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(54) LIPSTICKS

(71) We, L'OREAL, a French Body Corporate, of 14 Rue Royale, Paris 75008, France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a new cosmetic composition for making-up, which is in the form of a lipstick.

We have already proposed to use, for the production of make-up products and especially of lipsticks, certain polymers and especially homopolymers of vinyl esters or acrylic esters, as well as copolymers of vinyl esters.

In fact, it has been found that using a certain percentage of such polymers in these products, it is possible to improve the firmness of the sticks, and to impart gloss and better adhesion as well as good persistence of the film deposited on the lips.

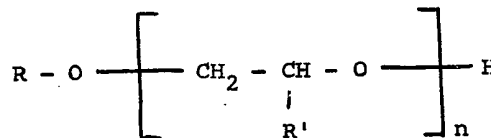
However, these lipstick compositions exhibit certain disadvantages, especially a migration of the dyestuffs into the corners of the lips.

We have now found, according to the present invention, that it is possible to avoid or reduce these disadvantages, which affect the aesthetic appearance, by linking the polymers previously proposed with a particular type of glyceryl ester-ether or glycol ether. In fact, by the combined use of these materials in a lipstick it is possible to

prevent the migration of the dyestuffs into the corners of the lips and moreover further to increase the gloss of the film thus deposited on the lips.

The present invention provides a lipstick which essentially contains:

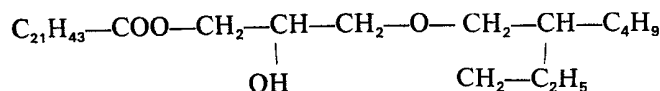
- (i) at least one lipo-soluble polymer containing vinyl ester-derived units,
- (ii) at least 10% by weight of 1-docosanoyloxy - 3 - (2 - ethyl) - hexyloxy - propan-2-ol or a product of the average formula:



in which R represents the radical derived from the alcohols of lanolin (lanolin alcohol), R' represents C₁₀H₂₁ and n is a number, especially about 1.5, i.e. a product obtained by reacting 11,12-epoxydodecane with lanolin alcohol,

- (iii) at least one fatty substance and
- (iv) at least one non-toxic dyestuff.

The 1-docosanoyloxy-3 - (2-ethyl) - hexyloxy-propan-2-ol which, in admixture with the polymer, prevents the migration of the dyestuffs into the corners of the lips, and has the following formula:



This compound, which is novel can be obtained by reacting behenic acid (or docosanoic acid) with glycidyl 2-ethyl-hexyl ether. The reaction is suitably carried out in

the presence of a basic catalyst such as sodium methylate or sodium ethylate and at a temperature of the order of 130°C for, say, 2 to 8 hours.

- The product can be isolated from the reaction mixture after several washes with water and after neutralisation with a base, and is then dried under reduced pressure.
- 5 The volatile products can be removed at about 130°C/10⁻³ mm Hg and the desired product can then be distilled at about 205°C/10⁻³ mm Hg. This product is solid at ambient temperature and has a final melting point of the order of 35° to 40°C.
- 10 The products of formula (I) which likewise prevent the migration of the dyestuffs can be obtained by reacting 11,12-epoxy-dodecane with lanolin alcohol in the presence of boron trifluoride (BF₃). The reaction is conveniently conducted at a temperature of about 80°C for, say, 1 to 2 hours. The product can be isolated after neutralisation and washing with water.
- 15 The product in which n is about 1.5 is novel; it is in the form of a slightly yellow soft wax having a melting point of about 40°C.
- 20 As indicated above, the 1-docosanoyloxy - 3 - (2-ethyl) - hexoxy-propan-2-ol or product of formula (I) is present in the composition at a concentration of at least 10% but preferably 10 to 30% (by weight).
- 25 These compounds partly replaces the fatty substance which may be either a wax, an oil or a mixture of a wax and an oil. According to this invention, the fatty substance is generally present in an amount from 35 to 75%.
- 30 Amongst the waxes which can be used as the fatty substance, there may in particular be mentioned ozocerites, lanolin, lanolin alcohol, hydrogenated lanolin, acetylated lanolin, lanolin wax, beeswax, candellila wax, micro-crystalline wax, carnauba wax, cetyl alcohol, stearyl alcohol, spermaceti, cacao butter, the fatty acids of lanolin, petrolatum, white petroleum jellies, monoglycerides, diglycerides and triglycerides which are solid at 25°C, fatty esters which are solid at 25°C, silicon waxes such as methyloctadecanoxo-polysiloxane and poly - (dimethylsiloxo) -
- 35 stearoxysiloxane, stearyl mono-ethanolamide, colophony and its derivatives such as glycol abietate and glycerol abietate, hydrogenated oils which are solid at 25°C, sucro-glycerides and the oleates, myristates, lanolates, stearates and dihydroxy-stearates of calcium, magnesium, zirconium and aluminium.
- 40 Amongst the oils which can be used as fatty substances there may in particular be mentioned paraffin oil, purcellin oil, perhydrosqualene, sweet almond oil, avocado oil, calophyllum oil, castor oil, caballine oil, lard oil, olive oil, mineral oils having a boiling point of 310 to 410°C, silicone oils such as dimethylpolysiloxanes,
- 45 linoleyl alcohol, linolenyl alcohol, oleyl alcohol, cereal germ oil such as wheat germ oil, isopropyl lanolate, isopropyl palmitate, isopropyl myristate, butyl myristate, cetyl myristate, hexadecyl stearate, 2-ethylhexyl stearate, butyl stearate, octyl hydroxy stearate, decyl oleate, acetyl-glycerides, octanoates and decanoates of alcohols and of poly-alcohols such as of glycol and glycerol, ricinoleates of alcohols and of poly-alcohols such as cetyl ricinoleate, isostearyl alcohol, isocetyl lanolate, isopropyl adipate, hexyl lanolate and octyl-dodecanol.
- 50 It is also possible to use as waxes or as oils, according to the invention, the derivatives of 1,2-alkanediols and especially the esters of 1,2-alkanediols with fatty acids such as those described in our British Specification No. 1,514,287 or one of the compounds described in British Specification Nos. 1,431,153 and 1,516,195.
- 55 The polymers having vinyl ester units which can be used in the lipsticks according to the present invention must be liposoluble, that is to say must have a high affinity for waxes and oils. As indicated above, these polymers are either homopolymers or copolymers and are generally present in the composition at a concentration of 10 to 35% by weight. Amongst the homopolymers there may in particular be mentioned those resulting from the homopolymerisation of vinyl hexanoate, of vinyl 2,2-dimethyl-pentanoate, of vinyl octanoate, of vinyl cekanoates (cekanoic acid being the tradename of a mixture of linear and branched fatty acids having the same number of carbon atoms, namely 8, 9 or 10), of vinyl laurate, of vinyl stearate and of vinyl isosteate.
- 60 Amongst the copolymers, there may in particular be mentioned those resulting from the copolymerisation of vinyl acetate with allyl stearate, of vinyl acetate with vinyl laurate, of vinyl acetate with vinyl stearate, of vinyl acetate with octadecene, of vinyl acetate with octadecyl vinyl ether, of vinyl propionate with allyl laurate, of vinyl propionate with vinyl laurate, of vinyl stearate with 1-octadecene, of vinyl acetate with 1-dodecene, of vinyl stearate with ethyl vinyl ether, of vinyl propionate with cetyl vinyl ether, of vinyl stearate with allyl acetate, of vinyl 2,2-dimethyl-octanoate with vinyl laurate, of allyl 2,2-dimethyl-pentanoate with vinyl laurate, of vinyl dimethylpropionate with vinyl stearate, of allyl dimethylpropionate with vinyl stearate, of vinyl propionate with vinyl stearate, of vinyl dimethylpropionate with vinyl laurate and of allyl propionate with allyl stearate.
- 65 These copolymers can optionally be crosslinked by means of a crosslinking agent, which has the effect of increasing the

molecular weight. Amongst these crosslinking agents there may in particular be mentioned tetraallyloxy-ethane, divinylbenzene, divinyl octanedioate, divinyl dodecanedioate and divinyl octadecanedioate.

Preferably, these homopolymers and copolymers have a molecular weight of 2,000 to 500,000 and preferably 6,000 to 300,000.

These homopolymers and copolymers of vinyl esters are described in detail in our British Specifications Nos. 1,476,194 and 1,476,195.

The dyestuffs employed in the compositions according to the invention are of course non-toxic; they are those used commonly in lipsticks. They are in general present in an amount from 2 to 30%.

Amongst them there may be mentioned the eosins and other halogenated derivatives of fluorescein (bromo-acids), and especially those known under the names of D and C Red No. 21, D and C Red No. 27 and D and C Orange No. 5, inorganic pigments such as iron oxides, chromium oxides, ultramarines (polysulphides of aminosilicates), titanium dioxides, and organic pigments such as D and C Red No. 36 and D and C Orange No. 17.

Finally, amongst the dyestuffs there may also be included the lakes such as the calcium lakes of D and C Red No. 6, 7, 21 and 27, the barium lakes of D and C Red No. 6 and 9, the Al lakes of D and C Red No. 21 and 27 and D and C Yellow No. 5 and 6, and the zirconium lakes of D and C Red No. 21 and of D and C Orange No. 5.

Of course, the compositions according to the invention can also contain other conventional ingredients such as, for example, pearlescent agents, suitably in an amount of 2 to 20%, perfumes, anti-sunburn agents, antioxidants and preservatives.

Suitable pearlescent agents include bismuth oxychloride, mica-, titania and guanine crystals.

Amongst the antioxidants there may in particular be mentioned those of the phenolic type, such as the propyl, octyl and dodecyl esters of gallic acid, butylated hydroxyanisole, butylated hydroxy-toluene and nordihydroguaiaretic acid.

In certain cases, it is also desirable or necessary to use certain solvents for the dyestuffs which are insoluble in the fatty substances. Amongst these solvents there may be mentioned the glycols, the tetrahydrofurfuryl esters, the polyethylene glycols and the monoalkanolamides.

Preparation of 1 - docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propan 2 ol

2.9 g of sodium methylate powder (50 milliequivalents) are added, whilst stirring, to

335 g of behenic (or docosanoic) acid (1.050 mols) which have been fused in a flask, and the temperature is then raised to 130°C under nitrogen.

Thereafter, 186 g (1 mol) of 2-ethylhexyl glycidyl ether are added dropwise, whilst stirring, and after the end of the addition the mixture is heated under nitrogen for 6 hours at 130°C.

The extent of the reaction is determined by measuring the residual acid number. The reaction is continued until the degree of conversion is about 95%.

The product thus obtained is washed once with 500 ml of boiling water containing the amount of sodium hydroxide required to neutralise the residual acidity.

After the water, 200 ml of isopropanol are added to improve the phase separation. The product is then washed twice with water at 80°C (about 500 ml per wash).

The product is then dried in vacuo, whilst stirring, on a boiling waterbath.

Thereafter, the product is purified by molecular distillation.

1) The volatile products are removed at 130°C under 10⁻³ mm Hg

2) The product is distilled at 205°C under 10⁻³ mm Hg,

Distillation yield: 78%

Overall yield: 67%

Analyses:

Saponification number:

theory: 1.98 milliequivalents/g

found: 2.0 milliequivalents/g

Hydroxyl number:

theory: 1.98 milliequivalents/g

found: 1.85 milliequivalents/g

Acid number: zero

Final melting point: 35°C.

Preparation of a product of formula (I)

5.2 ml. of a BF₃/ether complex was added to 740 g (2 mols) of lanolin alcohol which had been previously melted at about 80°C. 552 g (3 mols) of 11,12-epoxydodecane is then added drop by drop over about 1 hour. The temperature is maintained at 80°C. for about 10 minutes after the end of the addition to ensure that all the epoxide added has been consumed.

The product thus obtained is washed with water containing the necessary quantity of soda to neutralise the acidity due to the presence of the catalyst. The organic phase separated after decantation is washed again three times with hot water.

The product is then taken up in its own weight of water and the material distilled to eliminate extracted impurities and then the product is dehydrated completely under reduced pressure.

One thus obtains a product of formula (I) in the form of a soft slightly yellow wax.

Melting point=40°C. OH Index=1.90 meq/g (theory: 1.55).

The following Examples further illustrate the present invention.

5 Examples of Lipsticks

Example 1

A lipstick is prepared, according to the invention, by mixing the following ingredients:

10	Microcrystalline wax	12 g
	Acetylated lanolin	5.9 g
	Lanolin	10 g
	Hydrogenated lanolin	10 g
	Lanolin alcohols	11 g
15	Butylated hydroxy-anisole	0.1 g
	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	8 g
	Homopolymer of vinyl laurate	15 g
20	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propan - 2 - ol	15 g
	Colorants:	
	Titanium oxide	1 g
25	Al lake of D and C Red 27	7.5 g
	D and C Red 36	1 g
	Al lake of D and C Yellow 6	2.5 g
	Perfume	1 g
		100 g

30 A lipstick is prepared, according to the invention, by mixing the following ingredients:

35	Microcrystalline wax	9 g
	Acetylated lanolin	9 g
	Oleyl alcohol	11 g
	Liquid lanolin	8 g
	Mineral oil	10.9 g
	Butylated hydroxytoluene	0.1 g
40	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	10 g
	Homopolymer of vinyl laurate	11 g
45	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propan - 2 - ol	20 g
		3.5 g
50	Zirconium lake of D and C red 21	3.5 g
	Calcium lake of D and C Red 6	0.2 g
	D and C Red 36	1.5 g
	Al lake of D and C Yellow 6	1.5 g
	Perfume	0.8 g
55		100 g

Example 3

A lipstick is prepared, according to the invention, by mixing the following ingredients:

	Ozocerite	15 g	60
	Lanolin	8 g	
	Mineral oil	3 g	
	Oleyl alcohol	5 g	
	Triglycerides	3 g	
	Castor oil	8.4 g	65
	Butylated hydroxytoluene	0.1 g	
	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	10 g	
	Homopolymer of vinyl laurate	10 g	70
	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propan - 2 - ol	15 g	
	Colorants:		
	Al lake of D and C Red 27	1 g	75
	Ca lake of D and C Red 7	1 g	
	D and C Red 36	1 g	
	D and C Red 6	6 g	
	Al lake of D and C Yellow 5	1 g	
	Mica-titania	11 g	80
	Perfume	1.5 g	
		100 g	

Example 4

A lipstick is prepared, according to the invention, by mixing the following ingredients:

	Microcrystalline wax	8 g	
	Liquid lanolin	10 g	
	Mineral oil	4 g	
	Acetylated lanolin	6.9 g	90
	Hydrogenated palm oil	6 g	
	Compound of the formula		
	$\begin{array}{c} \text{R}-\text{COOCH}_2-\text{CH}-\text{R}' \\ \\ \text{OH} \end{array}$	5 g	
	R=C ₁₅ H ₃₁ Melting point=55—60°C		
	R'=C ₁₂ /C ₁₄		
	Butylated hydroxyanisole	0.1 g	95
	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	15 g	
	Homopolymer of vinyl laurate	10 g	100
	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propan - 2 - ol	15 g	
	Colorants		
	Titanium oxide	3.75 g	105
	Al lake of D and C Red 27	1.25 g	
	D and C Red 30	1 g	
	Al lake of D and C Yellow 6	1 g	
	Bi oxychloride	12 g	
	Perfume	1 g	110
		100 g	

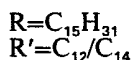
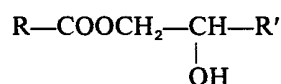
Example 5

A lipstick is prepared, according to the invention, by mixing the following ingredients:

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	Ozocerite	4 g	Colorants:		
	Microcrystalline wax	6 g	Titanium oxide	3 g	
	Acetylated lanolin	10 g	Al Laque D and C red 21	1 g	50
	Castor oil	10 g	Ca Laque D and C red 7	0.2 g	
5	Hydrogenated coconut oil	10 g	D and C red 6	2.5 g	
	Lanolin alcohols	3.9 g	Yellow iron oxide	1.1 g	
	Compound of the formula		Black iron oxide	0.35 g	
	$\begin{array}{c} \text{R}-\text{COOCH}_2-\text{CH}-\text{R}' \\ \\ \text{OH} \end{array}$	6 g	Al Laque D and C yellow 5	4.5 g	55
			Perfume	0.5 g	
				100 g	
	$\text{R}=\text{C}_{15}\text{H}_{31}$ Melting point 55—60°C $\text{R}'=\text{C}_{12}/\text{C}_{14}$ Acid number 0.02 milliequivalent/g		Example 7		
10	Butylated hydroxytoluene	0.1 g	A lipstick is prepared by mixing the following ingredients:		60
	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	8 g	Polyvinyl laurate	9.2 g	
15	Homopolymer of vinyl laurate	16 g	Polyethylene grease (MW=1,500)	33 g	
	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propan - 2 - ol	15 g	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexyloxy - propan - 2 - ol	14 g	65
			Compound of the formula:		
20	Colorants		$\begin{array}{c} \text{R}-\text{COO}-\text{CH}_2-\text{CH}-\text{R}' \\ \\ \text{OH} \end{array}$	4 g	
	Titanium oxide	6.5 g			
	Ca lake of D and C Red 7	0.5 g			
	D and C Red 36	0.5 g			
	Black iron oxide	0.4 g			
25	Al lake of D and C Yellow 5	2.6 g	$\text{R}=\text{C}_{15}\text{H}_{31}$ Melting point=55—60°C $\text{R}'=\text{C}_{12}/\text{C}_{14}$		70
	Perfume	0.5 g			
		100 g	Lanolin	9.7 g	
			Liquid lanolin	10.5 g	
			Amyl paradimethylamino-benzoate	1 g	
			D-panthenol	1 g	75
			Calendula oil	8 g	
30	Polyvinyl laurate	17.0 g	Polyethylene wax	2 g	
	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	8.75 g	Di - tertiarybutyl para - cresol	0.1 g	
	Microcrystalline wax	8.75 g	Mineral oil	3 g	
35	Di-tertiarybutyl para-cresol	0.1 g	Colourants:		80
	Product of formula (I) such as prepared in the Example above	17.5 g	Al Laque D and C red 21	0.2 g	
	Compound of the formula:		D and C red 6	0.5 g	
40	$\begin{array}{c} \text{R}-\text{COOCH}_2-\text{CH}-\text{R}' \\ \\ \text{OH} \end{array}$	4.35 g	Yellow iron oxide	0.3 g	
			Black iron oxide	0.4 g	
			Al Laque D and C yellow 5	2.5 g	85
			Perfume	0.6 g	
				100 g	
	$\text{R}=\text{C}_{15}\text{H}_{31}$ Melting point=55—60°C $\text{R}'=\text{C}_{12}/\text{C}_{14}$		Example 8		
			A lipstick is prepared by mixing the following ingredients:		90
45	Butyl ricinoleate	13 g	Polyvinyl laurate	7 g	
	Aceto glyceride	4.35 g	Polyethylene grease	27 g	
	Acetylated lanolin	4.35 g	Product of formula (I) such as prepared in the above Example	9.5 g	95
	Oleic alcohol	4.35 g			
	Liquid lanolin	4.35 g			

Compound of the formula:

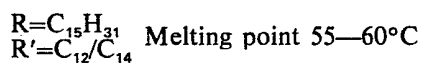
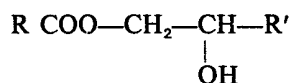


5	Lanolin	9.0 g
	Acetylated lanolin	9.5 g
	Mineral oil	9.5 g
	Polyethylene wax	3.5 g
	Tertiary-butyl anisole	0.1 g
10	Butyl ricinoleate	6.4 g
	Octyl-hydroxystearate	6.5 g
	Colourants:	
	Titanium oxide	2 g
	Al Laque D and C red 27	2 g
15	Ca Laque D and C red 7	1 g
	D and C red 6	3 g
	Perfume	0.5 g
		100 g

Example 9

- 20 A lipstick is prepared by mixing the following ingredients:

	Polyvinyl laurate	28 g
	Copolymer of 31.3% of vinyl acetate and 68.7% of allyl stearate	4.8 g
25	Polyethylene grease	33 g
	1 - Docosanoyloxy - 3 - (2 - ethyl) - hexyloxy - propan - 2 - ol	19.3 g
30	Compound of the formula:	



35	Tertiary-butyl anisole	0.1 g
	Acetylated lanolin	4.8 g
	Mineral oil	4.8 g
	Colourants:	
	Al Laque D and C red 21	0.6 g
	D and C red 36	0.3 g
40	D and C red 30	0.5 g
	D and C red 13	0.2 g
	Al Laque D and C yellow 5	2 g
	Perfume	0.6 g
		100 g

- 45 WHAT WE CLAIM IS:—
1. A lipstick, which contains:
(i) at least one lipo-soluble polymer containing vinyl ester-derived units,

(ii) at least 10% by weight of 1 - docosanoyloxy - 3 - (2 - ethyl) - hexyloxy - propan - 2 - ol or a product obtained by reacting 11,12-epoxydodecane with the alcohols of lanolin,

(iii) at least one fatty substance and
(iv) at least one non-toxic dyestuff.

2. A lipstick according to claim 1 in which the polymer is a homopolymer of vinyl hexanoate, vinyl 2,2-dimethyl-pentanoate, vinyl octanoate, vinyl cecanoates, vinyl laurate, vinyl stearate or vinyl isostearate.

3. A lipstick according to claim 1 in which the polymer is a copolymer of: vinyl acetate with allyl stearate, vinyl acetate with vinyl laurate, vinyl acetate with vinyl stearate, vinyl acetate with octadecene, vinyl acetate with octadecyl vinyl ether, vinyl propionate with allyl laurate, vinyl propionate with vinyl laurate, vinyl stearate with 1-octadecene, vinyl acetate with 1-dodecene, vinyl stearate with ethyl vinyl ether, vinyl propionate with cetyl vinyl ether, vinyl stearate with allyl acetate, vinyl 2,2-dimethyl-octanoate with vinyl laurate, allyl 2,2-dimethyl-pentanoate with vinyl laurate, vinyl dimethylpropionate with vinyl stearate, allyl dimethylpropionate with vinyl stearate, vinyl propionate with vinyl stearate, vinyl dimethylpropionate with vinyl laurate or allyl propionate with allyl stearate.

4. A lipstick according to any one of the preceding claims in which the polymer is one which has been cross-linked with tetraallyloxyethane, divinylbenzene, divinyl octanedioate, divinyl dodecanedioate or divinyl octadecanedioate.

5. A lipstick according to any one of the preceding claims in which the polymer is present in an amount from 10 to 35% by weight.

6. A lipstick according to any one of the preceding claims, characterised in that the 1 - docosanoyloxy - 3 - (2 - ethyl) - hexoxy - propane - 2 - ol or the product obtained from 11,12-epoxydodecane and lanolin alcohol is present in an amount from 10 to 30% by weight.

7. A lipstick according to any one of the preceding claims in which the fatty substance is at least one wax or at least one oil or a mixture of at least one wax and at least one oil.

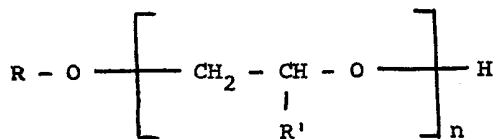
8. A lipstick according to claim 7 in which the fatty substance is present in an amount of from 35 to 75% by weight.

9. A lipstick according to any one of the preceding claims in which the non-toxic dyestuff is present in an amount from 2 to 30% by weight.

10. A lipstick according to any one of the preceding claims which also contains one or more pearlescent agents, perfumes, anti-sunburn agents, antioxidants, preservatives,

or solvents for the dyestuff if the latter is insoluble in the fatty substance.

- 5 11. A lipstick according to any one of the preceding claims in which the product obtained from 11,12-epoxydodecane and lanolin alcohol has the average formula:



- 10 in which R represents a radical derived from the alcohols of lanolin, R' represents $\text{C}_{10}\text{H}_{21}$ and n is about 1.5.

12. A lipstick according to any one of claims 1 to 10 in which component (ii) is 1-docosanoyloxy-3 - (2-ethyl) - hexyloxypropan-2-ol.

13. A lipstick according to claim 1 15 substantially as hereinbefore described.

14. A lipstick according to claim 1 substantially as described in any one of Examples 1 to 9.

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