alcohol systems. In general, the most stable emulsions and foams were obtained with Freon-12, Freon-12/Freon-114 (40/60)

or Freon-114. Freon-12/Freon-11 (50/50), Propellant 142b, and Propellant 152a gave less stable foams. The latter two propellants gave foams with the lowest density.

Triethanolamine-fatty acid systems with an excess of fatty acid were investigated to a limited extent. The data indicate that fatty acids also form complexes with the triethanolamine salts.

The results of the present study show that molecular complex formation can vary the properties of aerosol emulsions and foams over a wide range. These data were obtained with simple aerosol systems. The extent to which the present findings can be applied to modify the properties of practical aerosol products remains to be determined. Possible applications include the formulation of more heat stable foams, resulting from a potentially higher film drainage temperature with the foams containing molecular complexes, and the preparation of aerosol emulsion systems with powder suspensions. The increased viscosity of the aerosol emulsion systems with molecular complexes might retard settling and agglomeration of the powder sufficiently so that a practical product could be obtained.

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Book Reviews

ATOMIC ABSORPTION SPECTROSCOPY, by James W. Robinson, Marcel Dekker, Inc., New York, N. Y. 1966. 204 pages, illustrated and indexed. Price \$9.75.

Atomic absorption spectroscopy is a relatively new analytical tool but its importance is growing steadily. Despite the difficulties of interferences by extraneous elements, the rising popularity of this technique is due to its speed and relatively low cost. Atomic abscrption spectroscopy is, in effect, the reverse of emission spectroscopy. The major difficulty is the problem of generating the element (usually metallic) from its compounds before atomic absorption spectroscopy can be carried out. Thus, a large portion of this book is devoted to descriptions of atomizers, i.e., equipment which generates neutral atoms.

This book is concerned primarily with practical problems. Accordingly, the theory of spectroscopy is deemphasized; instead, much space is devoted to equipment and to methods for the determination of about 50 elements. This includes most of the metallic elements which are present in cosmetics either deliberately or as impurities. It is likely that this technique, for which relatively inexpensive commercial equipment is available, will soon receive wider application in the cosmetic and other industries. This book should prove to be a worthwhile introduction to these newer methods and a practical guide for the analyst.—M. M. RIEGER— Warner-Lambert Research Institute.

AGING, edited by William Montagna, Pergamon Press, Oxford, England. 1966. 237 pages, illustrated and indexed. Price \$15.

"Aging" is the report of the May, 1964, "Oregon Symposium"; it contains contributions from some of the foremost authorities in their respective fields. Thus, this reader looked forward eagerly to the task of reviewing this, the sixth volume in Montagna's Advances of the Biology of Skin. Unfortunately, his anticipation was not fulfilled by this book.

Publication more than two years later of the papers delivered at a meeting is a delay which materially reduces interest. In the meantime, several of the papers, especially those presenting new findings, have appeared in various scientific journals; repetition now can be justified only by the attempt to publish the complete proceedings of the symposium.

Aging of the skin and of its appendages is an important social problem for older members of a youthoriented society. It is surprising, therefore, that this book includes only one chapter on attempts to improve or rejuvenate aging skin by clinical methods. On the other hand, about 15 of the 17 chapters of this book deal with comparisons between young and old skin. Unfortunately, the reader is not alerted to the fact that horizontal aging studies-and this book is concerned only with comparisons between skin of different individuals of different ages—are not as definitive as longitudinal studies.

The reader of a scientific book is entitled to expect the author to delineate new problems, point out unusual findings, and if possible give some scientifically sound explanation. Thus, it is disturbing that one author neglected to mention that amino acid analyses of dermal tissue suggest that the total collagen content of elastotic skin is closer to that of skin from premature infants than that of normal (?) adults.

This reviewer is not qualified to judge the histological and morphological observations of aging skin which account for several major chapters in the book. On the other hand, some of the gross observations of the graying of hair appear to be not quite justified. One of the authors states that "graying appears first on the temples.... In a recent examination of eight octogenarians, virtually no hypomelanotic hairs were found in the axillae, presternum and pubis." From personal observation, this reviewer knows that graving in males frequently occurs first in the presternum area. In the same section it is noted that "before the hair becomes completely white, the pigmentation of the individual hairs that together with others look gray, may cover the entire range of color dilution." This statement implies that fibers gradually change color until they finally turn completely white and is, therefore, contrary to observed facts.

Despite the above-voiced objections to the book, this volume is important reading for those of us who are interested in the aging of skin and related phenomena. The book presents some significant new data, on one hand, and, at times, is an excellent and comprehensive review. The reader is, however, advised to analyze and examine observations and statements with considerable care before accepting all the conclusions made by the contributors.-M. M. RIEGER-Warner-Lambert Research Institute.

ENCYCLOPEDIA OF CHEMISTRY, 2nd Edition, edited by G. L. Clark and G. G. Hawley. Reinhold Publishing Corp., New York, N. Y. 1966. 1114 pages, illustrated and indexed. Price \$25.

The reviewing of an encyclopedia i.i not an easy task: Sometimes the value of an encyclopedia lies in the number of entries, and sometimes it is to be found in the completeness of coverage of the subject. Neither of these criteria can be applied to this particular volume. Instead, the original meaning of the Greek word, encyclopedia, i.e., instruction in a broad area of knowledge, describes this volume more precisely. In fact, this encyclopedia is an alphabetically arranged textbook of chemistry and chemical technology. The number of entries is limited, but each of them is detailed enough to provide broad coverage of the subject under scrutiny. This book is not for the novice but requires some background in chemistry. Each entry is carefully explained, and some derivation of equations is given; it serves as an introduction to the subject and at the same time is an excellent review for those who are already familiar with it. What is particularly noteworthy is the fact that each entry is truly a brief and readable discussion of an important subject. Typical is the three-page entry on foam. There are no separate entries for the Gibbs adsorption theorem, the Laplace and Marangoni effects, and antifoams; instead, these points are presented in a logical manner and in a clear and concise style, which makes for pleasurable reading.

This reviewer was impressed by the large list of contributors, many of whom are well-known experts in their respective fields. Although it may be presumptious to recommend reading of an encyclopedia from cover to cover, such an activity would be an excellent refresher course in chemistry. Even occasional browsing through this volume will be rewarding; and finally, this book will serve well as a desk reference. The emphasis of this encyclopedia is not on details (which can be found readily in the various handbooks of chemistry) but on the broad principles and theoretical aspects. Therein lies the main value of this book, which can be recommended to all unequivocally.---M. M. RIEGER-Warner-Lambert Research Institute.

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AUTHOR INDEX TO VOLUME XVII

- Abbott, D. C., see Thomson, J. Anderson, J. V., see Lange, W. E. Bannan, E. A., see Kooistra, J. A.

- Baruffini, A., see Gialdi, F. Baxter, B. H., see Puttnam, N. A.
- Bohac, S., see Root, M. J.

- Bond, E. M., Employment contracts, 93 Borselli, V. F., see Vinson, L. J. Brook, R. J., and Joyner, B. D., Analysis of aerosol propellants, 401
- Brown, M. R. W., Turbidimetric method for the rapid evaluation of antimicrobial agents-Inactivation of preservatives by nonionic agents, 185
- Caccialanza, P., see Gialdi, F. Cahn, M. M. Photosensitivity, 81
- Carrie, C., and Kuhl, M., On the action of detergents on skin with consideration of contact materials, 247
- Carter, R. O., see Kooistra, J. A.
- Conrad, L. I., Maso, H. F., and DeRagon, S. A., Surface modifying effects of lanolin derivatives, 157
- Czetsch Lindenwald, H. V., see El-Khawas, F
- Dansizer, C., see Rebenfeld, L. Davidson, H. R., The use of instrumentation in cosmetic color control, 329
- DeRagon, S. A., see Conrad, L. I. El-Khawas, F., Tawashi, R., and Czetsch Lindenwald, H. V., Water vapor sorption and suction potential of starch grains, 103

- Everett, M. A., see Master, K. J. Fang, V. S., see Lange, W. E. Fitch, G. R., Preparative gas chromatography, 657
- Fleming, J., Rapid method for the determination of hydroxyl values, 625
- Gialdi, F., Baruffini, A., Ponci, R., and Caccialanza, P., Cosmetic uses of a new synthetic antifungal agent, 575
- Godfrey, K. M., Cationic emulsifiers in cosmetics, 17
- Ham, G. V., Practical utilization of the photomicrography of cosmetics, 299
- Hladik, J., see Pokorny, J.
- Hodgson, G., Some principles and difficulties of topical treatment in dermatology, 29
- James, R. J., A new and realistic electronic approach to the evaluation of antiperspirant activity, 749
- Jewel, P. W., Annual report of the President, 67
- Jover, B. D., see Brook, R. J.

- Karjala, S. A., Williamson, J. E., and Karler, A., Studies on the substantivity of collagen-derived polypeptides to human hair, 513
- Karler, A., see Karjala, S. A.
- Kass, G. S., see Sorkin, M.
- Kennon, L., Product stability: Prognostication, placement, parameters-Part I, 135
- Kennon, L., Product stability: Prognostication, placement, parameters-Part II. 313
- Kligman, A. M., Blind man dermatology. 505
- Kooistra, J. A., Bannan, E. A., and Carter, R. O., Use of human subjects for product evaluation: An evaluation of antibacterial soap bars, 343 Kramer, R. A., The Seventeenth Medal
- Award—Sophie Louise Plechner, 71 Kuczera, K., The microscopic appearance of
- cosmetic preparations, 257
- Kuhl, M., see Carrie, C. Lange, W. E., and Anderson, J. C., Cyclic salicylanilides as antibacterial agents, 355
- Lange, W. E., and Fang, V. S., Aqueous topical adhesives, II. Spray-on bandage, 115
- Lee, S., and Puttnam, N. A., Determination of chlorinated phenols in powdered deodorants, 3
- Lee, S., see Puttnam, N. A.
- Lines, R. W., see Wood, W. M.
- Mao, I., see Menkart, J. Maso, H. F., see Conrad, L. I.
- Master, K. J., Sayre, R. M., and Everett, M. A., New evaluation techniques for sunscreens, 581
- Menkart, J., Wolfram, L. J., and Mao, I., Caucasian hair, Negro hair, and wool: Similarities and differences, 769
- Meyer-Rohn, J., The bacterial flora of skin of healthy and skin diseased humans, 287
- Neuwald, F., Rheological studies of new cream bases with the Brookfield Synchro-Lectric viscometer, 213
- Newcomb, E. A., Lanolin allergy?, 149
- Papa, C. M., The action of antiperspirants, 789
- J., and Hladik, J., Chromato-Pokorny. graphic separation of components of cosmetic cremes, 703
- Ponci, R., see Gialdi, F.

- Puttnam, N. A., and Baxter, B. H., IR spectroscopy of aqueous detergent solutions, 391
- Puttnam, N. A., Baxter, B. H., Lee, S., and Stott, P. L., Application of attenuated total reflectance IR spectroscopy to toilet articles and household products, 2. Quantitative analysis, 9
- Puttnam, N. A., see Lee, S.
- Rebenfeld, L., Weigmann, H. D., and Dan-sizer, C., Temperature dependence of the mechanical properties of human hair in relation to structure, 525
- Rogers, A. R., Spectral slit width and other sources of error in uv spectrophotometry, 641 Root, M. J., and Bohac, S., Hygroscopicity
- and hardness of hair spray resins at varying humidities, 595
- Rowell, N. R., Fluorescent antibody techniques in dermatology, 631
- Sanders, P. A., Molecular complex formation in aerosol emulsions and foams, 801
- Sayre, R. M., see Master, K. J.
- Scherin, A., Female sex hormones in products for the treatment of skin, 727
- Schwarz, G. W. G., Possibilities and limitations of shampoo analysis, 737
- Scott, G. V., see Waggoner, W. C. Scott Blair, G. W., The subjective assessments of the consistency of materials in relation to physical measurements, 45
- Shapiro, B., see Sorkin, M.
- Sharples, A., The relation between structure and properties in plastics used in packaging, 415

- Sherman, P., Techniques for assessing the rheological properties of toiletry and cosmetic products, 439
- Sorkin, M., Shapiro, B., and Kass, G. S., The practical evaluation of shampoos, 539
- Steiner, W., New investigation of the mechanism of smell in animals, 713
- Stolar, M. E., Evaluation of certain factors influencing oil deposition on skin after immersion in an oil bath, 607
- Stott, P. L., see Puttnam, N. A.
- Tawashi, R., see El-Khawas, F. Thomas, W. G., Jr., Protection of cosmetic colors by means of U.V. absorbers, 553
- Thomson, J., and Abbott, D. C., Thin-layer chromatographic techniques in residue analysis, 467
- Todd, Lord of Trumpington, The changing face of organic chemistry, 377
- Tronnier, H., Problems of dermatological testing of cosmetics, 275
- Vinson, L. J., and Borselli, V. F., A guinea pig assay of the photosensitizing potential of topical germicides, 123
- Waggoner, W. C., and Scott, G. V., Instrumental method for the determination of hair raspiness, 171
- Weigmann, H. D., see Rebenfeld, L.
- Williamson, J. E., see Karjala, S. A.
- Wolfram, L. J., see Menkart, J. Wood, W. M., and Lines, R. W., Particle size analysis using Coulter Counters, 197
- Zahn, H., Chemical processes during hydrogen-peroxide bleaching of wool and human hair, 687

SUBJECT INDEX TO VOLUME XVII

Adhesives, aqueous topical, 115 Aerosols, analysis, 401 complex formation in emulsions and foams, 801 Allergy, lanolin as cause of, 149 light-induced, 81 Aluminum salts, action on sweat ducts, 797 Amino acid analysis of bleached hair, 693 Analysis of cremes by column chromatography, 703 Antifungal agent, cosmetic use, 575 Antimicrobials, cyclic salicylanilides, 355 evaluation in soap bar, 343 inactivation by nonionic surface-active agents, 185 turbidimetric evaluation, 185 Antiperspirants, action, 789 efficacy, 279 evaluation of activity with aid of electronic approach, 749 Bacterial Flora of skin, 287 Bandage, aerosol, 120 Bleaching of human hair, 687 Cationic emulsifiers, 17 Chromatography, column, of creme components, 703 gas, preparative use, 657 thin-layer, techniques for, 467 Cohesion, measurement, 450 Collagen-derived polypeptides, substantivity to human hair, 513 Colors, cosmetic, protection by means of ultraviolet absorbers, 553 instrumental control, 329 Compressibility, measurement, 459 Cosmetics, antifungal agent in. 575 cationic emulsifiers for, 17 cohesion, 450 consistency, 45 dermatological testing, 275 hormones in, 727 instrumental color control, 329 microscopy, 299, 257 preservation, 185 rheological properties, 439 rigidity, 456 viscosity, 441 Coulter counter, 197 Cremes, separation of components by chromatography, 703 Cysteic acid, in bleached human hair, 693 Cystine, in hair, 769 Deodorants, chlorinated phenols in, 3 Dermatology, 29, 505 use of fluorescent antibody techniques, 631

Detergents, analysis in shampoos, 737 effect on skin, 247 Dyes, certified, stabilization against fading, 553 Employment contracts, 93 Emulsifiers, cationic in cosmetics, 17 lanolin derivatives as, 157 nonionic, inactivation of preservatives by, 185 Emulsions, stability testing, 314 Esters, fatty, deposition on skin, 610 Estrogenic hormones, 728 Fatty alcohol, effect on aerosol foams, 801 Fluorescent antibody techniques in dermatology, 631 Foams, aerosol, physical characteristics, 801 Formaldehyde, action on sweat duct, 795 Gas chromatography, preparative, 657 Gestational hormones, 729 Hair, acid binding, 769 amino acid composition, 769 Caucasian, comparison with Negro hair and wool, 769 chemical processes during bleaching with hydrogen peroxide and per-acids, 687 determination of raspiness, 171 dyeing behavior, 769 effect of bleaching on physical properties 688 mechanical properties, 769 mechanical properties, effect of temperature on, 525 moisture regain, 769 morphology, 769 sorption of peptides, 520 swelling, 769 Hair spray resins, hygroscopicity and hardness, 595 Hormones, use in cosmetics, 727 Hydroxyl values, rapid method for determination, 625 I.F.S.C.C. Fourth Congress, 510 Insects, use in the study of mechanism of smell, 713 Kligman, Albert M., eulogy, 502 Lanolin derivatives, deposition on skin, 610 surface effects, 157 Lanolin, safety, 149 Manuscripts, directions for preparation, 307 Mineral oil, deposition on skin in presence of surface-active agents, 612

- Moisture absorption by hair spray resins, 597
- Molecular complex formation in aerosol emulsions and foams, 801

Obituaries, Amsterdam, H. J., 512 Fowler, P. J., 65 Hibbot, H. W., 685 Holmes, H., 686 Myddleton, W. W., 246 Odor, perception in insects, 713 Oils, factors influencing deposition on skin, 607 Organic chemistry, changes of, 377 Particle size determination using Coulter counter, 197 Penetrometer, 448 Phenols, chlorinated, photosensitizing action, 123 spectrophotometric determination in powdered deodorants, 3 use in soap bars, 348 Photosensitivity, 81 Photosensitizers, drugs as, 86 guinea pig assay, 123 topical germicides as, 88, 123 Phyto-estrogens, 732 Plastics, structure and properties, 415 Plechner, Sophie L., eulogy, 72 Polypeptides, adsorption on human hair, 520 Product stability, kinetics for prediction of, 135measurement, 313 storage conditions, 144 Propellants, aerosol, analysis, 401 Rheological properties, of cosmetics, 213, 439relation to subjective assessment, 45 Rigidity, measurement, 456 Salicylanilides, cyclic, 355 halogenated, photosensitivity of, 123 in soap bar, 348 Sectilometer, 458 Shampoo, analysis, 737 practical evaluation, 539 Skin, bacterial flora, 287 deposition of oil, 607 effect of detergents, 247 treatment, 29 Smell, study of mechanism in animals, 713 Soap, evaluation of antibacterial, 343 Society of Cosmetic Chemists, Great Britain, Annual Meeting, 498 Annual Report, 493 Dinner Dance, 243 Diploma Course, 438, 500 Diploma Examination 1966, 679

Honorary Member, 436 Officers and Council 1965-6, 1 Program, 64, 243, 499, 683 Silver Medal, 390 Soiree, 63 Symposia, 63, 64, 244, 436, 684 Society of Cosmetic Chemists, U.S.A., Advisory Committee, 303 Annual Report, 67 By-laws, 361 California Chapter, 76 I.F.F. Award, 747 Medal Award, 71 Meetings 1966, 92, 170, 312 Midwest Chapter, 76 New England Chapter, 304 New Members, 133, 305, 510, 748 New York Chapter, 78 Twelfth Literature Award, 501 Spectrophotometry, U.V., effect of electrolytes, 651 error due to reflection, 645 slit width, 641 stray light, 647 Spectroscopy, I.R., aqueous detergent solution, 391 attenuated reflectance, quantitative analysis of household products, 9 direct approach compared with attenuated reflectance, 9, 393 household products, 9 Spectroscopy, U.V., chlorinated phenols, 3 sunscreens, 582, 641 Starch, water vapor sorption, 103 Sunscreens, efficacy, 280 evaluation, 581 Suspensions, stability testing, 391 Sweating, thermal use in antiperspirant evaluation, 749 Talcum powder, particle size distribution, 204Ultraviolet light absorbers for protection of cosmetic colors, 553 Viscometers, Brookfield synchoelectric, 213 comparison between, 443 parallel plate viscoelastomer, 452 Viscosity, measurement, 222, 441 Wool, chemical processes during bleaching,

- 687
 - comparison with human hair, 769
BOOK REVIEW INDEX TO VOLUME XVII

- Augustine, R. L., Catalytic Hydrogenation, 374
- Bassett, J., Inorganic Chemistry, 59
- Bell, M., Surface Active Agents, 433 Bennett, H., The Chemical Formulary, Vol-
- ume XII, 131
- Bible, R. H., Interpretation of NMR Spectra, 432
- Bradstreet, R. B., The Kjeldahl Method for Organic Nitrogen, 240
- Browning, E., Toxicity and Metabolism of Industrial Solvents, 571
- Bush, H., Histones and Other Nuclear Proteins, 57
- Campbell, P. N., and Greville, G. D., Essays in Biochemistry, Volume 2, 673
- Carriere, G., Lexicon of Detergents, Cosmetics and Toiletries, 624
- Chapman, D., The Structure of Lipids, 430
- Clark, G. L., and Hawley, G. G., Encyclopedia of Chemistry, 2nd Edition, 832
 Davidson, J. N., The Biochemistry of the
- Nucleic Acids, 235 Ferguson, L. M., Textbook of Organic Chemistry, 677
- Florkin, M., and Stotz, E. H., Comprehen-sive Biochemistry, Volume 6, Lipids and Amino Acids and Related Compounds, 131
- Florkin, M., and Stotz, E. H., Comprehen-sive Biochemistry, Volume 16, Hy-drolytic Reactions Cobamide and Biotin Coenzymes, 572
- Giddings, J. C., Dynamics of Chromatography, Part I: Principles and Theory, 623
- Giddings, J. C., and Keller, R. A., Advances in Chromatography-Volume I, 573
- Harper, N. J., and Simmonds, A. B., Advances in Drug Research, 431
- Hilditch, T. P., and Williams, P. N., The Chemical Constitution of Natural Fats, 239
- Holliday, A. K., and Massey, A. G., Inorganic Chemistry in Non-Aqueous Solvents, 238

- Jarrett, A., Spearman, R. I. C., and Riley, P. A., Dermatology: A Functional Introduction, 669
- Jolles, Z. E., Bromine and Its Compounds, 674
- Katritzky, A. R., Advances in Heterocyclic Chemistry, Volume 3, 57
- Katritzky, A. R., Advances in Heterocyclic Chemistry, Volume 5, 676 Kolthoff, I. M., and Elving, P. J., Treatise
- on Analytical Chemistry, Part I, Volume 5, 671
- Lloyd, L. E., Techniques for Efficient Research, 571
- Lyne, A. G., and Short, B. F., Biology of the Skin and Hair Growth, 373
- Macek, K., and Hais, I. M., Stationary Phase in Paper and Thin-Layer Chro-
- matography, 433 Mathieson, D. W., Interpretation of Or-ganic Spectra, 236 Miles, D. C., and Briston, J. H., Polymer
- Technology, 238
- Montagna, W., Aging. 831
- Morton, R. A., Biochemistry of Quinones, 61
- Noltingk, B. E., The Art of Research, 181 Patai, S., The Chemistry of Alkenes, 675
- Purkyne University Press, De Structura et Functione Stratorum Epidermis S. D. Barrierrae, 670
- Reed, R. I., Applications of Mass Spectrometry to Organic Chemistry, 429
- Reed, R. I., Mass Spectrometry, 670
- Robinson, J. W., Atomic Absorption Spec-troscopy, 831
- Stuttgen, G., Die Normale und Pathologische Physiologie der Haut. 182
- Szymanski, H. A., Interpreted Infrared Spectra, Volume I, 434
- Van Deenan, L. L. M., Progress in the Chem-istry of Fats and Other Lipids, Volume VIII, Part 1: Phospholipids and Biomembranes, 61
- Waddington, T. C., Non-Aqueous Solvent Systems, 58

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