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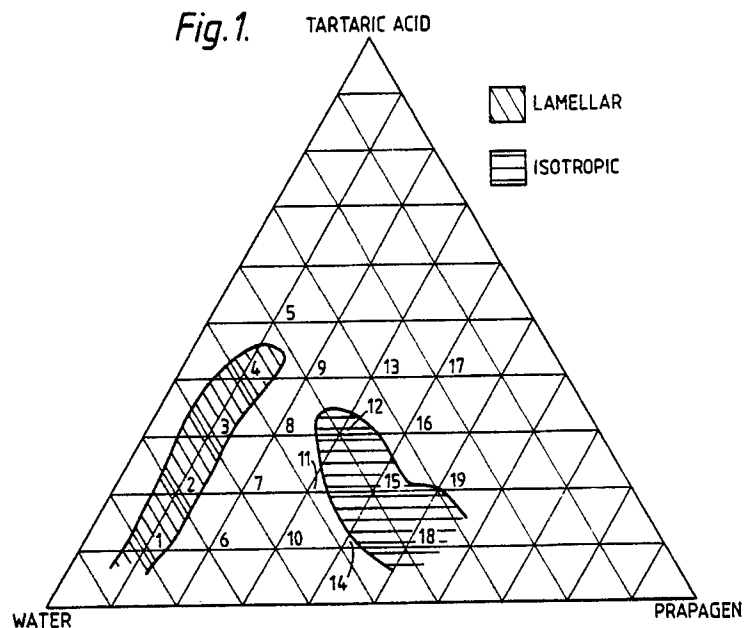
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(54) **Fabric softening composition.**

(57) Isotropic liquid fabric softening composition comprising:  
(i) at least 20% by weight of a fabric softening material; and  
(ii) at least 5% by weight of an organic acid preferably the composition has a clear appearance.

Fig.1.



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## FABRIC SOFTENING COMPOSITION

The present invention relates to fabric softening compositions suitable for softening fabrics in the rinse step of a fabric laundry process.

It has been proposed in DE 33 12 328 (BENCKISER) to prepare fabric softening compositions comprising a combination of fabric softening materials and organic acids, in specific polycarboxylic acids.

5 The compositions as disclosed in this document, however, are of low active levels of up to about 10% by weight. These compositions of low active level are of lamellar structure, wherein the molecules of the fabric softening material form an onion-like configuration comprising concentric bilayers of softening material, between which is trapped an aqueous phase containing the dissolved acid ingredient. By increasing the active level of these formulations, an onset of instability and/or gel formation is observed when the active level exceeds a certain critical level of about 13% by weight of the composition. Therefore up till now it was believed to be impossible to formulate stable fabric softening compositions containing high levels of fabric softening materials in combination with organic acids.

Surprisingly, it has now been found that by further increasing the active level of these compositions, a second class of stable liquid fabric softening compositions can be made. These compositions, unexpectedly are isotropic, they do not comprise a structure of active ingredients. These isotropic compositions are preferred over lamellar compositions in that they are more resistant to temperature fluctuations, and they allow a more flexible use of ingredients.

Accordingly, the present invention relates to isotropic liquid fabric softening compositions comprising

- (i) at least 20% by weight of a fabric softening material; and
- (ii) at least 5% by weight of an organic acid.

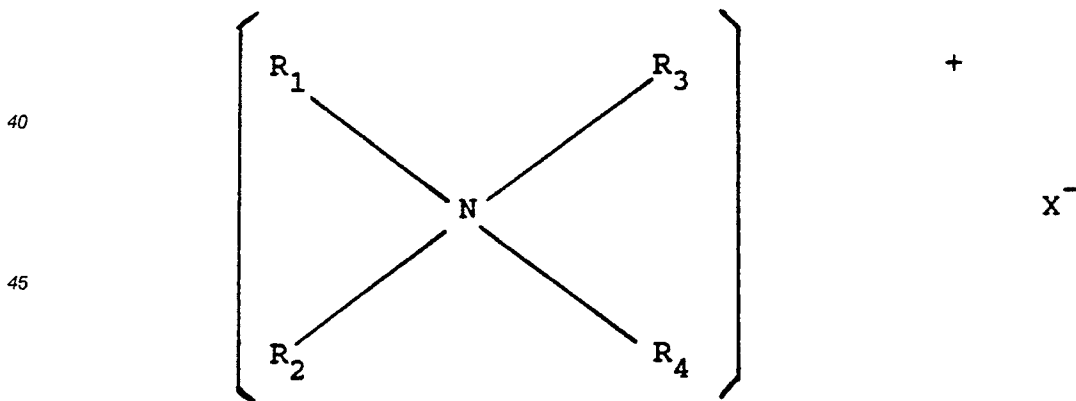
Preferably the isotropic compositions of the present invention are clear or translucent. Softening compositions which are clear are sometimes particularly appreciated by the consumer, because of their fresh and natural appearance. Clear compositions can be made by ensuring that all ingredients of the composition are completely dissolved in the liquid phase.

#### The fabric softening material

Compositions according to the invention contain at least 20% by weight of a fabric softening material. This material may be selected from cationic, nonionic and amphoteric softening materials, and mixtures thereof.

Suitable cationic fabric softener materials include water-insoluble cationic materials having a solubility in water at pH 2.5 and 20 °C of less than 10g/l. Highly preferred materials are cationic quaternary ammonium salts having two C<sub>12-14</sub> hydrocarbyl chains.

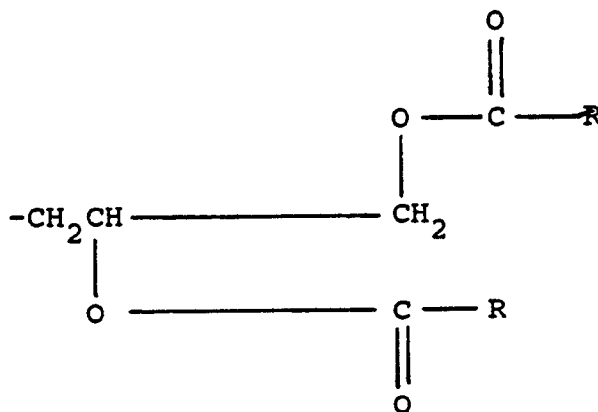
Well-known species of substantially water-insoluble quaternary ammonium compounds have the formula



wherein R<sub>1</sub> and R<sub>2</sub> represent hydrocarbyl groups from about 12 to about 24 carbon atoms; R<sub>3</sub> and R<sub>4</sub> represent hydrocarbyl groups containing from 1 to about 4 carbon atoms; and X is an anion, preferably selected from halide, methyl sulfate and ethyl sulfate radicals.

Representative examples of these quaternary softeners include ditallow dimethyl ammonium chloride; ditallow dimethyl ammonium methyl sulfate; dihexadecyl dimethyl ammonium chloride; di(hydrogenated tallow) dimethyl ammonium methyl sulfate; dihexadecyl diethyl ammonium chloride; di(coconut) dimethyl ammonium chloride. Ditallow dimethyl ammonium chloride, di(hydrogenated tallow) dimethyl ammonium chloride, di(coconut) dimethyl ammonium methosulfate are preferred.

Suitable materials also include dialkyl ethoxyl methyl ammonium methosulphate based on soft fatty acid, dialkyl ethoxyl methyl ammonium methosulphate based on hard fatty acid, and a material in which R<sub>3</sub> and R<sub>4</sub> represent methyl, R<sub>1</sub> is C<sub>13-15</sub>, R<sub>2</sub> is CH<sub>2</sub>CH<sub>2</sub>OCOR, where R is stearyl, and X is methosulphate. Materials in which R<sub>2</sub>, R<sub>3</sub> and R<sub>4</sub> each represent methyl, R<sub>1</sub> is the group



where R is hardened tallow and X is methosulphate or R<sub>2</sub> is methyl, R<sub>3</sub> and R<sub>4</sub> each represent

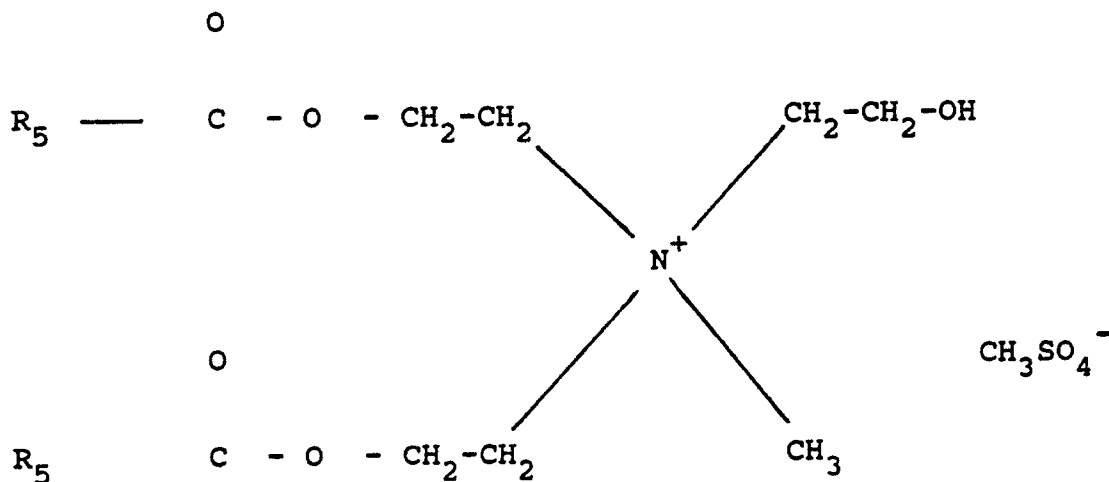
-CH<sub>2</sub>- $\overset{\text{O}}{\parallel}$ -C-R, where R is hardened tallow, R<sub>1</sub> is CH<sub>2</sub>CH<sub>2</sub>OH and X is methosulphate are also suitable.

Preferably cationic softeners are used which have an active melting point (transition from Lb to L state) of less than 25°C, more preferred less than 20°C. Examples of these materials are di-unhardened-tallow dimethyl ammonium chloride and di coconut dimethyl ammonium chloride.

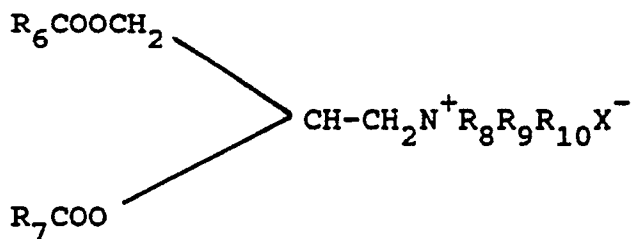
Other preferred cationic compounds include those materials as disclosed in EP 239,910 (P&G), which is included herein by reference.

In this specification the expression hydrocarbaryl group refers to alkyl or alkenyl groups optionally substituted or interrupted by functional groups such as -OH, -O-, -CONH-, -COO-, etc.

Other preferred materials are the materials of formula

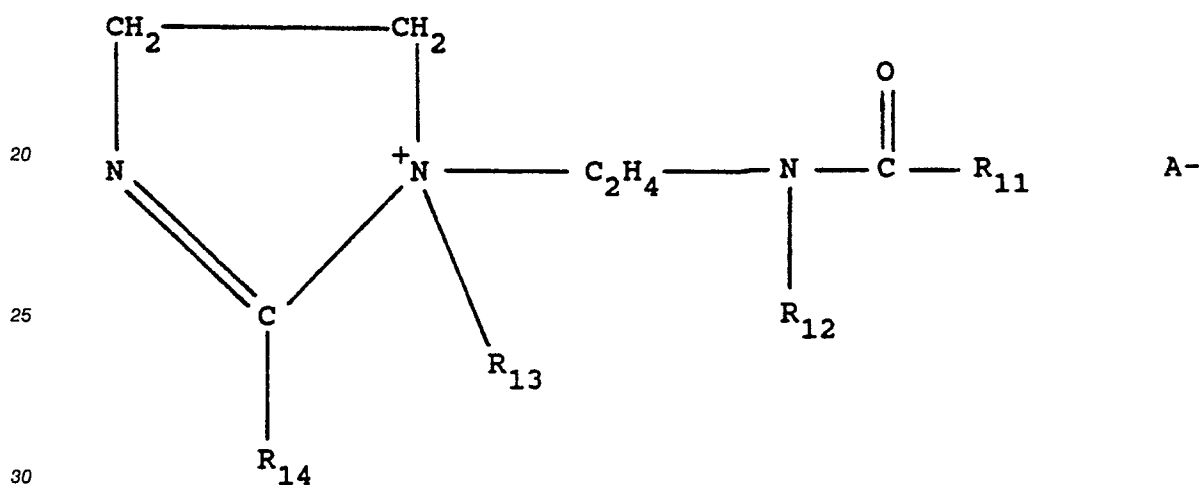


R<sub>5</sub> being talow, which is available from Stepan under the tradename Stepantex VRH 90 and



where  $\text{R}_8$ ,  $\text{R}_9$  and  $\text{R}_{10}$  are each alkyl or hydroxyalkyl groups containing from 1 to 4 carbon atoms, or a benzyl group.  $\text{R}_6$  and  $\text{R}_7$  are each an alkyl or alkenyl chain containing from 11 to 23 carbon atoms, and  $\text{X}^-$  is a water soluble anion, substantially free of the corresponding monoester.

Another class of preferred water-insoluble cationic materials are the hydrocarbylimidazolinium salts believed to have the formula:



wherein  $\text{R}_{13}$  is a hydrocarbyl group containing from 1 to 4, preferably 1 or 2 carbon atoms,  $\text{R}_{11}$  is a hydrocarbyl group containing from 8 to 25 carbon atoms,  $\text{R}_{14}$  is an hydrocarbyl group containing from 8 to 25 carbon atoms and  $\text{R}_{12}$  is hydrogen or an hydrocarbyl containing from 1 to 4 carbon atoms and  $\text{A}^-$  is an anion, preferably a halide, methosulfate or ethosulfate.

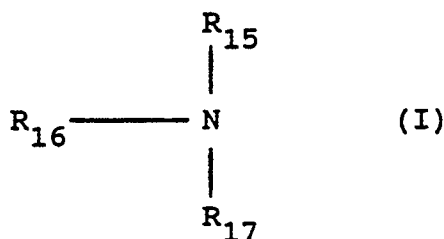
Preferred imidazolinium salts include 1-methyl-1-(tallowylamido-) ethyl -2-tallowyl-4,5-dihydro imidazolinium methosulfate and 1-methyl-1-(palmitoylamido) ethyl -2-octadecyl-4,5-dihydroimidazolinium chloride. Other useful imidazolinium materials are 2-heptadecyl-1-methyl-1- (2-stearyl-amido)ethyl-imidazolinium chloride and 2-lauryl-1-hydroxyethyl-1-oleyl-imidazolinium chloride. Also suitable herein are the imidazolinium fabric softening components of US patent No 4 127 489, incorporated by reference.

Representative commercially available materials of the above classes are the quaternary ammonium compounds Arquad 2HT (ex AKZO); Noranium M2SH (ex CEKA); Aliquat-2HT (Trade Mark of General Mills Inc), Stepanex Q185 (ex Stepan); Stepanex VP85 (ex Stepan); Stepanex VRH90 (ex Stepan); Synprolam FS (ex ICI) and the imidazolinium compounds Varisoft 475 (Trade Mark of Sherex Company, Columbus Ohio) and Rewoquat W7500 (Trade Mark of REWO).

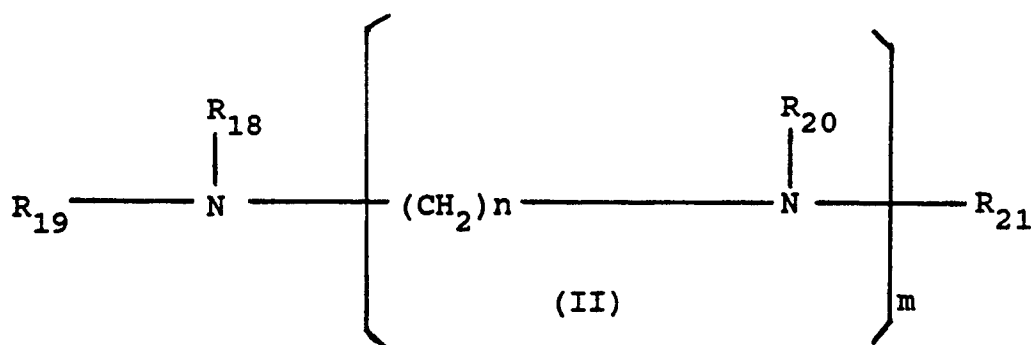
The fabric softening materials may also comprise instead of or in addition to cationic fabric softening agents, one or more amines.

The term "amine" as used herein can refer to

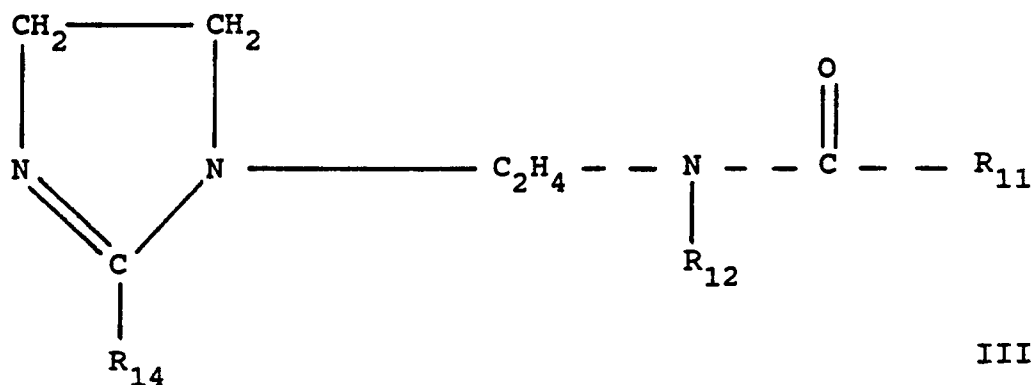
(i) amines of formula



wherein  $R_{15}$ ,  $R_{16}$  and  $R_{17}$  are defined as below;  
 (ii) amines of formula



wherein  $R_{18}$ ,  $R_{19}$ ,  $R_{20}$  and  $R_{21}$ ,  $m$  and  $n$  are defined as below.  
 (iii) imidazolines of formula



wherein  $R_{11}$ ,  $R_{12}$  and  $R_{14}$  are defined as above.

(iv) condensation products formed from the reaction of fatty acids with a polyamine selected from the group consisting of hydroxy alkylalkylenediamines and dialkylenetriamines and mixtures thereof. Suitable materials are disclosed in European Patent Application 199 382 (Procter and Gamble), incorporated herein by reference.

When the amine is of the formula I above,  $R_{15}$  is a  $C_6$  to  $C_{24}$  hydrocarbyl group,  $R_{16}$  is a  $C_1$  to  $C_{24}$  hydrocarbyl group and  $R_{17}$  is a  $C_1$  to  $C_{10}$  hydrocarbyl group. Suitable amines include those materials from which the quaternary ammonium compounds disclosed above are derived, in which  $R_{15}$  is  $R_1$ ,  $R_{16}$  is  $R_2$  and  $R_{17}$  is  $R_3$ . Preferably, the amine is such that both  $R_{15}$  and  $R_{16}$  are  $C_6$ - $C_{20}$  alkyl with  $C_{16}$ - $C_{18}$  being most preferred and with  $R_{17}$  as  $C_1$ - $C_3$  alkyl, or  $R_{15}$  is an alkyl or alkenyl group with at least 22 carbon atoms and  $R_{16}$  and  $R_{17}$  are  $C_1$ - $C_3$  alkyl. Preferably these amines are protonated with hydrochloric acid, orthophosphoric acid (OPA),  $C_1$ - $C_5$  carboxylic acids or any other similar acids, for use in the fabric conditioning compositions of the invention.

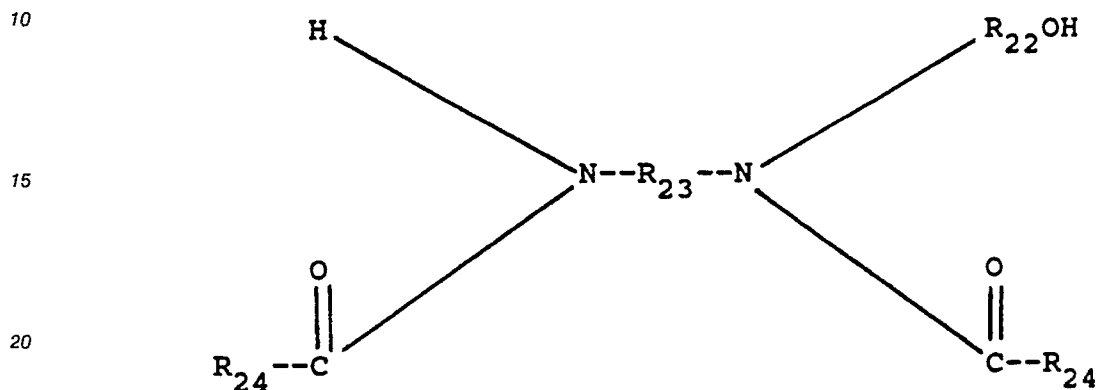
When the amine is of formula II above,  $R_{18}$  is a  $C_6$  to  $C_{24}$  hydrocarbyl group,  $R_{19}$  is an alkoxyated group of formula  $-(CH_2CH_2O)_yH$ , where  $y$  is within the range from 0 to 6,  $R_{20}$  is a group of formula  $-(CH_2CH_2O)_zH$  where  $z$  is within the range from 0 to 6 and  $m$  is an integer within the range from 0 to 6, and

is preferably 13. When m is 0, it is preferred that R<sub>18</sub> is a C<sub>16</sub> to C<sub>22</sub> alkyl and that the sum total of z and y is within the range from 1 to 6, more preferably 1 to 3. When m is 1, it is preferred that R<sub>18</sub> is a C<sub>16</sub> to C<sub>22</sub> alkyl and that the sum total of x and y and z is within the range from 3 to 10.

Representative commercially available materials of this class include Ethomeen (ex Armour) and  
5 Ethoduomeen (ex Armour).

Preferably the amines of type (ii) or (iii) are also protonated for use in the fabric conditioning compositions of the invention.

When the amine is of type (iv) given above, a particularly preferred material is



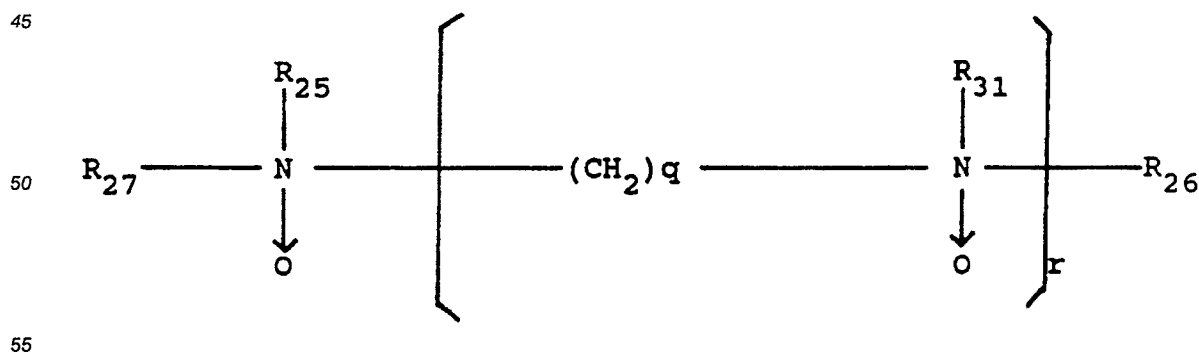
where R<sub>22</sub> and R<sub>23</sub> are divalent alkenyl chains having from 1 to 3 carbons atoms, and R<sub>24</sub> is an acyclic  
25 aliphatic hydrocarbon chain having from 15 to 21 carbon atoms. A commercially available material of this  
class is Ceranine HC39 (ex Sandoz).

Alkoxylated fatty amides or amines may also be used as fabric softening materials in compositions of the invention. Suitable materials are for instance disclosed in EP 159 921 (Unilever) and Ep 159 919 (Unilever). The fabric softening material may also comprise instead of or in addition to the above mentioned softening materials one or more fabric substantive amphoteric fabric softening materials. Suitable am-  
30 photeric materials form a particulate dispersion at a concentration of less than 1g/l at least one temperature between 0 and 100°C. For the purpose of this invention a fabric substantive amphoteric material is preferably an amphoteric or zwitterionic tertiary or quaternary ammonium compound having either one single very long hydrocarbyl side chain or two long hydrocarbyl chains. From these compounds the use of  
35 amphoteric or zwitterionics ammonium compounds having two long hydrocarbyl chains is particularly preferred for many reasons including costs, ease of processing and better stability and performance.

Amphoteric or zwitterionic ammonium compounds preferably have two long hydrocarbyl chains, each chain having 8-24C-atoms, preferably 10-20C-atoms, most preferred around 16C-atoms.

Suitable amphoteric fabric substantive materials for use in a fabric treatment composition according to the invention are for instance: ampholytes, hydrocarbonyl betaines, hydrocarbonylamide betaines, glycines, propionates and tertiary amine oxides. These materials are described in our co-pending patent application EP 89200545.5.

Particularly preferred amphoteric softening materials are tertiary amine oxides of the following formula



wherein:

a)  $R_{25}$  and  $R_{26}$  are  $C_{8-25}$  hydrocarbyl chains,  $R_{27}$  is an hydrocarbyl group containing 1-4 carbon

atoms or a group  $-(CH_2CH_2O)_nH$ ,  $R_{28}$ ,  $R_{29}$ ,  $R_{30}$  are  $-(CH_2)-$ , which can be interrupted with  $-O-$ ,  $-CONH-$ ,  $-COO-$  etc,  $R_{31}$  is  $R_{27}$ ,  $r$  is 0 or 1,  $n$  is an integer from 1-6,  $X$ ,  $Y$  are  $SO_3$ ,  $SO_3^-$  or  $COO^-$ ;

or

- b)  $R_{25}$  is a  $C_{16-50}$  hydrocarbyl chain,  $R_{26}$ ,  $R_{27}$  are hydrocarbyl groups containing 1-4 carbon atoms or a group  $-(CH_2CH_2O)_nH$ ,  $R_{28}$ ,  $R_{29}$ ,  $R_{30}$  are  $-(CH_2)_n-$  which can be interrupted by  $-O-$ ,  $-COHN-$ ,  $-COO-$ , etc,  $R_{31}$  is  $R_{27}$ ,  $r$  is 0 or 1,  $n$  is an integer from 1-6,  $X$ ,  $Y$  are  $SO_3$ ,  $SO_3^-$  or  $COO^-$ .

Preferably the amphoteric fabric substantive materials are water soluble and have a solubility in water at pH 2.5 at 20 °C of less than 10g/l.

The HLB of the amphoteric fabric substantive material is preferably less than 10.0.

- Examples of amphoteric materials of the above groups and their method of preparation are given in our co-pending European patent application 89200113.2.

- The compositions may also contain, instead of or in addition to the above mentioned fabric softening agents, non-cationic fabric softening agents, such as nonionic fabric softening agents. Suitable nonionic fabric softening agents, include glycerol esters, such as glycerol mono-stearate, fatty alcohols, such as stearyl alcohol, alkoxylated fatty alcohols  $C_9-C_{24}$  fatty acids such as Dobanol 91-6 (SHELL) and lanolin and derivatives thereof. Suitable materials are disclosed in European Patent Application 88 520 (Unilever PLC/NV case C1325), 122 141 (Unilever PLC/NV case C1363) and 79 746 (Procter and Gamble), the disclosure of which are incorporated herein by reference.

- Preferably compositions of the invention contain at least some cationic fabric softening materials. Preferably at least 50% of the fabric softening material is a cationic fabric softener material.

The total level of softening material is more than 20% by weight, more preferred more than 30% by weight, most preferred more than 35% by weight of the composition. Generally the total level of softening materials in the composition will be less than 70% by weight, more preferred less than 60% by weight, typically between 40 and 50% by weight.

- Preferably, the compositions of the present invention contain substantially no anionic material, in particular no anionic surface active material. If such materials are present, the weight ratio of the cationic fabric softening agent to the anionic material should preferably be more than 5:1.

### 30 The organic acid material

The organic acid material for use in compositions of the present invention may be selected from the group of mono-, di-, tri- or polycarboxylic acids preferably having a total number of carbon atoms of 8 or less, preferably 4 or less.

- Examples of suitable organic acid materials are succinic acid, malic acid, tartaric acid, citric acid, glutaric acid, acetic acid, propionic acid, and lactic acid or mixtures thereof. For obtaining stable products within a wide range of concentrations, the use of lactic acid and acetic acid is preferred. For obtaining products with a particularly interesting appearance and good colour stability it is preferred to use citric acid.

- The level of acid material in the composition (calculated on an anhydrous basis) of the acid material, is at least 5% by weight of the composition more preferred more than 10%. Generally the level of acid material will be less than 50%, more preferred less than 40%, typically between 15% and 35% by weight of the composition.

### 45 Optional ingredients

- The composition can also contain one or more optional ingredients selected from non-aqueous solvents such as  $C_1-C_4$  alkanols and polyhydric alcohols, pH buffering agents, rewetting agents, viscosity modifiers such as electrolytes, for example calcium chloride, antigelling agents, perfumes, perfume carriers, fluorescers, colourants, hydrotropes, antifoaming agents, hydrocarbons, antiredeposition agents, enzymes, optical brightening agents, opacifiers, stabilisers such as guar gum and polyethylene glycol, emulsifiers, anti-shrinking agents, lanolin or lanolin like materials, anti-wrinkle agents, fabric crisping agents, anti-spotting agents, soil-release agents, germicides, linear or branched silicones, fungicides, anti-oxidants, anti-corrosion agents, preservatives such as Bronopol (Trade Mark), a commercially available form of 2-bromo-2-nitropropane-1,3-diol, dyes, bleaches and bleach precursors, drape imparting agents, antistatic agents and ironing aids.

These optional ingredients, if added, are each present at levels up to 5% by weight of the composition, except for the hydrocarbons and the non-aqueous solvents which may be included at levels of up to 30%.

The pH of the composition is preferably below the pK of the organic acid, more preferred at least one unit below the pK, typically between 2.0 and 5.0.

Soil-release agents particularly preferred in the compositions according to the invention are polymers. Suitable polymers include alkyl and hydroxyalkyl cellulose ethers, such as methyl cellulose, and polyvinylpyrrolidone.

Silicones can be included in the compositions as the ironing aid, rewetting agent or the antifoaming agent. Suitable silicones for use in the compositions according to the invention include predominantly linear polydialkyl or alkylaryl siloxanes in which the alkyl groups contain one to five carbon atoms. The siloxanes can be amido or amino substituted. When the siloxane is amine substituted the amine group may be quaternised.

Fatty acid materials or other nonionic extenders can also be included in compositions of the present invention. Suitable materials and their amounts are for instance disclosed in EP 13780 (P&G) and DE 29 43 606 (Unilever).

The balance of the composition is preferably water. Although water-free systems are also within the ambit of the invention, preferably the amount of water in the composition is more than 20% by weight, more preferred more than 25% by weight. Generally the water level will be less than 70%, more preferred less than 60%, most preferred less than 55%, typically between 55% and 25%.

The viscosity of the product is preferably less than 250 mPas at 106 s<sup>-1</sup>.

In use compositions of the present invention may be prediluted or dosed in concentrated form into the rinse-cycle of a fabric washing process. Preferably the material is used at a concentration of between 0.01 and 2g/l, especially between 0.1 and 1g/l.

The invention will be further illustrated by means of the following examples:

#### Example I

Fabric softening compositions containing water, tartaric acid (on a dry basis) and a cationic fabric softening material Prapagen 3445 (70% di-unhardened tallow di-methyl ammonium chloride 20% isopropylalcohol, 10% water) were prepared by mixing under light agitation.

For each composition the structure and appearance were assessed, the results are given in table 1, and graphically expressed in figure 1.

COMPOSITION	WATER/PRAPAGEN/TARTARIC (%)	RESULT
1.	80/10/10	lamellar/milky
2.	70/10/20	lamellar/milky
3.	60/10/30	lamellar/milky
4.	50/10/40	lamellar/milky
5.	40/10/50	gel
6.	70/20/10	gel
7.	60/20/20	gel
8.	50/20/30	gel
9.	40/20/40	gel
10.	60/30/10	gel
11.	50/30/20	gel
12.	40/30/30	isotropic/clear
13.	30/30/40	phase separation
14.	50/40/10	gel
15.	40/40/20	isotropic/clear
16.	30/40/30	phase separation
17.	20/40/40	phase separation
18.	40/50/10	isotropic/clear
19.	30/50/20	isotropic/clear (some phase separation)

These examples clearly show that lamellar/milky products are formed at low active concentrations; by increasing the active level, unacceptable gelled products are obtained, but surprisingly by further increasing



the active levels isotropic/clear compositions can be obtained (compositions 12, 15, 18 and 19 according to the present invention).

## 5 Example II

Example 1 was repeated while tartaric acid was replaced by citric acid. The results are expressed in table 1 and figure 2.

COMPOSITION	WATER/PRAPAGEN/CITRIC (%)	RESULT
20.	80/10/10	lamellar/milky
21.	70/10/20	lamellar/milky
22.	60/10/30	lamellar/milky
23.	50/10/40	gel
24.	70/20/10	gel
25.	60/20/20	gel
26.	50/20/30	gel
27.	40/20/40	gel
28.	60/30/10	gel
29.	50/30/20	gel
30.	40/30/30	isotropic/clear
31.	30/30/40	phase separation
32.	50/40/10	isotropic/clear
33.	40/40/20	isotropic/clear
34.	30/40/30	isotropic/clear
35.	40/50/10	phase separation
36.	30/50/20	phase separation

Again, surprisingly isotropic liquids according to the invention (Compositions 30, 32, 33, 34) can be formulated by increasing the level of fabric softening materials. Especially interesting was the clear natural yellow appearance of the product, no decolouration was observed during storage.

## EXAMPLE III

Example I was repeated while replacing tartaric acid by acetic acid or by lactic acid.

By using acetic acid isotropic/clear compositions could be formulated even at levels of PRAPAGEN of 50% or more. At lower concentrations a similar behaviour as observed for tartaric acid was observed: lamellar systems at low concentrations, and gelled systems at intermediate concentrations of softening material.

By using lactic acid, similar results as with acetic acid are observed. This indicates that for stability reasons, especially at high levels of softening materials the use of acetic acid and lactic acid is preferred.

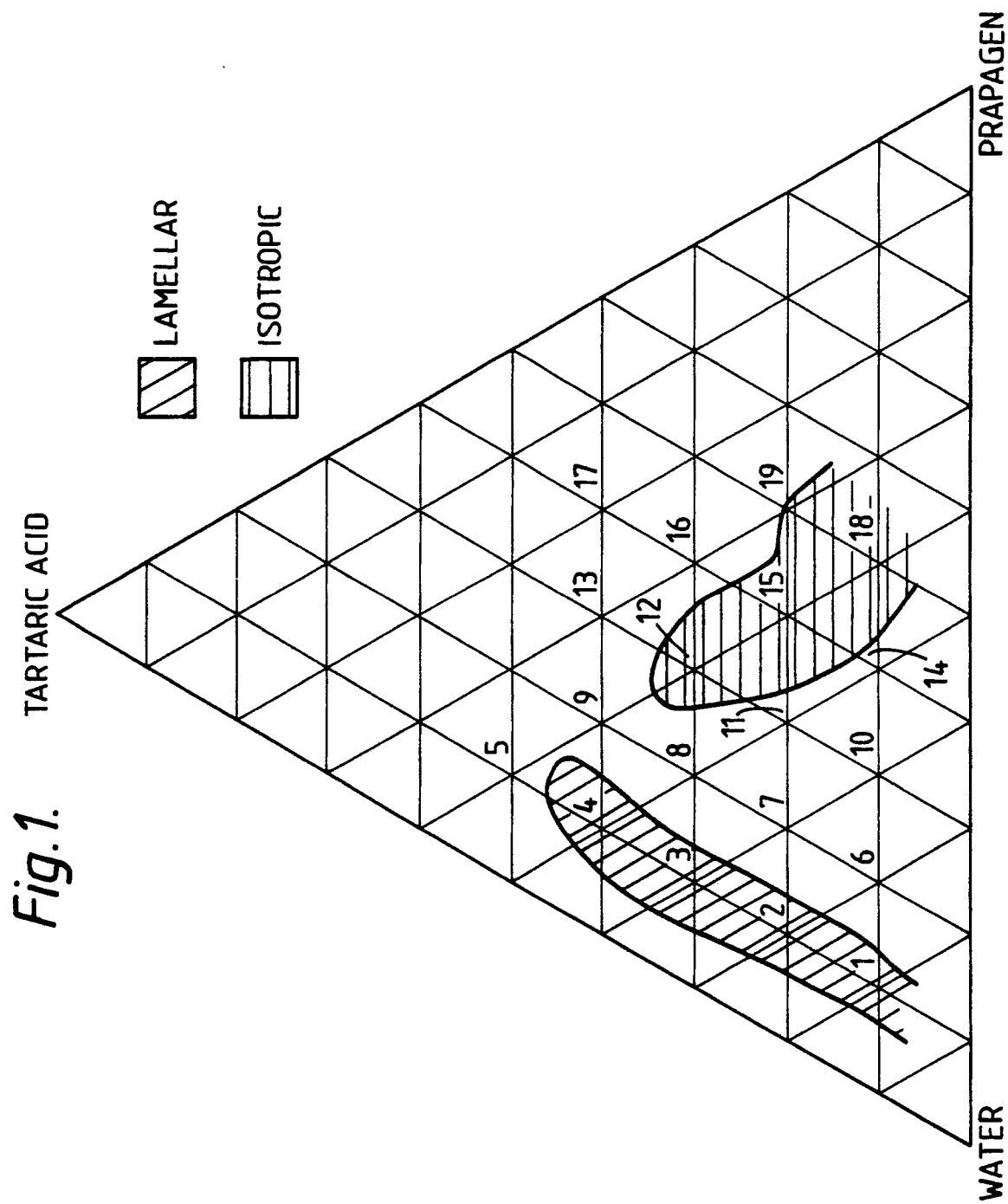
## EXAMPLE IV

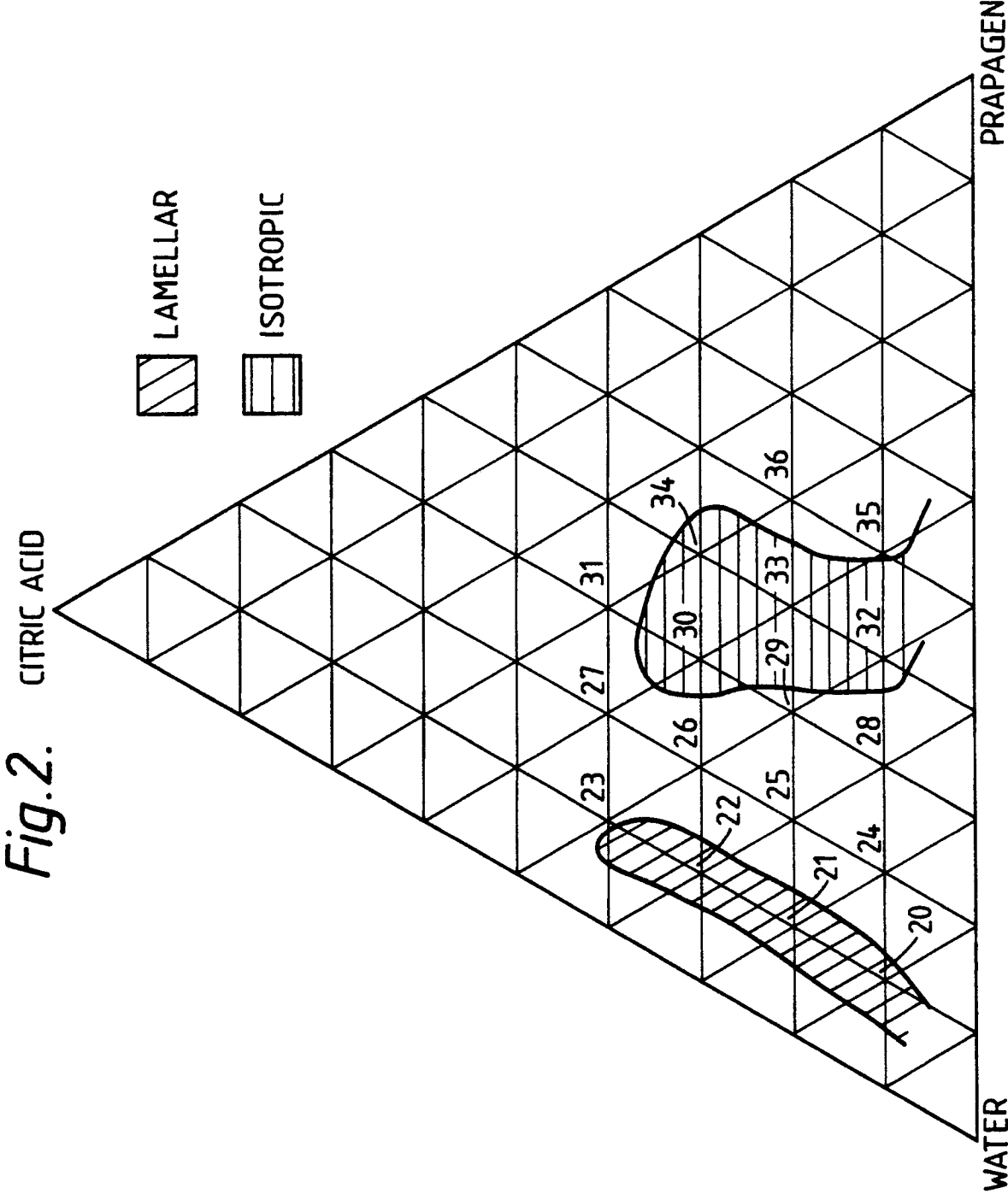
By replacing tartaric acid by propionic acid isotropic/clear compositions can be formulated even at levels of acid as low as 5%. Typical formulations are:

	Formulation % by weight			
	A	B	C	D
Arquad 2T	40	40	40	40
Isopropylalcohol	0	5	11	0
Propylene glycol	10	10	10	0
Propionic acid	10	10	5	30
Water	balance			

## Claims

1. Isotropic liquid fabric softening composition comprising:
  - (i) at least 20% by weight of a fabric softening material; and
  - (ii) at least 5% by weight of an organic acid.
2. Composition according to claim 1, having a clear appearance.
3. Composition according to one or more of the preceding claims, wherein the fabric softening material comprises a cationic fabric softening material.
4. Composition according to one or more of the preceding claims, wherein the organic acid is a carboxylic acid having a total number of carbon atoms of 8 or less.
5. Composition according to claim 4, wherein the organic acid has a total number of carbon atoms of 4 or less.
6. Composition according to claim 4, wherein the organic acid comprises propionic acid.
7. Composition according to claim 4, wherein the acid comprises lactic acid or acetic acid.
8. Composition according to one or more of the preceding claims, comprising at least 10% by weight of organic acid.
9. Composition according to one or more of the preceding claims comprising at least 20% by weight of water.
10. Method of treating fabric, comprising the contacting of fabrics with an aqueous liquor comprising from 0.01 to 10g/l of a composition according to one or more of the preceding claims.







European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number

EP 90 30 6600

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	US-A-4 060 505 (J.D. CIKO et al.) * column 2, lines 30-41; claims 1,3,5,7 *	1,3-5,7 -10	C 11 D 3/00 C 11 D 3/20 C 11 D 1/62
X	--- US-A-3 984 335 (J.D. CIKO et al.) * column 2, lines 18-27; column 3, lines 32-41; claims 1,3,5,6 *	1,3-5,7 -10	
X,P	--- WO-A-8 911 522 (HENKEL) * page 2, lines 19-30; page 6, lines 5-18; claims 1,3 *	1-3	
X	--- EP-A-0 043 622 (THE PROCTER & GAMBLE) * claims 1-3,6,8 *	1,2	
X	--- EP-A-0 123 999 (HENKEL) * claims 1-8 *	1,2,9	
X,D	--- DE-A-3 312 328 (BENCKISER) * claims 1-10 *	1,2	
A	--- US-A-3 954 630 (P. RAMACHANDRAN) * claims *	1-10	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	--- US-A-4 832 856 (H. RUTZEN) * claims * -----	1-10	C 11 D 3/00 C 11 D 1/62
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 25-09-1990	Examiner PELLI-WABLAT B
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			