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3,003,969

DETERGENT COMPOSITIONS FOR THE HAIR

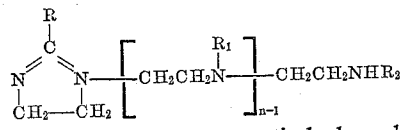
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No Drawing. Filed Nov. 8, 1954, Ser. No. 467,639
Claims priority, application Switzerland Nov. 17, 1953
5 Claims. (Cl. 252—142)

It is known that cation active compounds can be used for the manufacture of detergent compositions for the hair. As compared with anion-active detergents, cation active detergents have the advantage that they favorably influence the properties of the hair. The feel, softness and combability of the hair are considerably improved. The hair acquires a beautiful gloss and impoverished and chemically attacked hair is strengthened to such an extent that further treatments are facilitated. However, although they possess these advantages the cation-active compounds hitherto used possess the disadvantage, as compared with the anion-active products, that their solutions do not foam sufficiently.

The present invention provides a cation-active detergent composition for the hair, which comprises in the form of a water-soluble salt, including a water-soluble quaternary ammonium salt thereof, at least imidazoline containing at least one non-aromatic radical having at least 7 carbon atoms and also containing at least one side chain containing nitrogen, and at least one compound of acid reaction, and, if desired, other compounds known as an addition to detergent compositions for the hair, especially a non-ionogenic cleansing agent.

Among the imidazoline derivatives defined above there come into consideration, more especially, compounds of the general formula



in which R represents a non-aromatic hydrocarbon radical containing 7–18 carbon atoms, R_1 and R_2 represent hydrogen, a radical as defined for R or the atomic grouping



(in which R_3 represents an alkyl radical containing 1–17 carbon atoms), provided that R, R_1 and R_2 together contain not more than 20 carbon atoms, and in which n represents a small whole number.

Especially advantageous are the products in which the sum of the carbon atoms present in the radicals R, R_1 and R_2 is within the range of 10–15.

The imidazoline derivatives can be made by methods in themselves known from polyalkylene-polyamines and non-aromatic carboxylic acids of high molecular weight of the general formula R—COOH or reactive derivatives thereof.

The polyamines used as starting materials for making the above products may contain further substituents, in addition to hydrocarbon radicals and the basic nitrogen atoms. For example, there may be used diethylene triamine, triethylene tetramine or tetraethylene pentamine, and also polyamines such as are obtainable by heating an ethylene dihalide with ammonia or an amine.

As non-aromatic carboxylic acids for use in preparing the imidazoline derivatives there may be mentioned: caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, oleic acid and linoleic acid. As functional derivatives of these acids there may be used their halides, esters or anhydrides.

Inorganic acids may be used for salt formation, for example, hydrochloric acid, sulfuric acid and methylsulfuric acid, and especially advantageous are organic acids, such as acetic acid, lactic acid, formic acid or citric acid. There may also be used for salt formation organic compounds which are capable of reacting with amines to form salts, including quaternary ammonium salts. Such compounds are, for example, benzyl chloride or dimethyl sulfate.

The salts of the imidazolines are easily soluble in water and yield a stable foam in solutons of acid reaction. In order to produce a suitable pH value there is incorporated with the detergent compositions for the hair, in accordance with the invention, at least one compound of acid reaction. As such compounds there may be used salts of acid reaction, such as primary phosphates; but there are especially suitable the organic acids customarily used for this purpose in cosmetics for example, aliphatic hydroxy-carboxylic acids such as glycollic acid and citric acid, and especially lactic acid.

It may also be of advantage to incorporate with the detergent composition a non-ionogenic cleansing agent, in addition to the imidazoline derivative. As such cleansing agents there come into consideration more especially the known ethylene oxide condensation products of fatty alcohols, fatty amines, and alkyl mercaptans and alkylphenols of high molecular weight. These compounds may be derived, for example, from octadecyl alcohol, oleylamine, dodecyl mercaptan or octyl-, nonyl- or dodecyl-phenol.

The detergent compositions for the hair are made by simple mixing and dissolution of the components. There may be used single members of each class of compound constituting the composition or a mixture of two or more compounds of the same class may be used. There may also be added substances customary in detergent compositions for the hair, such as perfumes, dyestuffs, bleaching agents or thickening agents. The new detergent composition can be made up in said, paste-like or liquid form. They are used in the manner usual in the treatment of the hair. As a result of the treatment the hair is usually well defatted, acquires a soft feel and a beautiful gloss, and can be easily brushed since its tendency to become charged electrostatically is considerably reduced.

The following examples illustrate the invention the parts being by weight unless otherwise stated:

Example 1

10 parts of the lactate described below are mixed with 15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The lactate may be prepared as follows:

200 parts of distilled coconut oil fatty acid, 103 parts of diethylene triamine, 0.2 part of boric acid and 150 parts of xylene are heated under reflux until water ceases to be split off. The xylene which flows out of the reflux condenser is passed, before it returns to the reaction vessel, through a water separator in which the water is separated continuously. After about 8 hours the reaction is finished, and the solvent is distilled off under reduced pressure.

16.8 parts of the imidazoline so obtained are heated with 6 parts of lactic acid of 89.4 percent strength on a water bath, while stirring, until a homogeneous mixture is obtained. The resulting mixture is a soft mass which, when taken up in water, yields a strongly foaming solution.

Example 2

10 parts of the lactate described below are mixed with

15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The lactate may be prepared as follows:

239 parts of commercial myristic acid, 103 parts of diethylene triamine, 2 parts of boric acid and 150 parts of xylene are heated under reflux in the manner described in Example 1 until water ceases to be split off. The reaction is finished in 8 hours. The solvent is distilled off under reduced pressure.

15.3 parts of the imidazoline so obtained are stirred with 5.1 parts of lactic acid of 89.4 percent strength on a water bath until a homogeneous mixture is obtained. The mixture is a soft mass which yields a strongly foaming solution in water.

Example 3

10 parts of the lactate described in Example 1 are mixed with 10 parts of the condensation product of 1 mol of para-tertiary-octyl-phenol and 8 mols of ethylene oxide, 15 parts of lactic acid of 89.4 percent strength and 55 parts of water. There is obtained a solution which, when diluted with water, yields a strongly foaming solution.

Instead of the aforesaid addition product of ethylene oxide and para-tertiary-octyl-phenol, there may be used the condensation product from 1 mol of tertiary-dodecyl mercaptan and 8–10 mols of ethylene oxide.

Example 4

10 parts of the lactate described below are mixed with 15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The lactate is prepared as follows:

60 parts of lauric acid, 31 parts of diethylene triamine, and 0.6 part of boric acid in 50 parts of xylene are heated at the boil under reflux in the manner described in Example 1 until water no longer splits off.

The reaction period is about 5 hours, after which period 2 molecular proportions of water calculated on 1 molecular proportion of lauric acid will have split off.

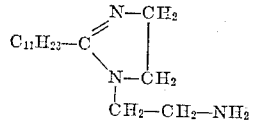
13.4 parts of the imidazoline so obtained are stirred in the warm with 5.1 parts of lactic acid of 89.4 percent strength, and in this manner a soft mass is obtained which is easily soluble in water.

Example 5

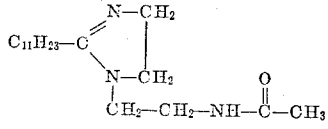
10 parts of the acetate described below are mixed with 15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The aforesaid acetate may be prepared as follows:

26.7 parts of the imidazoline of the formula



described in Example 4 are added in small portions at 80–85° C. to 10.2 parts of acetic anhydride, and then the reaction mixture is heated for 2 hours in a boiling water bath, while stirring. The resulting acetate of the acetylated imidazoline of the formula



is a thickly liquid oil, which, when taken up in water, yields a foaming solution.

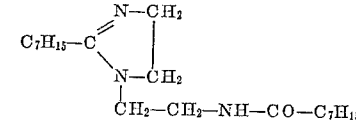
Example 6

10 parts of the lactate described below are mixed with 15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The aforesaid lactate may be prepared as follows:

14.4 parts of caprylic acid, 10.3 parts of diethylene triamine, 0.1 part of boric acid and 50 parts of xylene are heated in the manner described in Example 1 at the boil under reflux until water ceases to be split off. 14.4 parts of caprylic acid are then run in, and the whole is heated at the boil with the use of a water separator until water is no longer split off. After distilling off the xylene, 33.7 parts of the reaction product are obtained.

10 parts of the imidazoline so obtained of the formula



are heated with 3.1 parts of lactic acid of 89.4 percent strength, while stirring, and a thickly liquid oil is so obtained which, when taken up in water, yields a strongly foaming solution.

Example 7

10 parts of the quaternary ammonium salt described below are mixed with 5 parts of lactic acid of 89.4 percent strength and 85 parts of water. The quaternary ammonium salt may be prepared as follows:

10 parts of the imidazoline of the formula given in Example 6 are heated with 3.8 parts of dimethyl sulfate at 50–55° C. for 4 hours, and the quaternary ammonium salt is obtained in the form of a thickly liquid oil. It is easily soluble in water to form a strongly foaming solution.

Example 8

In order to wash living hair a solution of 20 parts of the product described in the first paragraph of Example 1 in 80 parts of warm water is prepared. The hair washed with this strongly foaming solution of acid reaction is well defatted and possesses a beautiful gloss and a pleasant soft feel. Furthermore, the tendency for the hair to become electrostatically charged during brushing is strongly reduced.

Example 9

10 parts of the lactate described below are mixed with 15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The lactate may be prepared as follows:

93 parts of the product described in the penultimate paragraph of Example 1 are heated in a current of nitrogen to 120–130° C. and 43 parts of propylene oxide are introduced as a finely dispersed current of gas at the stated temperature.

39.5 parts of imidazoline so obtained are heated with 15.7 parts of lactic acid of 89.4 percent strength on a water bath until a homogeneous mixture is obtained. The resulting mixture is a viscous mass which, when taken up in water, yields a strongly foaming solution.

Example 10

10 parts of the lactate described below are mixed with 15 parts of lactic acid of 89.4 percent strength and 75 parts of water.

The lactate may be prepared as follows:

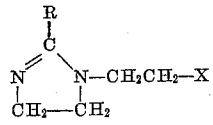
148.5 parts of the product described in the penultimate paragraph of Example 1 are heated in a current of nitrogen to 120° C., and 44 parts of ethylene oxide are introduced at 120–125° C. as a finely dispersed current of gas.

175 parts of the imidazoline so obtained are heated with 45.8 parts of lactic acid of 89.4 percent strength on a water bath until a homogeneous mixture is obtained. The resulting mixture is a viscous mass which, when taken up in water, yields a strongly foaming solution.

What is claimed is:

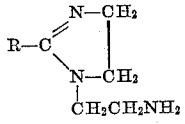
1. A cation-active detergent composition for the hair,

which comprises as a cation-active detergent a water soluble acid addition salt of an imidazoline of the formula



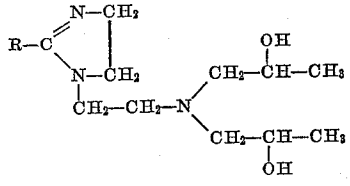
in which R represents an aliphatic hydrocarbon radical containing 11 to 13 carbon atoms and X represents a member selected from the group consisting of a primary amino group, an acylamino group derived from a saturated fatty acid containing at the most 8 carbon atoms, and a hydroxy-alkylated amino group containing at least one hydroxy-alkyl radical which contains at the most 3 carbon atoms, and a sufficient amount of a water soluble aliphatic hydroxy-carboxylic acid to produce an acid reaction in aqueous solution.

2. A cation-active detergent composition for the hair, which comprises as a cation-active detergent the lactate of the imidazoline of the formula



wherein R is the hydrocarbon radical of coconut oil fatty acid, and a sufficient amount of lactic acid to produce an acid reaction in aqueous solution.

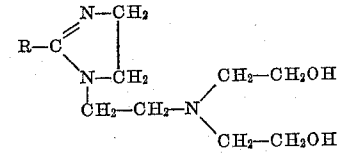
3. A cation-active detergent composition for the hair, which comprises as a cation-active detergent the lactate of the imidazoline of the formula



wherein R is the hydrocarbon radical of coconut oil fatty acid, and a sufficient amount of lactic acid to produce an acid reaction in aqueous solution.

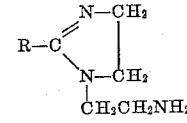
4. A cation-active detergent composition for the hair,

which comprises as a cation-active detergent the lactate of the imidazoline of the formula



wherein R is the hydrocarbon radical of coconut oil fatty acid, and a sufficient amount of lactic acid to produce an acid reaction in aqueous solution.

5. A cation-active detergent composition for the hair, which comprises as a cation-active detergent the lactate of the imidazoline of the formula



wherein R is the hydrocarbon radical of coconut oil fatty acid, a sufficient amount of lactic acid to produce an acid reaction in aqueous solution and the condensation product of 1 mol of tertiary dodecyl mercaptan with 8–10 mols of ethylene oxide.

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UNITED STATES PATENT OFFICE

CERTIFICATION OF CORRECTION

Patent No. 3,003,969 October 10, 1961

Otto Albrecht

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 39, for "said" read -- solid --.

Signed and sealed this 10th day of April 1962.

(SEAL)

Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents