J. Soc. Cosmet. Chem., **25**, 621–638 (November 1974)

Antiperspirants: New Trends in Formulation and Testing Technology

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Presented December 11, 1973, New York City

Synopsis—Aluminum chlorhydroxide has been the most widely used active ingredient in ANTIPERSPIRANT FORMULATIONS. Recently, new chemicals, such as basic aluminum bromide, and combinations of aluminum, zirconium, and other metal salts have been introduced. In addition, new product forms are constantly being developed with different performance and cosmetic characteristics. The properties of the new active ingredients and the new formulations are discussed with respect to formulation VERSATILITY, COSMETIC ELEGANCE and EFFICACY. General methods used to evaluate staining potential, and deodorant and antiperspirant efficacy of these products are reviewed. A normal activity method for determining antiperspirant efficacy is compared with a method based on a thermally controlled environment, and the results obtained with these two techniques are discussed.

Introduction

Antiperspirant formulations based upon metal salts such as aluminum, zinc, or magnesium chlorides, sulfates, acetates, or sulfocarbolates as the active ingredients have been known for a long time (1, 2). The most important antiperspirant chemical used is basic aluminum chlorhydroxide (ACH) which is safer, less corrosive, and readily formulated into a variety of products (3).

The deodorant and antiperspirant market has changed dramatically over the years. Until *ca.* 1960, the most important product forms were lotions, creams, sticks, or powders, representing a 100 million dollar per year business. In 1960, aerosol deodorants, primarily alcoholic solutions containing an antimicrobial agent, came into the market, and by 1966, doubled the size of the business (4). Early attempts to develop antiperspirants in an aerosol form ran into trouble because of the acidic nature of the active ingredients. Most problems involved packaging incompatibility, valve clogging, and perfume stability. They mostly were solved by the mid-sixties (5), resulting in the introduction of a number of different types of aerosol antiperspirants.

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The early aerosol antiperspirants introduced were the so-called powder-in-oil type formulations containing 3-4% of active ingredients suspended in an oil base. There were also solution-type formulations, some in glass aerosols because of corrosion problems.

In 1971, a cosmetically different antiperspirant product was developed, an "aerosol powder"; another version, a so-called "hybrid" appeared in the marketplace one year later. The hybrid has a somewhat higher active level than the regular powder-in-oil and powder antiperspirants and lies between these products in cosmetic elegance.

The most important performance attributes of an antiperspirant are its anhydrotic and deodorant effects. In addition, the formulation must possess aesthetic and cosmetic qualities. One frequently observed drawback of these products has been their tendency to cause fabric staining. It is the purpose of this paper to discuss recent trends in the formulation of aerosol antiperspirants and some of the testing methods used to evaluate performance characteristics.

Aerosol Antiperspirant Formulations

Most aerosol antiperspirant formulations contain the following components: the active ingredients, usually aluminum chlorhydroxide or similar salts, a liquid system which serves either to solubilize or to suspend the active ingredient or is part of a water-in-oil emulsion, miscellaneous additives such as talc, perfume, suspending agents, and propellants. Suspension systems represent the most commercially important examples. Some products based on solution systems have been marketed, but have not been too successful. While providing somewhat greater antiperspirant efficacy, solutions are cosmetically less pleasing.

Many combinations of raw materials are available for the formulation of aerosol antiperspirants and their selection must be carefully considered, since the surface chemistry of the system can affect sedimentation and dispersion characteristics of the formula. In addition, formulations must provide maximum antiperspirant and deodorant effectiveness, maximum safety, cosmetic elegance, and minimum staining.

Active Ingredients

Aluminum chloride has been recognized for many years as an excellent antiperspirant. However, because of its low pH, it will cause fabric damage and skin irritation (6). This had led to the development of various basic aluminum compounds which are less acidic than the parent product. The most frequently used of these derivatives is basic aluminum chlorhydroxide. Other metal salts that have been formulated into antiperspirants are shown in Table I. In addition to these compounds which are considered to interfere

Table I

Active Ingredients for Antiperspirant Formulations

Aluminum chlorhydrate
Aluminum chloride
Basic aluminum bromide
Basic aluminum hydroxychloride–zirconyl hydroxy oxychloride
Aluminum hydroxychloride–zirconyl hydroxy oxychloride–glycine complex
Basic aluminum nitrate
Basic aluminum bromide–zirconyl hydroxy oxybromide
Magnesium aluminum zirconium gluconate chloride
Basic aluminum iodide

with the sweat duct (7), other materials that have been reported include formaldehyde (8) and compounds such as anticholinergic scopolamine derivatives (9).

Amongst the metal salts which have gained some commercial importance are basic aluminum bromide (BAB) and certain zirconium complexes. Zirconium oxychloride ($ZrOCl_2$) and zirconyl hydroxychloride [ZrO(OH)Cl] have good antiperspirant activity, but the aqueous pH of these compounds is very low. The use of zirconium compounds as antiperspirant actives has required raising the pH without causing precipitation. Commercially, this has been achieved by buffering the zirconium salts with basic aluminum salts (10–14). While this can cause considerable gelling (14), it was found that the addition of certain amino acids such as glycine (11, 14) can control the gelation problem. There are also patents covering aluminum zirconium systems using different buffering systems in antiperspirant formulations (15, 16). A number of experimental salts have also been investigated, including basic aluminum nitrates, iodides, and mixed metal systems.

Suspending Oils

Some of the cosmetic oils used in powder-in-oil formulations are listed in Table II. They include isopropyl and propylene glycol esters of various long-

Table II

Cosmetic Oils Used in Aerosol Powder-in-oil Antiperspirants

Isopropyl myristate
Isopropyl palmitate
Mixed isopropyl esters of various fatty acids
Propylene glycol dicaprate
Propylene glycol 400 monolaurate
Propylene glycol dipelargonate
Triethyl citrate
Dibutyl phthalate
Organosilicones

chain fatty acids, homopolymers of polypropylene oxide, dibutyl phthalate, Tween 60, and certain organosilicones. Properties of these oils include low odor, low viscosity, and good stability.

Suspending Agents

Suspending agents are frequently used in aerosol powder-in-oil systems where the active material is dispersed rather than dissolved. The suspending agents reduce the rate of settling of the dispersed materials.

An effective suspending agent commonly used is Bentone 34,* an organic derivative of hydrous magnesium aluminum silicate. Bentone requires batch heating and extensive homogenization. Homogenization causes the formation of hydrogen bonds between silica sites, resulting in lattice formation.

Another frequently used suspending agent is Cab-O-Sil,† a fire-dry fumed silica. Chains are formed *via* hydrogen bonds, and with shear mixing, an effective lattice structure can be created. Cab-O-Sil is quite sensitive to the presence and the ionic nature of other materials in the formulation. Syloid 244‡ is another high-porosity micron-sized silica that has also been used.

The type of suspending agent used in antiperspirant powder formulations can be readily identified from X-ray diffraction determinations.

Propellants

The propellants used in aerosol antiperspirants serve several important functions: they deliver the product, serve as diluents and/or solvents, and assist in product "drying." General factors such as bounce and coldness are directly related. The colder sprays generally produce less bounce, and this is a function of the boiling points of the various propellants used. Several trade-offs are frequently necessary in optimizing an antiperspirant propellant system.

Powder-in-Oil Formulations

Powder-in-oil formulations represent the most important category of products presently on the market. This category contains three different types: regular powder-in-oil formulations, the so-called aerosol powders, and hybrid formulations. There are very noticeable differences from the cosmetic and aesthetic point of view between these three product forms. A typical powder-in-oil formulation is the following:

*NL Industries, 111 Broadway, New York, N.Y.
†Cabot, 125 High St., Boston, Mass.
‡W. R. Grace & Co., Baltimore, Md.

Ingredient	Per Cent
Aluminum chlorhydroxide	3.5
Isopropyl myristate	6.0
Cab-O-Sil	0.3
Perfume	0.2
Propellant	90.0

Aerosol powder formulations contain about the same level of ingredients as powder-in-oils versions, but in addition also contain 1% of talc. Also, the oil level is considerably lower. A typical formula is shown below:

Ingredient	Per Cent
Aluminum chlorhydroxide	3.5
Talc	1.0
Suspending oil	1.5
Cab-O-Sil	0.3
Perfume	0.2
Propellant	93.5

The hybrid-type formulations are similar to powder-in-oil formulations except for a higher active ingredient level (5% *vs.* 3.5%).

The differences between these three formulations are summarized in Table III. As can be seen, the ratio of active ingredient levels to the amount of oil used ranges from 0.6 for the powder-in-oil formula, to a 1.1 ratio for the hybrid product, and a ratio of 2.3 for the powder.

Table III

Formulation Differences between Aerosol Powder-in-oil, Powder, and Hybrid Antiperspirants

Formulation Type	% Active	% Oil	% Talc	Active: Oil Ratio
Powder-in-oil	3.5	6.0	...	0.6
Hybrid	5.0	4.5	...	1.1
Powder	3.5	1.5	1.0	2.3

Three hybrid-type products have appeared in the marketplace. While fitting into the above general formulation system, there have been significant differences in the active ingredients and the suspending oils that were used, as shown in Table IV. All contain approximately 5% of active ingredient, but the actives differ considerably. The first contains the traditional basic aluminum chlorhydroxide powder, the second, a buffered aluminum chlorhydroxide/zirconyl hydroxy oxychloride complex, while the third uses a buffered aluminum chlorhydroxide/zirconyl hydroxy oxychloride glycine complex. The suspending oils also differ considerably.

The hybrid formulations provide somewhat higher antiperspirant efficacy than regular powder-in-oil formulations, probably because of the higher ac-

Table IV

Formulation Differences in Active Ingredients and Suspending Oils of Three Hybrid Antiperspirants

Product	Active Ingredients	Suspending Oil
1	5% aluminum chlorhydroxide	Mixed silicones
2	5% basic aluminum hydroxychloride–zirconyl hydroxy oxychloride	Mixed silicones/triethyl citrate
3	5% aluminum hydroxychloride–zirconyl hydroxychloride–glycine complex	Dibutyl phthalate

tive level. All three are cosmetically very acceptable. Two of the products have eliminated the staining problem often associated with aerosol antiperspirants, while one still causes considerable staining.

Evaluation Procedures

Key performance criteria for which aerosol antiperspirants are tested include the following: antiperspirant efficacy, deodorant efficacy, staining potential, and cosmetic acceptability.

Antiperspirant Efficacy Evaluation

Antiperspirant efficacy may be measured utilizing both *in vitro* and *in vivo* methods. Qualitative procedures fashioned after the work of Minor (18), who used starch-iodine indicators, involved development of so-called sweat pore patterns. Various dyes and indicators have been used in conjunction with papers, pastes, films, and lacquers (19). Zahejsky and Rovensky (20) described the use of a contact indicator spot test which was based on a color reaction between pyrogallol and ferric hydroxide in the presence of water from sweat. These procedures have value in locating and studying the activity of sweat glands.

Some *in vitro* methods are based upon the fact that aluminum salts are astringents and are capable of denaturing proteins (21), and upon the observation that permeability to sodium and iodide ions increases after treatment with aluminum salts (22).

Quantitative measures of the reduction in perspiration are usually made using gravimetric or humidity sensing techniques with human volunteers. Some attempts have been made to use animals, such as cats, mice, or rats for sweat testing purposes, but the sweat glands in these species are confined to the foot pads, and even though they have eccrine glands, they differ histologically from those of man (22–24). Recently, Lansdown evaluated the use of rat food pads as a model for examining antiperspirants (25).

In human volunteers, gravimetric measurement of the total amount of perspiration secreted may be accomplished by the use of weighed pads or by the use of dessicant cups (26). Other types of measurement utilized resistance hygrometry, collection coils, phosphorus pentoxide cells, and electrolytic cells (27–30).

Factors Affecting Sweating Rate

Fredell and Read (31, 32) observed that there are differences in the amount of sweat produced from right to left axilla, but that the ratio of these differences is fairly consistent. They recommended that a ratio between treated and control axillae be used in judging effectiveness. Segar (27) demonstrated that sweating is a cyclical process and is not proportional to the number of total glands.

Uttley (33) recently reviewed some of the factors influencing sweating which must be considered in a test procedure. These include: relative humidity, equilibrium of sweating rate, emotional or mental stimulation, position of the body during sweating, skin area being tested, skin temperature, conditioning to environment, sex differences, and metabolic rate.

Based upon our studies, we agree with the observations of earlier workers (19, 32, 34) that a gravimetric method employing absorbent pads is an adequate procedure. Reller (19) observed that absorbent pads are more acceptable physiologically than cups.

Methods of Evaluation

We have tested two basic variations of the gravimetric method, the major difference being in the procedure used for stimulation and collection of perspiration. The first method, termed "normal activity method," utilizes normal environment conditions. The second method, termed "controlled environment method," employs thermally controlled environmental conditions. In both methods, a ratio of sweat produced by the left and right axilla is determined in a series of controlled collections. The effect of antiperspirant materials on the perspiration rate of each individual is determined by comparing the post-treatment ratio to the subjects' average control ratio. For each individual, the per cent change in sweat rate is calculated as:

$$\% \text{ change in sweat rate} = 100 \left(1 - \frac{\text{post-treatment ratio}}{\text{average control ratio}}\right)$$

These data are statistically treated by applying the Student t distribution to establish 85% confidence limits on the mean per cent change in sweat ratio.

Normal Activity Method

In the normal activity method, a group of panelists are recruited who remain together as a panel for at least 12 months. Prior to the start of

using the panel for test purposes, their left-to-right axillary sweating ratio is determined 12 times over a 4-week period. The average result from these determinations is used as the control ratio for each panelist for the life of the panel.

Before the start of any test, there is a 5-day period in which the panelists use no underarm products except a placebo soap. Following this are three consecutive days of antiperspirant testing. There are 15–20 people in a mixed-sex panel. Test material is applied only to the left axilla, the right axilla serving as the control. For roll-on formulations, 1 g of product is applied with a soft brush. For aerosols, a 1-sec spray from a distance of 6 in. is applied uniformly to the axillary vault. Sample application is made by a technician, using a metronome to time the spray. After 5 min, to permit evaporation of any volatile materials, a preweighed moisture collection pad is applied. These pads, measuring 2 in. x 2 in., are fashioned from sanitary napkins by stripping away several layers of cellulose filler in order to get a better axillary fit. Pads are held in place by strips of hypoallergenic tape. The exterior surface of the pad itself is covered with Saran Wrap in order to retain absorbed moisture.

The panelists are then free to pursue normal activities for 4 hours. At the end of the 4-hour period, the absorbent pads are removed and placed in tared plastic ointment jars and weight of perspiration is determined by difference.

Controlled Environment Method

In the controlled environment method, approximately the same number of panelists are used. They are asked to abstain from the use of all antiperspirant materials at least one week prior to enrollment through the completion of the test. Controlled sweat collections are made on Monday and Tuesday of the test week. Post-treatment sweat collections are made on Wednesday, Thursday, and Friday. Test materials are assigned in such a way that samples are applied to the right axilla of half the panelists and to the left axilla of the remaining panelists. Axillae are rinsed with clear, warm water and dried just before each application. For aerosol products, the axillae are sprayed from the distance of 6 in. for 2 sec. Materials are applied immediately following the controlled sweat collection on Tuesday, 1 hour prior to sweat collection on Wednesday, Thursday, and Friday.

Sweating is induced by having the subject sit in a room maintained at 100° ±2°F and at a relative humidity of 35%. During the first 40 min of the sweat stimulation period, the panelists hold unweighed pads of Webril,* a nonwoven cotton, in their axillae. This preliminary warmup period is followed by two successive 20-min collection periods during which panelists hold pre-weighed Webril pads in the axillae. Panelists are allowed to drink ice wa-

*Kendall Co., Walpole, Mass.

ter as desired throughout the collection period. At the end of each collection period, the pads are removed and placed in tared bottles for reweighing. Antiperspirant activity is calculated as described previously.

Deodorant Efficacy Evaluation

Deodorant effectiveness is a recognized attribute of many metal salts (35–37). Efficacy is evaluated by comparison of the effect of one treatment in one axilla *versus* a second treatment in the other axilla of the individuals in panel groups (34, 38). Similar comparative methods may employ a crossover procedure. Product comparisons using a split axilla treatment without crossover in a single group suffer in reliability because odor intensity of the axillae of an individual varies. However, when the test sites in a panel are randomized by a crossover procedure, effects due to inherent differences in the axillae are minimized.

Axillary odor may be judged by the panel participants themselves, by trained judges, or both. Length of deodorant effect is usually determined at various intervals during the test, or at cessation to treatment. Evaluation may be made by estimating the degree of odor of both the axillae and the undergarment at the side of contact. Odor judgments obtained are usually based upon arbitrary numerical scales (38–40). Because these are subjective evaluations, several investigators have attempted to eliminate sources of error through the use of osmometers (41).

Utilizing a crossover method in which odor evaluations were made by trained judges, a clear deodorant effect was shown for a powder-in-oil formulation and an aerosol powder formulation, both containing 3.5% aluminum chlorhydroxide. An odor scale of 1 (little or no odor) to 5 (strong or disagreeable odor) was used. The results are shown in Table V. The data from the test and control periods were subjected to Student's t test and were highly significant.

A recent advance in evaluating deodorant efficacy is that developed by Dravnieks and coworkers (42, 43). Their method is based on the development of chromatographic patterns of axillary odors.

Staining Potential

Soon after the introduction of aerosol antiperspirants, it became apparent that fabric staining in the axillary area was a major problem. Initially, formulations were tested for staining propensity by applying the product directly to the fabric. It was found that this was not a satisfactory procedure since it did not reflect actual use conditions and a comparative procedure was developed.

In this procedure, a panel is used made up of 10 men and 10 women. Subjects are required to abstain from the use of all antiperspirants and deodorants or other products applied to the axillary area, and are required to use

Table V

In Vivo Deodorant Evaluation of Two Powder-In-Oil Antiperspirants
(Summary of Individual Mean Odor Scores[a])

Subject	Control Period		Test Period	
Number	Dry Powder	Powder-In-Oil	Dry Powder	Powder-In-Oil
1	4.5	4.7	2.0	1.9
2	4.0	4.3	2.8	2.4
3	4.9	4.7	1.3	1.5
4	4.1	4.1	1.8	1.8
5	5.0	5.0	1.9	1.8
6	4.8	4.8	2.8	2.4
8	2.9	3.0	2.0	2.2
9	4.0	4.3	1.5	1.5
10	3.6	3.8	1.9	1.7
11	4.4	4.6	2.9	2.8
12	4.0	3.7	2.9	2.7
14	4.4	3.9	2.3	1.5
15	4.2	4.5	2.3	2.5
16	4.5	4.5	3.6	3.2
17	3.1	3.2	2.2	2.1
18	4.3	4.3	2.1	1.9
19	3.5	3.5	2.5	2.6
20	4.6	3.6	2.4	2.0
21	4.2	4.4	3.1	2.9
22	4.3	3.2	2.0	1.9
Mean	4.16	4.11	2.32	2.16
Standard Deviation	0.5518	0.5862	0.5677	0.5008
Standard Error	0.1233	0.1310	0.1269	0.1119

[a] A five-point scoring system was used, 1 being weak and 5 being strong and disagreeable odor.

only Ivory soap* during the period of the test. T-shirts are supplied to the subjects at the beginning of the test period. A standard 3.5% powder-in-oil isopropyl myristate formulation is used for reference purposes. Subjects are evaluated for stain propensity in each axilla using the following schedule. On day 1, a 3-sec spray of the product is applied by a technician using a metronome for timing. The new T-shirt is then worn. Four hours later, the spraying procedure is repeated. Two hours later, the T-shirts are collected and laundered. The same routine is then followed for 2 more days. At the end of the third day, the axillary areas of each T-shirt are evaluated for intensity of stain using a photovolt reflectometer equipped with tristimulus filters. Readings are taken using each of the three filters and the staining value is calculated from the formula:

*Procter & Gamble Co., Cincinnati, Ohio.

$$\frac{\text{amber-blue}}{\text{green}} \times 10$$

A typical value for the control formula is 0.7. A minimally visible stain yields a value of 0.3. A value of 1 or higher indicates a product which stains fabric considerably.

When evaluating test products, the procedure is the same as during the standardization except that the test product is substituted for the control. Application to the left and right axilla is randomized. Calculations are normalized to the control value of 0.7 so that results are reproducible. Some of the results obtained using this comparative method are shown in Table VI. There were considerable variations in stain potential of products, depending on product form and the type of suspending oils used.

Table VI

Staining Propensity of Aerosol Antiperspirant Formulations
(Three-Day Test Period)

	Formulation (Active/Oil)	Formulation Type	Staining Value
5%	aluminum hydroxychloride–zirconyl hydroxychloride–glycine complex/dibutyl phthalate	Hybrid	1.0
5%	aluminum chlorhydrate/mixed silicones	Hybrid	0.26
3.5%	aluminum chlorhydrate/mixed silicones	Aerosol powder	0.17
3.5%	aluminum chlorhydrate/isopropyl myristate	Powder in-oil	0.70

Cosmetic Acceptability

In considering the overall properties of an antiperspirant, cosmetic and perfume qualities are a key consideration. The dispensing oils, when not runny, can create a pleasant tactile feel. The propellant, when optimized, can reduce coldness of the spray when applied to the skin. Overall, a product is cosmetically better when it goes on dry, without caking, and is gentle to both skin and clothing. The best technique for evaluating these properties and their overall effect on product acceptability is by full scale market research procedures, though some laboratory procedures for individual properties are also used.

Safety

The criteria for evaluating safety of antiperspirant formulations include a consideration of the effect of the product on the skin and its aerosolized properties. Routine safety evaluation of a new formulation comprises determination of acute oral and dermal toxicity, primary eye and skin irritation, acute and subacute inhalation studies in rabbits and monkeys, and sensitization in guinea pigs and in humans.

Aerosol antiperspirants have received considerable publicity as a result of potential inhalation hazards that may be associated with low-level, long-term exposure to propellant gases and particulate aerosols in general. Recently, Drew (44) reported that persistent lung granulomas were observed in hamsters exposed to an aerosolized liquid system based on a propylene glycol complex of ACH at 50 mg/m^3 for 6 hours a day for 10, 20, and 30 days.

Antiperspirants are classified as drugs and are subject to the Food and Drug Administration's OTC review procedure. A request to provide safety and efficacy data for review by the OTC panel has been made (45) and hearings started in the early part of 1974.

Antiperspirant Test Results

From the earlier description of the two methods for evaluating antiperspirant efficacy used in our laboratory, namely, the normal activity method and the controlled environment method, it seemed plausible to hypothesize that since methodologies were basically similar, the results too would be of the same order of magnitude, or at least provide similar directional guidelines.

Normal Activity Method

The result obtained when testing a standard powder-in-oil formulation on four different occasions spread over a period of a year and a half are shown in Table VII. The results are 3-day averages and are expressed with a confidence limit of 95%.

Table VII

Antiperspirant Efficacy of a 3.5% Aluminum Chlorhydrate Powder-in-oil Formulation (Normal Activity Method)

Test Number	Mean % Sweat Reduction
1	20.6 ± 1.8
2	21.6 ± 2.6
3	21.0 ± 2.4
4	27.8 ± 2.7

Table VIII

Antiperspirant Efficacy of Different Actives
in Powder-in-oil Formulations (Normal Activity Method)

	Per Cent Active	Mean % Sweat Reduction
Aluminum chlorhydroxide	3.5	22.8 ± 2.7
Basic aluminum bromide	3.5	23.6 ± 4.0
Basic aluminum nitrate	3.5	23.2 ± 3.9
Basic aluminum bromide–zirconyl hydroxy oxybromide	3.5	25.5 ± 4.0
Basic aluminum hydroxy chloride–zirconyl hydroxy oxychloride	3.5	25.1 ± 3.1
Magnesium aluminum zirconium gluconate chloride	3.5	22.4 ± 4.0
Magnesium aluminum zirconium gluconate bromide	3.5	22.7 ± 2.9

In a second series, different active materials were examined in a powder-in-oil formulation. The compounds tested and the efficacy results are listed in Table VIII. It was found that regardless of the active material used, the results all fell within a very narrow range.

The results obtained comparing the sweat reduction efficacy of different types of antiperspirant formulations including an aerosol powder-in-oil, an aerosol powder, a hybrid formula, a roll-on, and a 20% aluminum chlorhydroxide solution are shown in Table IX. In the solution forms (roll-on or 20% aqueous) between 35 and 40% sweat reduction was observed. The two powder versions reduced perspiration by 22%, while the hybrid formula gave a slightly higher result, namely 27%, a difference which is real from the statistical point of view, and probably due to the higher level of active ingredient (5% *vs.* 3.5% ACH) of the hybrid version.

Table IX

Antiperspirant Efficacy of Different
Formulation Types (Normal Activity Method)

Product Category	Mean % Sweat Reduction
3.5% powder-in-oil	22.8 ± 2.7
3.5% dry powder	22.1 ± 3.3
20% ACH solution	39.9 ± 4.0
Hybrid formulation	27.0 ± 4.1
Roll-on formulation	37.5 ± 2.6
Placebo	2.8 ± 2.8

Table X

Sweat Reduction as a Function of Time for 3.5% Aluminum Chlorhydroxide Powder-in-oil Formulation (Normal Activity Method)

Time (hrs)	Mean % Sweat Reduction		
	Test 1	Test 2	Test 3
3	22.4 ± 3.8	21.1 ± 2.9	24.6 ± 2.4
6	20.0 ± 4.3	20.3 ± 4.0	24.0 ± 2.7
9	18.9 ± 3.6	18.0 ± 3.7	19.2 ± 3.0
12	14.0 ± 4.1	12.4 ± 2.6	17.1 ± 2.5

Table XI

Effect of Spraying Time (Concentration) on Sweat Reduction (Normal Activity Method)

Duration of Spray (sec)	Mean % Sweat Reduction	
	Formula A	Formula B
0.5	8.5 ± 2.5	10.7 ± 2.5
1	22.8 ± 2.9	25.1 ± 4.5
2	28.0 ± 3.2	30.3 ± 3.7
4	34.9 ± 3.8	35.7 ± 3.7

Since the various powder-in-oil formulations gave results in a very narrow efficacy range, regardless of active ingredient, it was decided to test the sensitivity of the method. A series of sweat reduction determinations were carried out as a function of time. Four collections were made following one application, at 3, 6, 9, and 12 hours on 3 successive days. The data obtained in three separate tests with a 3.5% powder-in-oil formulation are shown in Table X. Each test, conducted independently, produced essentially the same per cent decreases in sweat reduction over time. The method was able to distinguish quite successfully and distinctly between relatively small variations in sweat reduction.

In a second series of tests in which different spraying times were used, data were similarly obtained after one application in 3 consecutive days. The data are in Table XI. As the spraying time increased, the per cent sweat reduction also increased.

The effect of the aluminum chlorhydroxide particle size on efficacy was also evaluated. Three different grades were used: Chlorhydrol Ultrafine,* one grade with a smaller particle size (56% retained on 10-μ screen), and one with a larger particle size (96% retained on 10-μ screen). No significant differences were found between the grades, as shown in Table XII.

*Reheis Chemical Co., Phoenix, Ariz.

Table XII

Effect of Particle Size
on Sweat Reduction (Normal Activity Method)

	Mean % Sweat Reduction
Regular (UltraFine ACH)	22.8 ± 2.9
Smaller particle size than UltraFine ACH (96% retained on 10-μ screen)	24.9 ± 3.3
Larger particle size than UltraFine ACH (56% retained on 10-μ screen)	23.9 ± 3.1

Table XIII

Antiperspirant Efficacy of a 3.5% Aluminum Chlorhydrate
Powder-in-oil Formulation (Controlled Environment Method)

Test	Mean % Sweat Reduction
1	12.6 ± 3.6
2	33.6 ± 5.2
3	16.0 ± 4.1
4	22.3 ± 9.8

Table XIV

Antiperspirant Efficacy of a 3.5% Basic Aluminum Bromide
Powder in-oil Formulation (Controlled Environment Method)

Test	Mean % Sweat Reduction
1	35.0 ± 4.2
2	6.7 ± 6.0
3	21.2 ± 4.2

Controlled Environment Method

The same 3.5% powder-in-oil formulation used in the normal method was evaluated under the controlled environmental conditions. The results obtained are summarized in Table XIII. Results ranged from 12.6% to 33.6% sweat reduction. In another series, a 3.5% basic aluminum bromide powder-in-oil formulation was tested. The results are shown in Table XIV and again showed little reproducibility.

Discussion of Results

In general, it was found that in our hands when using the controlled environment method, there was a wide spread of results when the same formu-

lations and the same active ingredients were tested. Because of these difficulties, we have fewer results to report, and are continuing to investigate this technique.

On the other hand, the normal activity method gave reproducible results under a variety of conditions. It did not show any real differences between the effectiveness of different active ingredients under the same formulation conditions. That did not mean that the normal activity method was insensitive, since it very effectively distinguished between powder-in-oil formulations and roll-ons, and clearly indicated variations of antiperspirant efficacy as a function of time or concentration.

The controlled environment method differs from the normal activity method in several ways. The controlled method employs a 2-sec spray, whereas the normal method utilizes a 1-sec spray. Treatment and collection times are different; in particular, the time periods for collection between the normal and controlled methods differ in length (4 hours *vs.* 40 min) and conditioning i.e., the controlled method utilizes a conditioning period before the pads are applied for collection purposes.

Another reason for the differences observed between the normal and the controlled testing techniques is possibly due to the conditions used in the controlled test. Going into a 100°F sweat chamber at 35% humidity can be a challenging stimulus to the body. Consequently, not only is perspiration elicited as a function of temperature, but also due to the challenge to the central nervous system (19)

Conclusions

We have concluded that the normal activity method is a more useful tool for providing guidance on the relative efficacy of different formulations and different active ingredients. The method also closely approximates what the consumer experiences in actual use and we have been able to correlate efficacy data with observations reported in large-scale, carefully designed consumer tests.

Certain additional considerations must be kept in mind when considering gravimetric procedures for the determination of antiperspirant efficacy. Bakiewicz (46) has shown that when thermal stimulation is used to induce perspiration, results can vary with body position. Results can also be influenced by drinking cold liquids, variations in the relative humidity of the room, or even by selecting panelists who are "high sweaters" or "low sweaters." For example, Tronnier and Rentschler (47) reported that the same product under controlled environmental conditions gave a 20% sweat reduction when applied to a low sweater, while there was a 50% reduction with a group of people classified as high sweaters.

To summarize our views on antiperspirant test methodology: regardless of the methods used by us or reported in the literature, most of our results for

sweat reduction efficacies for aerosol powder-in-oil and hybrid formulas fall between 20% to 30%. These figures can be restated by saying that a subject was sweating with a 70% to 80% efficiency rather than his normal 100% efficiency. Thus, even if laboratory procedures know how to measure these differences, it is debatable whether the consumer can distinguish between them. While not negating the use of these gravimetric methods to provide data to use as a guide to optimize formulations, or to compare new active ingredients, or to evaluate interaction of materials, care must be taken when these numbers are used for promotional purposes. Small differences, even though statistically significant, should not be magnified out of proportion. It is important that management understands and appreciates the differences in testing methods, and recognizes the limitations and specialized meaning of the data derived from these techniques.

(Received April 2, 1974)

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