United States Patent Office 3,257,449 Patented June 21, 1966

3,257,449

SURFACE ACTIVE AMINE SALTS

Gregoire Kalopissis, 64 Rue Vauvenargue, and Andre Viout, 302 Rue des Pyrenees, both of Paris, France

No Drawing. Filed Apr. 30, 1965, Ser. No. 452,358
8 Claims. (Cl. 260—501)

This application relates to the permanent waving of living hair, more in particular to processes and compositions for treating the hair during the waving process.

This application is a continuation-in-part of prior application Serial No. 150,612, filed October 24, 1961, now abandoned. This application was a division of application Serial No. 798,805, filed March 12, 1959, now patent 3,093,146, claiming the priority of French application filed March 14, 1958.

Permanent waving of living hair is generally obtained in a known manner by two successive operations viz. a first operation in which the hair is impregnated with a reducing solution so as to insure a more or less complete breakdown of the disulphide bridges of the keratin, while imparting to the hair a sufficient plasticity to lend itself to the curled form which is given thereto on hair curlers, and a second operation during which the hairdresser impregnates the hair with a neutralizing solution, most often of an oxidizing character, intended to give again to the hair in the new form thereof the mechanical properties which it has lost during the first operation, said properties resulting most often from the rebuilding of the aforementioned disulphide bridges.

However, permanent waving of the hair so effected is only a preliminary step in hair-dressing art. The hairdresser's final purpose is an effect to realize by a hair disposition a style of hair-dressing adapted to the up-to-date taste and to the treated hair; such disposition is so-called a setting in the art. During said setting, the hairdresser arranges wet hair, previously curled by the permanent waving, in locks which he rolls at his will and dries when the whole of the operation is completed.

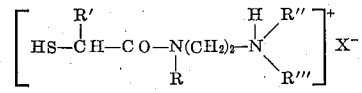
Various attempts have been made to improve the lasting of such setting (and consequently the hair-dressing duration) by pretreating the hair with different natural or synthetic resin compositions, or surface active compounds, the application being effected by using a lotion, a rinsing or a spraying, for example in aerosol form. However, such products do not insure a durable result, since the action thereof on hair is only superficial.

The present invention aims to simplify and facilitate the hairdresser's work for such hair setting which constitutes the essential part of his art, owing to the fact that it makes unnecessary the previous permanent waving proper of the hair and that it makes it possible to accomplish the hair setting in a single operation while giving to the hair a better lasting which extends the duration of the hair-dressing in a noticeable manner.

This invention has for a primary object a hair setting process eliminating any previous permanent waving, characterized by the fact that the hair is impregnated with an aqueous solution of a compound having a reducing effect analogous to that of mercaptans, so as to impart to the hair the desired plasticity, and having moreover the characteristics of a cation-active detergent which cleans the hair and comprises a fatty chain susceptible of adhering to the cleaned hair while giving the same a brightness and a suppleness which facilitate the hair setting, that such hair setting is performed after rinsing of the hair and that the hair is air dried so as to permit an oxidation which gives again to the set hair its natural elasticity.

The present invention has for another object to provide a cation active addition salt of an amine comprising a thiol group reducing the keratin disulphide bridges to put into practice the aforementioned hair setting process, said

salt being characterized by the fact that it belongs to the general formula:



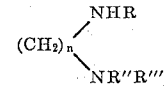
in which R' is a hydrogen atom or a lower alkyl radical comprising not more than 6 carbon atoms, R'' is a lower alkyl radical, any either of substituents R, R''' being selected from hydrogen and a lower alkyl radical and the other being a water repelling fatty radical having from 8 to 24 carbon atoms, and X being an acid anion harmless to the live hair and to the scalp.

The compound according to this invention may be used advantageously in the form of a salt obtained by reacting this compound with an inorganic or organic acid.

Further, one object of the present invention is to provide a process for making a cation-active compound of the aforementioned Formula I, characterized by the fact that it consists in bringing into reaction equimolecular amount of a mercaptan having the general formula:

(II) $HS—CHR'—COOR_1$, in which R_1 is a lower alkyl radical and of an alkylene diamine having the general formula:

(III)



R, R', R'' and R''' having the significations recited for the Formula I.

It may be expected that the compounds of Formula I, owing to their chemical constitution, resulting from this reaction have a moderate reducing activity on the hair keratin. On the contrary, said reducing activity, which would not be sufficient for the permanent waving, is appropriate especially for the operation of so-called hair setting.

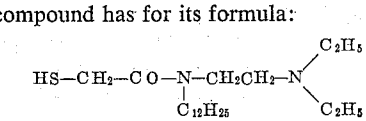
The reduced bridges —S—S—, in a relatively low number by the application of these reducing surface-active products, may be rebuilt by the sole action of the oxygen of air during the conventional operations of drying in a draught, under a helmet, without using an oxidizing solution as in the case of a permanent waving.

When one or more compounds of Formula I are used according to the present invention to effect a hair setting, their foaming cationic detergent properties allow them to be applied as a shampoo as it is applied in a conventional manner previously to the purely manual operation of hair-setting; so as to avoid, in particular, when first applied any excessive dilution by water after which the hair is rinsed and the hair-setting and drying operations are effected according to the usual practice.

In the following examples, given in an illustrative and by no means limitative way, there is described the preparation of some compounds of the aforementioned Formula I:

Example I.—Mercapto-N-lauryl-N-(2-diethylaminoethyl) acetamide

Such compound has for its formula:



Preparation.—There was prepared a hydro-alcoholic solution containing 1 mol of N-lauryl, N'-diethylethylene diamine and 1 mol +10% of ethyl thioglycolate; this solution was heated under reflux during 10 hours under a stream of N_2; after vacuum concentration, there was obtained a residue containing 62% of active product.

The efficiency of the amide formation may be increased by reacting under the same conditions as those mentioned

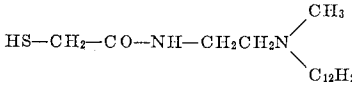
above new amounts of ethyl thioglycolate, once or twice, taking into consideration the amount of non reacted amine.

It is thus possible to obtain finally the desired amide in the form of a waxy body having a 80% purity. The efficiency of the condensation amounts to 85% of the amine used. The corresponding salt of lactic acid is obtained from this raw product, because purification is rather difficult.

The product may be purified by using its insoluble cadmium salt (by reacting cadmium acetate) which can be separated and decomposed later by hydrogen sulphide. The practically pure product is thus obtained in the form of an aqueous solution.

Example II.—Mercapto-N(2-methyl, 2-lauryl, aminoethyl) acetamide

This compound has the following formula:

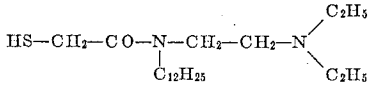


There was added 1 mol (+ a 10% excess) of ethyl thioglycolate in an alcoholic solution to 1 mol of N-lauryl-N-methyl-ethylene-diamine. This solution was heated under reflux during 4 hours and concentrated under vacuum. The resulting residue contained 85% of amide. After a period of 48 hours, it set partly. There were obtained crystals melting at 30° C.–32° C. The yield of the condensation was 75% based on the amine used.

This product having a 85% purity was directly treated with lactic acid. There was obtained a gel, which was dissolved in water at 30% to 35% concentration. This solution is used as a shampoo.

Example III.—Alpha-mercapto-N-lauryl-N-(2-diethylaminoethyl)-acetamide

This composition has the following formula:



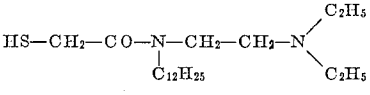
A dilute alcohol solution containing 1 mol/gr. of N-lauryl-N'-diethyl ethylene diamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen. On concentration under vacuum, a residue containing 62% of active product is obtained.

The salt of formic acid is obtained starting with this crude product, because purification is rather difficult.

The action of the formic acid gives rise to a gel, which is dissolved in water to a concentration of 15% in order to obtain an excellent shampoo.

Example IV.—Alpha-mercapto-N-lauryl-N-(2-diethylaminoethyl)-acetamide

This composition has the following formula:



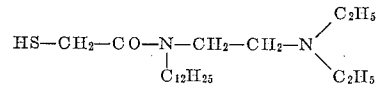
A dilute alcohol solution containing 1 mol/gr. of N-lauryl-N'-diethyl-ethylene-diamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen, and concentrated under vacuum. The resulting residue contained 62% of the amide.

The corresponding citric acid salt was obtained starting with this crude product, because purification is rather difficult.

The action of the citric acid forms a gel which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example V.—Alpha-mercapto-N-lauryl-N(2-diethylaminoethyl)acetamide

This composition has the following formula:



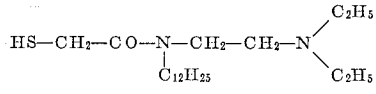
A dilute alcohol solution containing 1 mol/gr. of N-lauryl-N'-diethyl-ethylene-diamine and 1 mol/gr. (+ a 10% excess) of ethylthioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen, and concentrated under vacuum. The resulting residue contained 62% of the amide.

The corresponding hydrochloric acid salt was obtained starting with this crude product, because purification is rather difficult.

The action of the hydrochloric acid forms a gel which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example VI.—Alpha-mercapto-N-lauryl-N(2-diethylaminoethyl)-acetamide

This composition has the following formula:



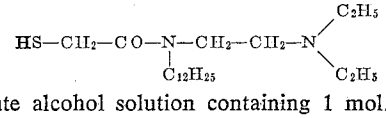
A dilute alcohol solution containing 1 mol./gr. of N-lauryl-N'-diethyl-ethylene-diamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen, and concentrated under vacuum. The resulting residue contained 62% of the amide.

The corresponding acetic acid salt was obtained starting with this crude product, because purification is rather difficult.

The action of the acetic acid forms a gel which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example VII.—Alpha-mercapto-N-lauyl-N-(2-diethylaminoethyl)-acetamide

This composition has the following formula:



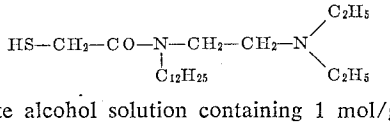
A dilute alcohol solution containing 1 mol/gr. of N-lauryl-N'-diethyl-ethylene-diamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen, and concentrated under vacuum. The resulting residue contained 62% of the amide.

The corresponding tartaric acid salt was obtained starting with this crude product, because purification is rather difficult.

The action of the tartaric acid forms a gel which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example VIII.—Alpha-mercapto-N-lauryl-N-(2-diethylaminoethyl)-acetamide

This composition has the following formula:



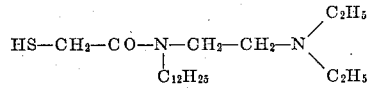
A dilute alcohol solution containing 1 mol/gr. of N-lauryl-N'-diethyl-ethylene-diamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen, and concentrated under vacuum. The resulting residue contained 62% of the amide.

The corresponding phosphoric acid salt was obtained starting with this crude product, because purification is rather difficult.

The action of the phosphoric acid forms a gel which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example IX.—Alpha-mercapto-N-lauryl-N-(2-diethylaminoethyl)-acetamide

This composition has the following formula:



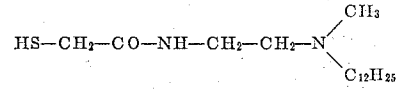
A dilute alcohol solution containing 1 mol/gr. of N-lauryl-N'-diethyl-ethylene-diamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate was prepared. This solution was heated under reflux for 10 hours under nitrogen, and concentrated under vacuum. The resulting residue contained 62% of the amide.

The corresponding hydrobromic acid salt was obtained starting with this crude product, because purification is rather difficult.

The action of the hydrobromic acid forms a gel which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example X.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)-acetamide

This composition has the following formula:



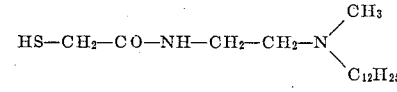
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methyl-ethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting at 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with formic acid.

A gel is obtained, which is dissolved in water to a contration of 20–25%. This solution is employed as a shampoo.

Example XI.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)-acetamide

This composition has the following formula:



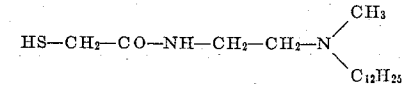
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methyl-ethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting at 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with citric acid.

A gel is obtained, which is dissolved in water to a concentration of 20–25%. This solution is employed as a shampoo.

Example XII.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)acetamide

This composition has the following formula:



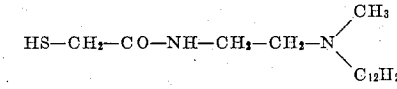
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methyl-ethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with hydrochloric acid.

A gel is obtained, which is dissolved in water to a concentration of 20–25%. This solution is employed as a shampoo.

Example XIII.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)acetamide

This composition has the following formula:



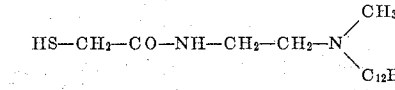
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methyl-ethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting at 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with acetic acid.

A gel is obtained, which is dissolved in water to a concentration of 20–25%. This solution is employed as a shampoo.

Example XIV.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)acetamide

This composition has the following formula:



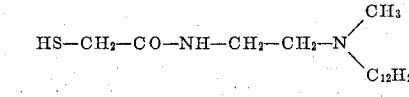
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methyl-ethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting at 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with tartaric acid.

A gel is obtained, which is dissolved in water to a concentration of 20–25%. This solution is employed as a shampoo.

Example XV.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)acetamide

This composition has the following formula:



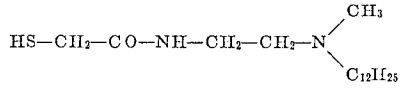
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methyl-ethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting at 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with phosphoric acid.

A gel is obtained, which is dissolved in water to a concentration of 20–25%. This solution is employed as a shampoo.

Example XVI.—Alpha-mercapto-N-(2-methyl-2-laurylaminoethyl)acetamide

This composition has the following formula:



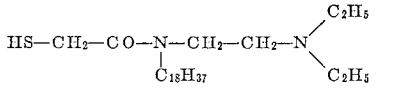
1 mol (+ a 10% excess) of ethyl thioglycolate in alcoholic solution is added to 1 mol of N-lauryl-N-methylethylenediamine. This solution is heated under reflux for 4 hours, and concentrated under vacuum. The residue which is obtained contains 85% of the amide. After 48 hours, it is partially congealed in a mass. Crystals melting at 30–32° are obtained. The yield of the condensation is 71% based on the quantity of the amide used.

This product, which has a purity of 85%, is treated directly with hydrobromic acid.

A gel is obtained, which is dissolved in water to a concentration of 20–25%. This solution is employed as a shampoo.

Example XVII.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

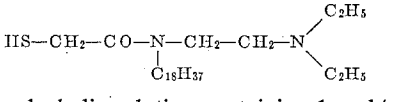
The corresponding lactic acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The section of the lactic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XVIII.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity

of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

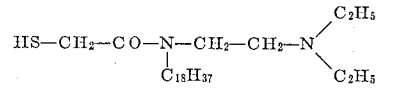
The corresponding formic acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the formic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XIX.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

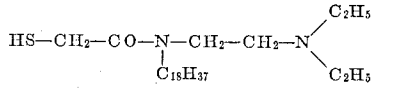
The corresponding citric acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the citric acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XX.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

The corresponding hydrochloric acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

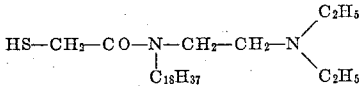
The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate),

which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the hydrochloric acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXI.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

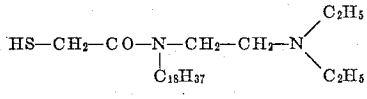
The corresponding acetic acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the acetic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXII.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

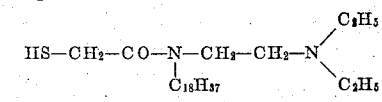
The corresponding tartaric acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the tartaric acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXIII.—Alpha-mercapto-N-stearyl-N-(2-diethylaminoethyl)acetamide

This composition corresponds to the following formula:



A dilute alcoholic solution containing 1 mol/gr. of N-stearyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen, and concentrated to obtain a residue containing 71% of the active product.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The yield of the condensation is of the order of 85% based on the amount of the amine employed as a starting material.

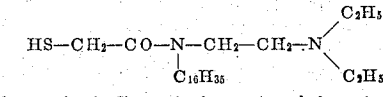
The corresponding hydrobromic acid salt of the crude product is consequently obtained, since the purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the hydrobromic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXIV.—Alpha-mercapto-N-cetyl-N-(2-diethylaminoethyl)-acetamide

This composition corresponds to the formula:



A dilute alcoholic solution containing 1 mol/gr. of N-cetyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen. On concentration, a residue containing 71% of the active product is obtained.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The condensation yield is of the order of 85% based on the amount of the amine used as a starting material.

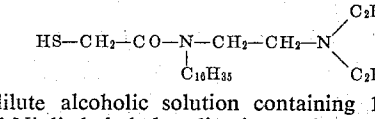
The corresponding lactic acid salt of the crude product is obtained, since purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the lactic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXV.—Alpha-mercapto-N-cetyl-N-(2-diethylaminoethyl)-acetamide

This composition corresponds to the formula:



A dilute alcoholic solution containing 1 mol/gr. of N-cetyl-N'-diethylethylenediamine and 1 mol/gr. (+ a

10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen. On concentration, a residue containing 71% of the active product is obtained.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The condensation yield is of the order of 85% based on the amount of the amine used as a starting material.

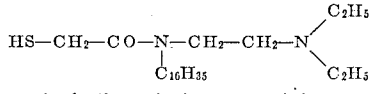
The corresponding formic acid salt of the crude product is obtained, since purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the formic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXVI.—Alpha-mercapto-N-cetyl-N-(2-diethylaminoethyl)-acetamide

This composition corresponds to the formula:



A dilute alcoholic solution containing 1 mol/gr. of N-cetyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen. On concentration, a residue containing 71% of the active product is obtained.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The condensation yield is of the order of 85% based on the amount of the amine used as a starting material.

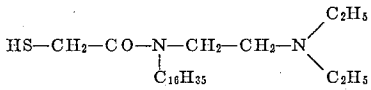
The corresponding hydrochloric acid salt of the crude product is obtained, since purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the hydrochloric acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXVII.—Alpha-mercapto-N-cetyl-N-(2-diethylaminoethyl)-acetamide

This composition corresponds to the formula:



A dilute alcoholic solution containing 1 mol/gr. of N-cetyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen. On concentration, a residue containing 71% of the active product is obtained.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The condensation yield is of the order of 85% based on the amount of the amine used as a starting material.

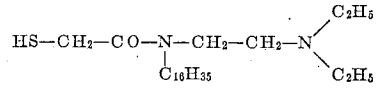
The corresponding acetic acid salt of the crude product is obtained, since purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the acetic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXVIII.—Alpha-mercapto-N-cetyl-N-(2-diethylaminoethyl)-acetamide

This composition corresponds to the formula:



A dilute alcoholic solution containing 1 mol/gr. of N-cetyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen. On concentration, a residue containing 71% of the active product is obtained.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate, This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The condensation yield is of the order of 85% based on the amount of the amine used as a starting material.

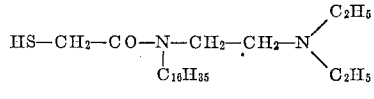
The corresponding tartaric acid salt of the crude product is obtained, since purification of the amide is rather difficult.

The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate), which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the tartaric acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

Example XXIX.—Alpha-mercapto-N-cetyl-N-(2-diethylaminoethyl)-acetamide

This composition corresponds to the formula:



A dilute alcoholic solution containing 1 mol/gr. of N-cetyl-N'-diethylethylenediamine and 1 mol/gr. (+ a 10% excess) of ethyl thioglycolate is prepared. This solution is heated for 10 hours under nitrogen. On concentration, a residue containing 71% of the active product is obtained.

The yield of the amide formation is purified by reacting the product obtained, under the same conditions set out above, with new amounts of ethyl thioglycolate. This is done one or two times, taking into account the quantity of the unreacted amine. The amide is obtained in the form of a waxy mass, having a purity of 80%. The condensation yield is of the order of 85% based on the amount of the amine used as a starting material.

The corresponding hydrobromic acid salt of the crude product is obtained, since purification of the amide is rather difficult.

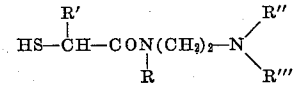
The product can be purified through the medium of its insoluble cadmium salt (by adding cadmium acetate),

which can be isolated. This can be decomposed afterwards by hydrogen sulfide. The practically pure product is obtained in the form of an aqueous solution.

The action of the hydrobromic acid on the product produces a gel, which, when dissolved in water to a concentration of 15%, gives an excellent shampoo.

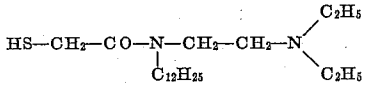
What is claimed is:

1. A surface active product which is the lactic acid addition salt of a compound having the formula

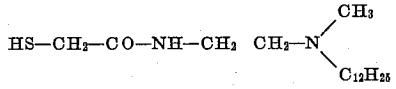


wherein R′ is a member of the group consisting of hydrogen and lower alkyl having not more than 6 carbon atoms and R″ is lower alkyl and one of the substituents R and R‴ is selected from the group consisting of hydrogen and lower alkyl, the other being water repelling fatty acid residue having from 8 to 24 carbon atoms.

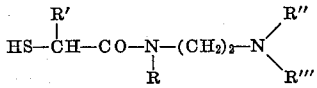
2. A surface active product which is the lactic acid addition salt of the compound having the formula



3. A surface active product which is the lactic acid addition salt of the compound having the formula

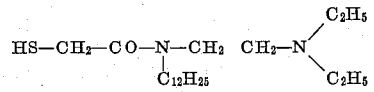


4. A surface active product which is the acid addition salt of a compound having the formula



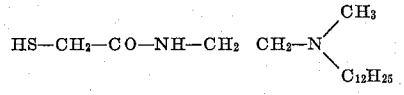
wherein R′ is a member of the group consisting of hydrogen and lower alkyl having not more than 6 carbon atoms and R″ is lower alkyl and one of the substituents R and R‴ is selected from the group consisting of hydrogen and lower alkyl, the other being water repelling fatty acid residue having from 8 to 24 carbon atoms the said acid having an anion harmless to the scalp.

5. A surface active product which is the acid addition salt of the compound having the formula



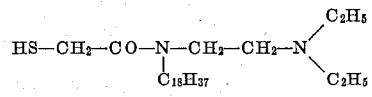
the said acid having an anion harmless to the scalp.

6. A surface active product which is the acid addition salt of the compound having the formula



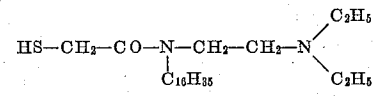
the said acid having an anion harmless to the scalp.

7. A surface active product which is the acid addition salt of the compound having the formula



the said acid having an anion harmless to the scalp.

8. A surface active product which is the acid addition salt of the compound having the formula



the said acid having an anion harmless to the scalp.

References Cited by the Examiner

UNITED STATES PATENTS

2,714,119 7/1955 Crounse ---------- 260—561

LORRAINE A. WEINBERGER, *Primary Examiner.*

M. WEBSTER, *Assistant Examiner.*