Patented Dec. 10, 1946

2,412,535

UNITED STATES PATENT OFFICE

2,412,535

ANTIPERSPIRANT PREPARATION

Earl Leroy Richardson, Bound Brook, and Kenneth Lyman Russell, Nutley, N. J., assignors to Colgate-Palmolive-Peet Company, Jersey City, N. J., a corporation of Delaware

No Drawing. Application November 29, 1944, Serial No. 565,810

6 Claims. (Cl. 167—90)

1

The present invention relates to improved preparations for retarding or inhibiting perspiration.

Certain chemical compounds have the property of retarding, eliminating or inhibiting the flow of perspiration, and many of them are now widely employed in preparations for that purpose. Of these compounds, the most commonly used are aluminum salts, such as aluminum chloride, aluminum sulphate, and the like, but other compounds, such as zinc, iron, tin and bismuth salts, have also been proposed for the same purpose. Such compounds are termed anti-perspirant compounds or agents.

An undesirable consequence of the use of anti-perspirant compounds to stop the flow of perspiration has been found to be the corrosive effect of the composition on clothing. When a preparation containing these agents is applied to that area of the skin where retarding of the perspiration is desired, a portion of the preparation may come into contact with and adhere to the clothing of the user. If the garment is ironed, pressed or otherwise heat-treated before the anti-perspirant agent is removed by laundering or cleaning, the corrosive effect of the composition is immediately manifested by a reduction in the strength of that portion of the garment with which it has come into contact, sometimes even resulting in holes, that is, a complete 100% deterioration of the garment in specific areas. Long continued contact of the anti-perspirant agent with a garment, even at ordinary temperatures, may cause deterioration or reduction in strength of the garment. The corrosive action of perspiration-retarding compounds is particularly noticeable on fabrics containing cellulosic materials, such as cotton, rayon, and other synthetic fibers; however, it is not limited thereto, and may be observed on other types of fabrics.

Many methods have been proposed to reduce this undesirable effect of the perspiration-retarding agents on fabrics, including the incorporation in the preparation containing the anti-perspirant agent of such addition agents as urea and other amides, and oxides, hydroxides and carbonates of aluminum, magnesium and zinc, and other compounds. Some of these addition agents do reduce or neutralize the corrosive effect of the anti-perspirant compound, but some of them also reduce the amount of anti-perspirant agent which has been incorporated in the preparation for the purpose of retarding perspiration. No compound has yet been proposed which not only reduces the corrosive effect of the anti-perspirant agent on fabrics, but also is itself an anti-perspirant agent.

2

It is an object of this invention, therefore, to provide preparations which inhibit or retard perspiration and which do not have a corrosive effect on fabrics.

It is another object of this invention to provide preparations which possess the full perspiration-retarding effect of the agents incorporated therein for that purpose, and which do not exhibit undesirable corrosive effect on fabrics.

Other objects will be apparent from the disclosures herein.

The objects of this invention are achieved by providing preparations suitable for use as perspiration-inhibiting or retarding compositions, containing aluminum salts now recognized as possessing that property, and also containing certain salts of acids of phosphorus which have the effect of reducing or eliminating the corrosive effect of the perspiration-retarding agents upon fabrics. These preparations may contain, as agents for inhibiting, retarding or eliminating perspiration in localized areas of the skin to which they are applied, water-soluble astringent salts of aluminum. The aluminum salts found most suitable for this purpose are aluminum sulphate and aluminum chloride, but other aluminum salts recognized by the art will serve the same purpose.

The salts which have the effect of inhibiting or preventing the corrosive action of the anti-perspirant agents on fabrics are neutralized salts of acids of phosphorus, which are stable in solution, as illustrated by tertiary orthophosphates. Metaphosphates are not suitable since they are not stable in solution but gradually hydrolyze to form primary orthophosphates; and the primary and secondary orthophosphates, the so-called acid salts, are not suitable to achieve the purposes desired.

The inhibiting salt may be selected to serve also as additional anti-perspirant agent in preparations in which it is used. In such preparations, the cation of the inhibiting salt should be aluminum ion or the cation of some other perspiration-inhibiting agent. A highly desirable preparation may contain aluminum sulphate or chloride and aluminum orthophosphate or aluminum pyrophosphate. In the making of these preparations or compositions, the aluminum phosphates are added as such.

If an increase in anti-perspirant agent is not desired, the inhibiting compounds may be produced in situ. If it is desired to produce these

compounds in situ, that is, admixed with the anti-perspirant agent, a suitable soluble salt of an acid of phosphorus is added to a solution containing the anti-perspirant agent. For example, tetrasodium pyrophosphate or trisodium orthophosphate may be added to a solution of aluminum sulphate, which results in the formation of aluminum pyro- or orthophosphate. Likewise, the corrosive effect of the anti-perspirant composition may be inhibited by using tricalcium or trimagnesium orthophosphate.

These novel compositions may be made as emulsions, solutions, creams, salves, and the like, and may contain any other ingredients which do not effect a chemical change in the perspiration-inhibiting agent or in the fabric-protecting compounds and which are not irritating or otherwise unsuited for such preparations. That is, the novel preparations may contain common cosmetic ingredients, coloring bodies, perfumes, and the like.

The usual methods of compounding these preparations, such as are suitable for the preparation of emulsions, solutions, extracts, salves, and the like, may be employed.

The proportion of the anti-perspirant agent to the total composition may be widely varied and is influenced by the type of preparation, the frequency of use, and other like factors, but is generally a matter of choice. As has been disclosed in the prior art, any proportion within the range of 10% to 30% of hydrated aluminum sulphate makes a satisfactory preparation. In general, the same proportions of aluminum chloride have been found suitable.

The proportion of phosphate salt to the anti-perspirant agent is dictated by the requirements or uses of the preparation. In those cases where garments which have come into contact with the preparation are heat-pressed, or otherwise subjected to high temperatures, without first being laundered, a greater proportion of the corrosion-inhibiting compound is required than otherwise, a proportion of two to three mols of aluminum orthophosphate to one mol of aluminum sulphate being necessary for complete or substantially complete protection from corrosion. It will be understood that even in those instances the temperature of the iron or pressing device has a direct relationship to the inhibiting power of the phosphate salt. For example, when the temperature of the iron is no more than 250° to 275° F., two mols of aluminum orthophosphate to one mol of aluminum sulphate will afford substantially complete protection from corrosion. However, another factor to be considered is the length of time during which the area containing the anti-perspirant agent is subjected to the ironing process. For the average time of ironing or pressing at 250° to 275° F., i. e., five to ten seconds, the proportion specified of two mols of aluminum orthophosphate to one mol of aluminum sulphate is satisfactory. Should the garment be such that a higher temperature and/or a longer period of pressing is required, the proportion should be higher.

In view of the general method of laundering and ironing garments, in which the cleaning process removes the anti-perspirant agent or substantially all of it, a smaller proportion of aluminum orthophosphate or pyrophosphate to the anti-perspirant agent is satisfactory. Preparations to be used under conditions of careful laundering may contain one mol of the phosphate salt to one mol of the inhibiting agent, or even less.

It is appreciated that the cosmetic formulator cannot determine all the uses to which his preparations are to be directed. However, the foregoing instructions will enable the manufacturer to select the proportion most suitable to his product and, in cases of doubt, to include a safe proportion of the corrosion-inhibiting compound.

It appears that aluminum phosphate in solution is itself an anti-perspirant agent, and that, when added as such (that is, not formed by the reaction of the anti-perspirant compound with a suitable soluble phosphate), it increases the potential anti-perspirant action of the preparation.

The maximum proportion of the protective aluminum phosphate that may be used is dependent upon the type of preparation to be made; there is, in fact, no maximum within practical limits. If a clear anti-perspirant solution is desired, the maximum quantity of the corrosion-inhibiting aluminum phosphate which may be incorporated therein, that is, in the aluminum sulphate or aluminum chloride solution, is the greatest quantity of phosphate which may be dissolved in the solution. (Aluminum orthophosphate is soluble only to a negligible degree in water, but it readily dissolves to a considerable degree in a solution of aluminum sulphate.)

In creams, emulsions and other non-transparent preparations, the quantity of aluminum phosphate may exceed that which will dissolve, particularly when the excess phosphate has no undesirable influence on the stability, appearance, or other characteristics of the preparation.

When the inhibiting compound is formed in situ, as by the addition of sodium or potassium orthophosphate or pyrophosphate to a solution of aluminum sulphate, the effect, if any, of the by-product sodium or potassium sulphate on the preparation should be considered. If this by-product salt is undesirable for any reason in the preparation, the inhibiting compound should not be formed in situ.

Thus, it will be seen that the limits of the proportions of the aluminum phosphate corrosion-inhibiting compound may vary over a wide range, and may be any desirable proportion that will reduce or eliminate the corrosive action of the anti-perspirant agent as a minimum and will dissolve or not undesirably affect the preparation as a maximum.

In order to determine the corrosive effect, if any, of the novel preparations on fabrics, certain solutions and preparations were compounded, applied to fabrics, and the effect thereof upon the tensile strength of the fabric determined.

In testing anti-perspirant creams, 2½ grams of the preparation were applied evenly along a one-inch wide band, in the direction of the filling threads, in the center of a desized cotton muslin strip 24 inches long (in the direction of the filling threads) and 6 inches wide (in the direction of the warp threads). The treated fabric was incubated at about 80° F. and 85–90% relative humidity for twenty-four hours. The entire strip was then pressed for five seconds with the cream side down against the pad of a G. E. flat bed ironer. The temperature of the ironer, which had been calibrated, was 293–305° F. The ends of the strip were reversed, and the fabric was again pressed for five seconds. The test fabric was then returned to the humidity chamber and reconditioned for three hours, after

which time it was broken in the warp direction in a tensile strength testing machine by the 4" x 6" grab method, as outlined by A. S. T. M. (American Society for Testing Materials), Designation D39–39. As a control, a similar piece of the same cotton fabric (with no cream applied) was treated in the same way along with the test strip. The difference between the average tensile strength of the control and test pieces was the loss (or gain) in tensile strength due to the effect of the cream on the fabric.

In the case of anti-perspirant solutions, the muslin strip was folded along its width, and the folded edge was immersed in the anti-perspirant solution until there was a one-inch band of solution along the center of the fabric. The excess solution was gently squeezed out, and the fabric was allowed to hang, to dry partially, for five minutes. It was immediately pressed under the same conditions as outlined for creams.

The following examples are formulae of preparations which serve to illustrate the invention, and are not limiting in any sense thereon.

A suitable preparation embodying the principles of this invention may contain the following ingredients and percentages:

Example I

Part I:	Parts
Acid stabilized glycerol monostearate	16
Spermaceti wax	5
Part II:	
$Al_2(SO_4)_3.18H_2O$	19
$AlPO_4$	7
Water	53

The cream given in Example I caused a 32% reduction of the tensile strength of cotton fabric when subjected to the test previously described.

Using no inhibitor, that is, substituting water for the aluminum phosphate, the same test gave an 84% loss in tensile strength. The oil phase (part I) can be varied to suit the consistency desired, as can the water phase (part II), but the ingredients of part II are the important agents in controlling the anti-perspirant effectiveness and the corrosiveness of the preparation. Accordingly, the use of 10 parts of aluminum phosphate with a subsequent reduction of water gives an anti-perspirant solution which shows no corrosive effect upon cotton fabrics. In fact, the test (see Example II) showed a slight increase in tensile strength of cotton fabric. This solution and other anti-perspirant solutions and their effect on cotton fabric are noted herewith:

Example II

	Parts
$Al_2(SO_4)_3.18H_2O$	19
$AlPO_4$	10
H_2O	45

Tensile strength gain of 6.3%.

Example III

	Parts
$Al_2(SO_4)_3.18H_2O$	16
$AlPO_4$	4
H_2O	55

Tensile strength loss of 2.5%.

Example IV

	Parts
$Al_2(SO_4)_3.18H_2O$	18
$AlPO_4$	6
H_2O	51

Tensile strength loss of 1.3%.

Example V

	Parts
$Al_2(SO_4)_3.18H_2O$	25
$AlPO_4$	3.25
H_2O	45.75

Tensile strength loss of 54%.

Example VI

	Parts
A 34% solution of $AlCl_3$	22.4
H_2O	52.6

Tensile strength loss of 72.6%.

Example VII

	Parts
A 34% solution of $AlCl_3$	22.4
$AlPO_4$	14.0
H_2O	38.6

Tensile strength loss of 34%.

Example VIII

	Parts
$Al_2(SO_4)_3.18H_2O$	25
$Na_3PO_4.12H_2O$	10
H_2O	40

Tensile strength loss of 1.7%.

Example IX

	Parts
$Al_2(SO_4)_3.18H_2O$	25
$Ca_3(PO_4)_2$	4
H_2O	46

Tensile strength loss of 22%.

Example X

	Parts
$Al_2(SO_4)_3.18H_2O$	25
$Mg_3(PO_4)_2.5H_2O$	5.5
H_2O	44.5

Tensile strength gain of 5.2%.

The corrosive effect of the preparations given in the foregoing examples was determined according to the tests described hereinbefore. It is unlikely that any garment which might come into contact with these preparations during use would be subjected to such drastic conditions as those represented by these tests. Under less drastic conditions, particularly where fabrics are washed or otherwise cleansed after contact with the anti-perspirant preparation and prior to pressing or other heat treatment, preparations such as illustrated by Examples I, V, VII and IX would have a much less corrosive effect thereon.

It will be understood by chemists skilled in the art that a solution made by dissolving aluminum sulphate or aluminum chloride in water, and then dissolving aluminum phosphate therein, probably does not contain these compounds as such, but contains aluminum, sulphate (or chloride) and phosphate ions, and possibly complex ions. Also, when sodium phosphate is dissolved in a solution of aluminum sulphate, the solution contains aluminum, sulphate, phosphate and sodium ions. A similar condition obtains by the addition of the other salts which have been referred to hereinbefore as suitable for formation of the aluminum phosphate in situ.

Therefore, in view of the difficulty of determining the nature of the various substances in the solution, whether added as such or presumably formed therein, the solution will be designated as a solution of the aluminum sulphate or aluminum chloride, or other astringent aluminum salt, and the aluminum phosphate, whether added as aluminum phosphate or formed therein by the addition of the salt, such as sodium, calcium,

magnesium, etc., phosphate, referred to hereinbefore.

We claim:

1. A perspirant inhibiting or retarding composition comprising a water-containing vehicle, an aluminum salt selected from the group consisting of aluminum sulphate and aluminum chloride, and a corrosion inhibiting compound selected from the group consisting of aluminum orthophosphate and aluminum pyrophosphate, the mol ratio of the phosphate to the salt of the first group being at least one to one.

2. A perspiration inhibiting or retarding composition comprising a water-containing vehicle, an aluminum salt selected from the group consisting of aluminum sulphate and aluminum chloride in amount equal to from 10% to 30% of the composition, and a corrosion inhibiting compound selected from the group consisting of aluminum orthophosphate and aluminum pyrophosphate, the mol ratio of the phosphate to the salt of the first group being at least one to one.

3. A perspiration inhibiting cosmetic cream containing water, 10% to 30% of a substance selected from the group consisting of aluminum sulphate and aluminum chloride, an oil phase,

and an aluminum salt from the group consisting of aluminum orthophosphate and aluminum pyrophosphate, the mol ratio of the aluminum phosphate to the aluminum salt of the first group being at least one to one.

4. A perspiration inhibiting preparation comprising aluminum sulphate and aluminum orthophosphate, the aluminum sulphate comprising at least 10% of the total preparation and the mol ratio of aluminum orthophosphate to aluminum sulphate being at least one to one.

5. A perspiration inhibiting preparation comprising aluminum sulphate and aluminum pyrophosphate, the aluminum sulphate comprising at least 10% of the total preparation and the mol ratio of aluminum pyrophosphate to aluminum sulphate being at least one to one.

6. A perspiration inhibiting preparation comprising aluminum chloride and aluminum orthophosphate, the aluminum chloride comprising at least 10% of the total preparation and the mol ratio of aluminum orthophosphate to aluminum chloride being at least one to one.

EARL LEROY RICHARDSON.
KENNETH LYMAN RUSSELL.