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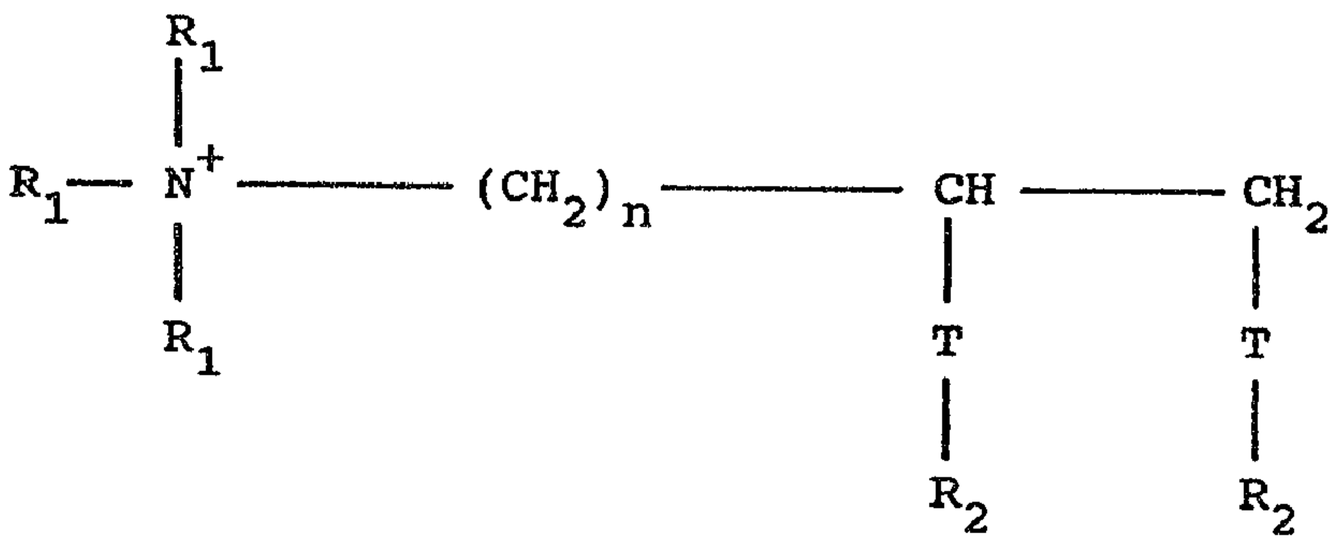
(54) **ASSOUPLISSEUR DE TISSUS**

(54) **FABRIC SOFTENING COMPOSITION**

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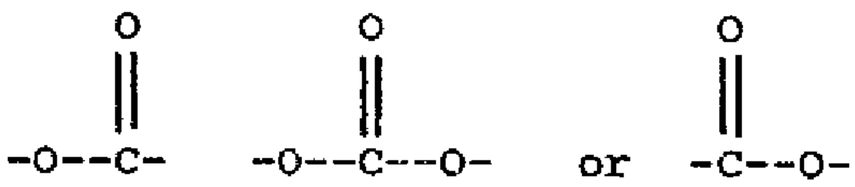


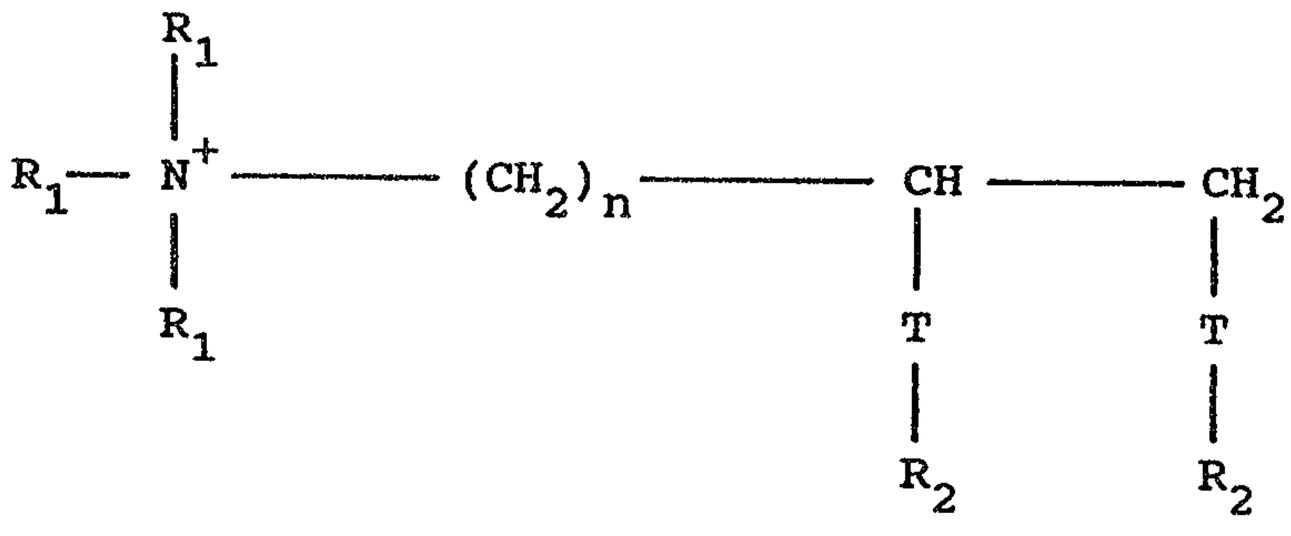

II III IV

(57) A fabric softening composition which comprises: (i) an ester-linked quaternary ammonium material of the formula: (see formula I) wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hydroxyalkyl groups; each $R_2$ group is independently selected from C8-28 alkyl or alkenyl groups; T is (see formula II) (see formula III) or (see formula IV); and n is an integer from 0-5; and (ii) an extender material selected from: (a) quaternary ammonium compounds, comprising one long alkyl or alkenyl group; (b) predominantly linear nonionic materials; or (c) mixtures thereof; said composition being obtainable by heating a mixture comprising the materials (i) and (ii) to a temperature of above 40 °C, followed by dispersing the mix into water.

ABSTRACT

A fabric softening composition which comprises:

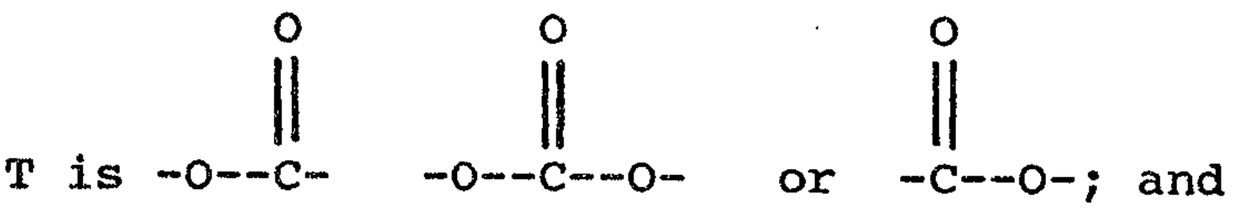
(i) an ester-linked quaternary ammonium material of the formula:



(I)

wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hydroxyalkyl groups;

each $R_2$ group is independently selected from C8-28 alkyl or alkenyl groups;

T is  and

n is an integer from 0-5; and

(ii) an extender material selected from:

(a) quaternary ammonium compounds, comprising one long alkyl or alkenyl group;

(b) predominantly linear nonionic materials; or

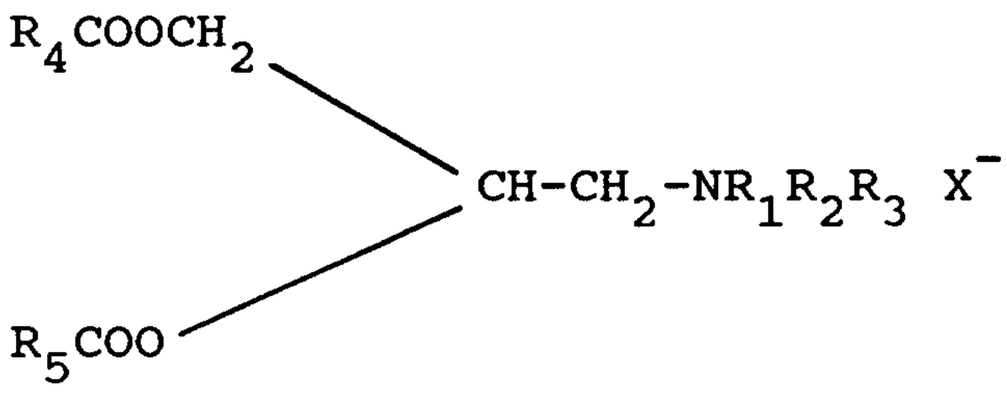
(c) mixtures thereof;

said composition being obtainable by heating a mixture comprising the materials (i) and (ii) to a temperature of above 40°C, followed by dispersing the mix into water.

# FABRIC SOFTENING COMPOSITION

The present invention relates to a fabric softening composition and to a method for its preparation. Specifically the present invention relates to a fabric softening composition for use in the rinse step of a fabric washing process.

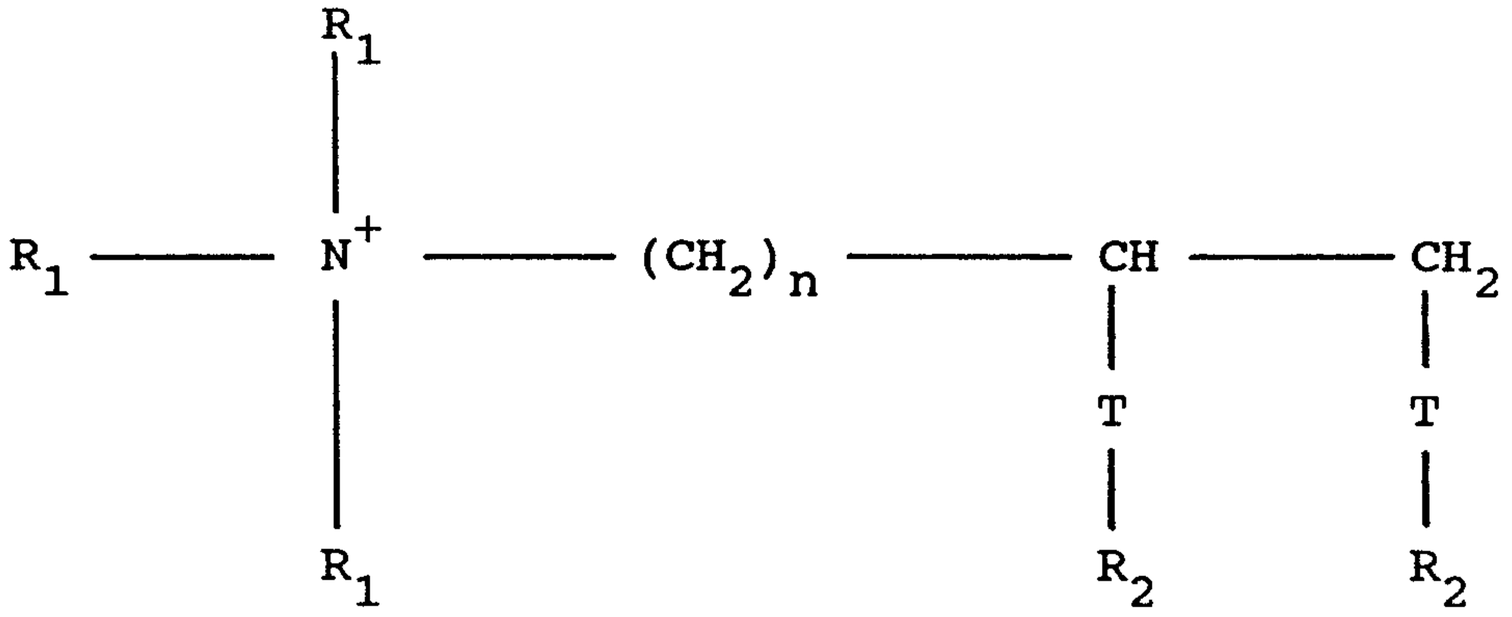
In US 4 137 180 (Lever Brothers Company) cationic diesters of the formula:



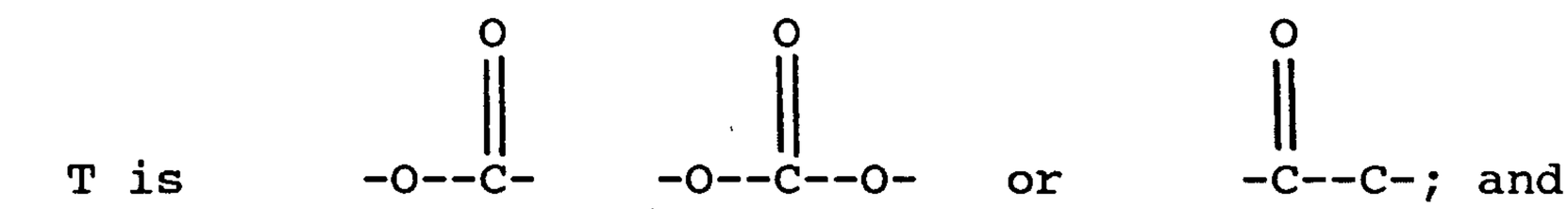
wherein $R_1$, $R_2$ and $R_3$ are each an alkyl or hydroxyalkyl group containing from 1 to 4 carbon atoms, or a benzyl group, $R_4$ and $R_5$ are each alkyl chains containing from 11 to 23 carbon atoms and $X^-$ is a water soluble anion are disclosed.

It has been proposed in EP 239,910 (P&G) to formulate

fabric conditioning compositions comprising ester-linked quaternary ammonium materials of the following formula:



Wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hydroxyalkyl groups; each $R_2$ group is independently selected from C12-24 alkyl or alkenyl groups;

T is  and

n is an integer from 0-5.

These ester-linked quaternary ammonium materials are particularly preferred for use in fabric conditioners because they are more biodegradable than conventional quaternary ammonium materials.

In the past difficulty was experienced in preparing fabric softening compositions containing solely the above mentioned softening material at levels above 0.1% by weight by the known method of pre-melting the material followed by dispersing the melt into hot water while stirring because upon melting a highly viscous phase was formed which was very difficult to disperse. The

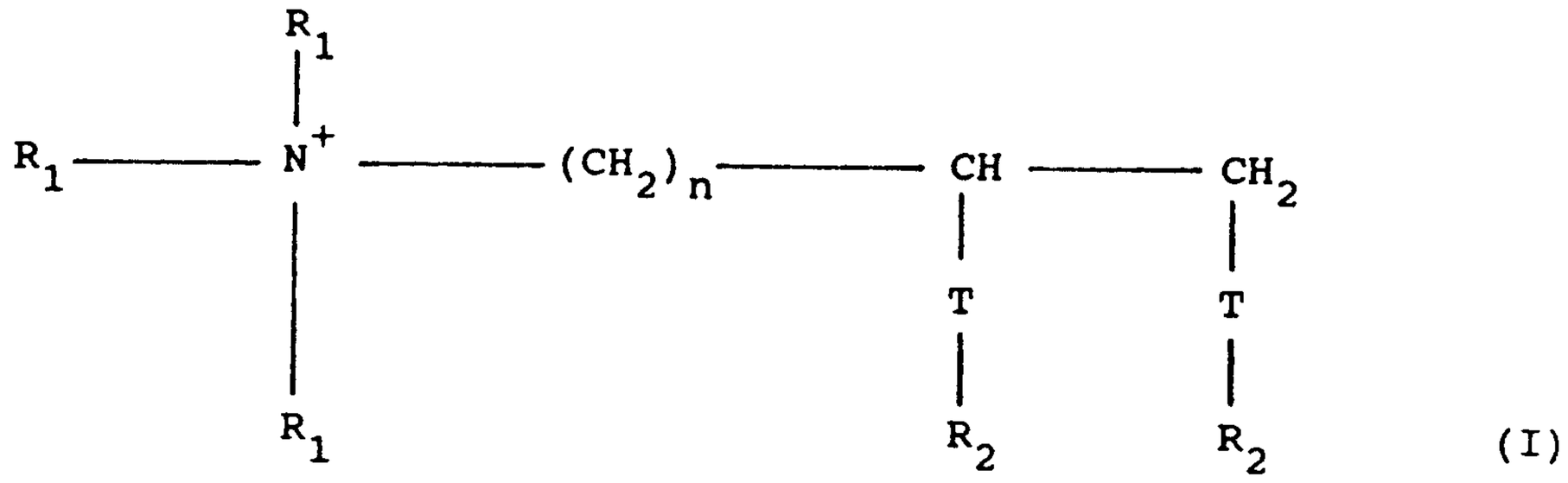
products obtained from such a method were not of satisfactory stability and tended to thicken on storage to an unacceptably high viscosity. Attempts to prepare fabric softening compositions by cold dispersion resulted in inhomogeneous products of unacceptably high viscosity.

It is an object of the present invention to provide fabric softening compositions which comprise the above mentioned biodegradable quaternary ammonium compounds. It is a second object of the invention to provide biodegradable fabric softening compositions which are of acceptable stability. Further objects of the invention are to provide fabric softening compositions which can easily be prepared and which provide adequate softening to fabrics treated therewith.

Surprisingly, it has been found that one or more of the above mentioned problems can be overcome and/or one or more of the above mentioned objects can be met, if the specific ester-linked quaternary ammonium materials as mentioned above are used in combination with one or more specific extender materials.

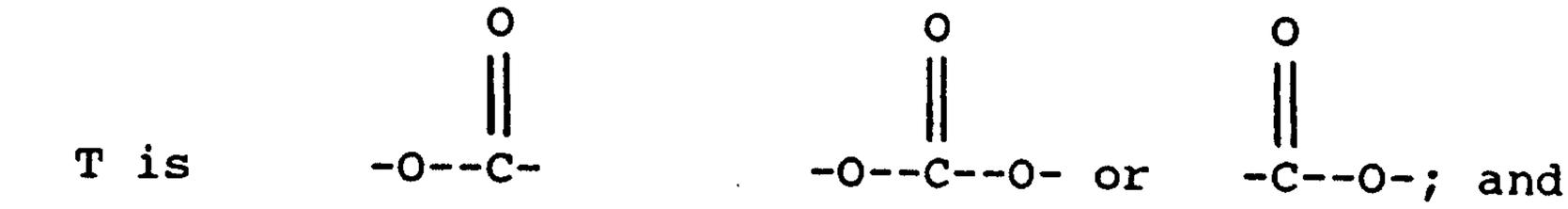
Accordingly the present invention relates to a fabric softening composition which comprises:

(i) at least 1% by weight of an ester-linked quaternary ammonium material of the formula:



(I)

Wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hdyroxyalkyl groups; each $R_2$ group is independently selected from C8-28 alkyl or alkenyl groups;

T is 

n is an integer from 0-5; and

(ii) at least 0.5% by weight of an extender material selected from:

(a) a tertiary or quaternary ammonium compounds, comprising a single $C_8$-$C_{28}$ alkyl or alkenyl group;
(b) predominantly linear nonionic materials; selected from predominantly linear ester materials, predominantly linear $C_8$-$C_{28}$ fatty alcohols, esters of predominantly linear $C_8$-$_{28}$ fatty acids with polyhydric alcohols;
(c) mixtures thereof

said composition being obtainable by heating a mixture comprising the materials (i) and (ii) to a temperature of above 40°C, followed by dispersing the mix into water.

The compositions of the invention may take a variety of forms such as pastes, liquids etc and also they may be impregnated onto substrates for example for use in tumble dryers. Preferably compositions of the invention are liquids, comprising an aqueous base, wherein the active ingredients are dispersed.

Suitable ester-linked quaternary ammonium materials (i) and their method of preparation are for example described in US 4 137 180 (LEVER BROTHERS). A preferred material is 1,2-ditallow oxy-3-trimethyl ammoniumpropane chloride.

Preferably the level of ester-linked quaternary ammonium material (i) is at least 3% by weight of the composition, especially interesting are compositions of high active level, e.g. which comprise more than 8% of the ester linked quaternary ammonium compound. The level of ester-linked quaternary ammonium materials (i) preferably is less than 70% by weight of the composition, more preferred less than 50%, typically from 3 to 30% by weight of the composition.

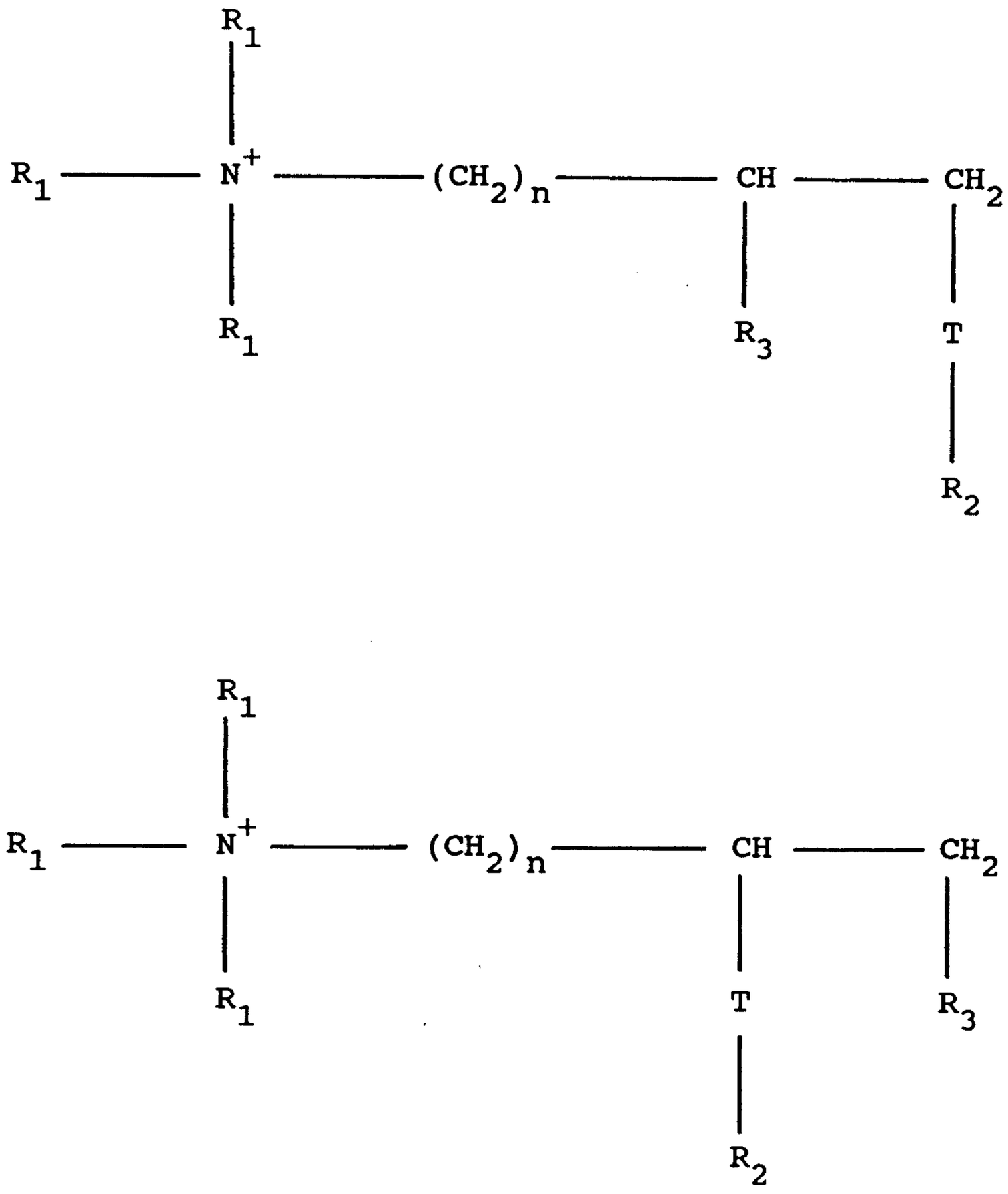
The extender material (ii) can be selected from mono-long alkyl or alkenyl tertiary or quaternary ammonium compounds and predominantly linear nonionic materials or mixtures thereof. In this specification the expression predominantly linear nonionic material refers to materials having a molecular backbone whereto no- or only a small number of side groups are attached. Preferably the molecular backbone constitutes more than 75% by weight of the molecule, more preferred more than 90%. In selecting the extender materials for use in a composition of the

invention it is preferred only the use materials which are of acceptable biodegradability.

Especially useful extender materials are:

(A) Tertiary amines containing one C 8-28 alkyl or alkenyl group and two C1-4 alkyl, alkenyl or hydroxyalkyl groups. These materials preferably are used in protonated form.

(B) Ester-linked mono-long alkyl or alkenyl quaternary ammonium compounds, containing one C8-28 alkyl or alkenyl group, connected to the quaternary ammonium molecule via an ester link. Preferred materials within this class are materials of the formula:



wherein $R_1$, $R_2$, T and n are as defined for formula (I) and $R_3$ is a hydrogen or hydroxy group. Of these materials, those are preferred, wherein $R_3$ is an hydroxy-group. These materials show a surprisingly good softening performance.

(C) Predominantly linear nonionic ester materials, for example esters of C8-28 fatty acids and C1-30 monohydric alcohols. Especially preferred is the use of ethylstearate, lanolin and Jojoba oil.

(D) Predominantly linear C8-28 fatty alcohol materials, C8-28 fatty acid materials or their salts. Of these materials especially the use of free mono-carboxylic fatty acid materials is preferred.

(E) Esters of predominantly linear C8-28 fatty acids with polyhydric alcohols. Preferred esters are glycerol monostearate, sorbitan monostearate and the monoesters of polyethyleneglycol of molecular weight between 200 and 2000.

Preferably the level of extender material is more than 0.2%, especially preferred are systems comprising more than 0.5% of extender material. The level of extender material will usually not exceed 50% by weight of the composition, more preferred the level is less than 30%, typically from 0.5 to 10% by weight of the composition.

The weight ratio of ester-linked quaternary ammonium materials (i) to extender materials (ii) is preferably from 20:1 to 1:10, more preferred from 10:1 to 1:2, especially preferred from 9:1 to 1:1.

Compositions of the invention are obtainable by heating a mixture of the materials (i) and (ii) to a temperature of above 40°C followed by dispersing the mix into water. Preferably the materials are heated to a temperature of above 50°C and dispersed in water of elevated temperature. Compositions made by this method show a surprising good stability and no or only a small viscosity increase upon storage. It is believed that this increased stability may be caused by the particularly good homogeneity of the products obtained by using this method.

Therefore particular preferred products in accordance with the present invention comprise the above mentioned two materials (i) and (ii) dispersed in water, wherein the mean particle diameter of the dispersed softener phase is less than 10$\mu$m, preferably less than 7$\mu$m, especially preferred less than 3$\mu$m. Such small particle dispersion are often referred to as microdispersions.

The compositions of the invention preferably have a pH of more than 2.0. Preferably the pH of the composition is less than 8.0, more preferably less than 5.0. Especially preferred are pH values in the range of from 2.5 to 4.0.

Compositions of the invention may in addition to the above described two ingredients also comprise other active ingredients such as further fabric conditioning materials.

In selecting the optional ingredients it is preferred to use materials which do not substantially affect the biodegradability of the composition. Therefore it is preferred to minimize the level of non-biodegradable or less bio-degradable materials.

Preferably compositions of the invention do not comprise large amounts of conventional non-ester linked quaternary ammonium compounds, preferably the compositions of the invention are substantially free of these quaternary ammonium compounds.

Softening materials which may be incorporated in a composition of the invention are amines, such as for instance described in EP 326213 published August 2, 1989, or biodegradable amphoteric softener materials such as for instance described in EP 326213.

Preferably the amount of active materials other than the above mentioned ester-linked materials and extender materials is relatively low, i.e. the weight ratio of other softener materials to the total of the two essential softener components is preferably less than 2:1, more preferred less than 1:1, most preferred less than 0.5:1.

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 such as strong or weak acid e.g. HCl, $H_2SO_4$, phosphoric, benzoic or citric acids, rewetting agents, viscosity modifiers such as electrolutes, for example calcium chloride, antigelling agents, perfumes, perfume carriers, fluorescers, colourants, hydrotropes, antifoaming agents, antiredeposition agents, enzymes, optical brightening agents, opacifiers, stabilisers such as guar gum and polyethylene glycol, emulsifiers, anti-shrinking agents, 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, preferably are present at levels up to 5% by weight of the composition.

A preferred method to prepare compositions according to the invention is to form a molten mixture of the ester-linked quaternary ammonium materials (i) and the extender materials (ii) eventually in combination with other softening materials and/or other optional

ingredients, and adding this premix to water under stirring to form a dispersion and thereafter adding any other optional ingredients.

In use, the fabric conditioning composition of the invention may be added to a large volume of water to form a liquor with which the fabrics to be treated are contacted. Generally, the total concentration of the ester linked quaternary ammonium compounds (i) and the extender compound in this liquor will be between about 30ppm and 1000ppm. The weight ratio of the fabrics to liquor will generally be between 1:1 and 1:20.

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

EXAMPLE I

The following compositions were prepared by heating the active ingredients to a temperature of 60°C and dispersing the premelt into water of 60°C under stirring. The resulting compositions according to the invention were homogeneous dispersions, wherein the mean diameter of the dispersed active particles was less than 1$\mu$m.

| INGREDIENT (%wt) | COMPOSITION | | | |
|---|---|---|---|---|
| | A | B | C | D |
| DTTMAPC[1]) | 3.5 | 4.5 | 2.45 | -- |
| THTMPAC[2]) | 1.5 | 0.5 | 1.05 | -- |
| IPA[3]) | 0.5 | 0.8 | 0.5 | -- |
| *Sokolan SC9327X[4]) | -- | -- | 1.5 | -- |
| *Stepantex VRH 90[5]) | -- | -- | -- | 5.55 |
| demin water | ---------- to 100% ---------- | | | |

[1]) 1,2 ditallow oxy trimethyl ammonium propane chloride
[2]) 1-tallow oxy 2-hydroxy trimethyl ammoniumpropane chloride
[3]) isopropylalcohol
[4]) stearyl ester of choline
[5]) commercially available biodegradable softener 90% active, 10% IPA.

2g of the formulations A-D was added to 1 liter of tap water of ambient temperature containing 0.001% by weight of sodium alkyl benzene sulphonate to simulate the carry over of anionic detergent active from the wash. 800ml of the obtained solution was put in a tergotometer pot and four pieces of terry towel (40g total weight) were added. The cloths were treated for 5 minutes at 60rpm, spin dried and line dried. The dried fabrics were assessed for softness by an expert panel using a Round Robin test protocol.

The order of preference for the above mentioned compositions was:

B > C > A > D

*denotes trade mark

These results show a surprisingly good softening effect for compositions of the present invention (A-C) compared to currently available biodegradable softener actives (D).

EXAMPLE 2

The following compositions were prepared as in example 1.

| INGREDIENT (wt%) | Composition A | B | C | D | E |
|---|---|---|---|---|---|
| DTTMAPC | 9.0 | 10.5 | 3.5 | 8.0 | 12.0 |
| THTMAPC | 1.0 | 4.5 | -- | -- | -- |
| Sokolan SC | -- | -- | 1.5 | -- | -- |
| Tallow fatty acid | -- | -- | -- | 2.0 | -- |
| Lanolin (ex BP) | -- | -- | -- | -- | 3.0 |
| IPA | 1.5 | 2.0 | 0.5 | 1.5 | 2.0 |
| Perfume | 0.4 | 0.6 | 0.2 | 0.4 | 0.6 |
| demin water | ---------- to 100% ---------- | | | | |

EXAMPLE 3

The following compositions were prepared as in example 1.

| | COMPOSITION | | | | |
|---|---|---|---|---|---|
| INGREDIENT (%wt) | A | B | C | D | E |
| DTTMAPC | 9.0 | 9.0 | 6.0 | 6.0 | 7.0 |
| THTMAPC | 1.0 | 1.0 | -- | -- | -- |
| *Armeen M2HT[3] | 3.5 | -- | -- | -- | -- |
| Amine [1]) | -- | 3.5 | -- | -- | -- |
| octadecanol | -- | -- | 1.0 | -- | -- |
| lanolin alcohol | -- | -- | -- | 1.0 | -- |
| glycerolmonostearate | -- | -- | -- | -- | 3.0 |
| IMS[2]) | -- | -- | -- | -- | 1.4 |
| IPA | 1.5 | 1.5 | 1.2 | 1.2 | -- |
| Perfume | 0.5 | 0.5 | 0.25 | 0.25 | 0.25 |
| Water | ---------- to 100% ---------- | | | | |

[1]) 1-tallowamidoethyl-2-tallowimidazoline (protonated).
[2]) industrial methylated spirit
[3]) methyl di hardened tallow amine (protonated)

EXAMPLE 4

The following compositions were prepared as in example 1.

*denotes trade mark

| INGREDIENT | A | B | C | D | E |
|---|---|---|---|---|---|
| DTTMAPC | 7.0 | 7.0 | 12.0 | 12.0 | 12.0 |
| sorbitan ester | 3.0 | -- | -- | -- | -- |
| PEG 400* monostearate | | 3.0 | -- | -- | -- |
| IMS | 1.4 | 1.4 | -- | -- | -- |
| ethyl stearate | -- | -- | 1.5 | -- | -- |
| octadecyl acetate | -- | -- | -- | 1.5 | -- |
| Armeen DMHT[1]) | -- | -- | -- | -- | 3.0 |
| IPA | -- | -- | 1.8 | 1.8 | 1.8 |
| Perfume | 0.25 | 0.25 | 0.5 | 0.5 | 0.5 |
| Demin water | ---------- to 100% ---------- | | | | |

[1]) dimethyl hardened tallow amine (protonated).

EXAMPLE 5

The following compositions were prepared as in Example 1.

| INGREDIENT | A | B | C |
|---|---|---|---|
| DTTMAPC | 12.15 | 11.44 | 4.76 |
| THTMAPC | 0.60 | 0.56 | 0.24 |
| HTFA 1 | 2.25 | 3.00 | |
| Lanolin (et BP) | | | 6.20 |
| Glycerol monostearate | | | 3.80 |
| Dobanol* 45-11[2] | | | 1.09 |

[1]) hardened tallow fatty acid

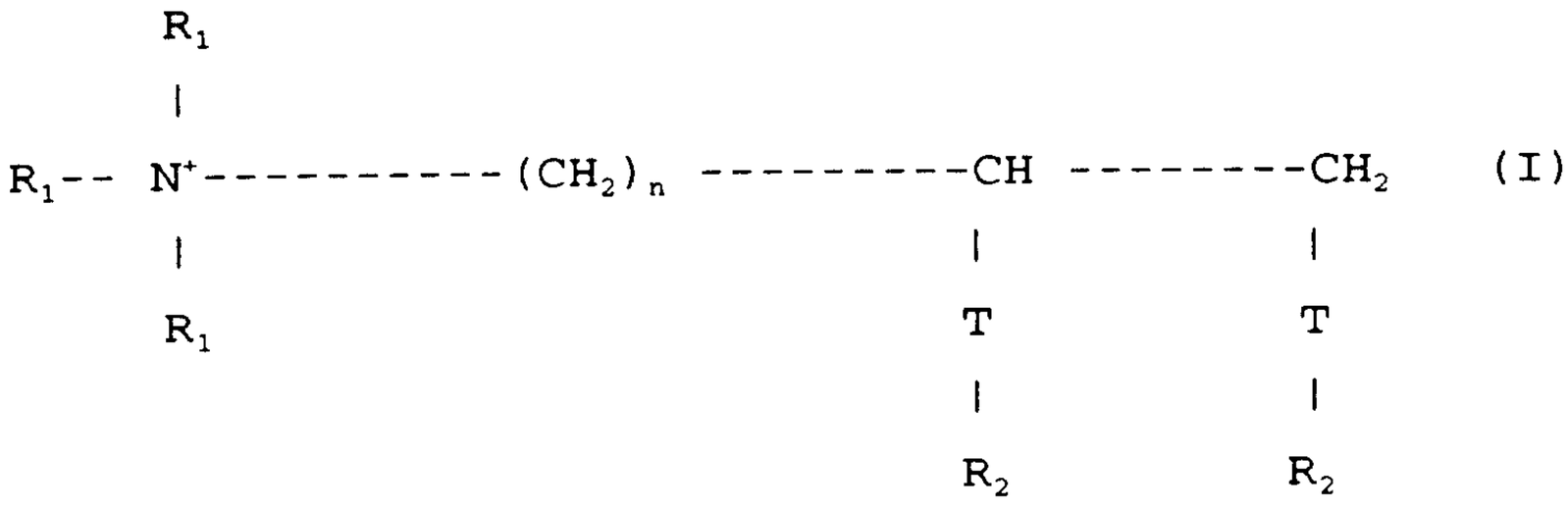
[2]) $C_{14-15}$ alcohol ethoxylate with 11 moles of ethylene oxide.

*denotes trade mark

**THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:**

1. A fabric softening composition comprising

(i) at least 1% by weight of an ester-linked quaternary ammonium material of the formula:



(I)

wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hydroxyalkyl groups; each $R_2$ group is independently selected from $C_{8-28}$ alkyl or alkenyl groups;

T is  and

n is an integer from 0-5; and

(ii) at least 0.5% by weight of an extender material selected from:

(a) a tertiary or quaternary ammonium compounds, comprising a single $C_8$-$C_{28}$ alkyl or alkenyl group;

(b) predominantly linear nonionic materials; selected from predominantly linear ester materials, predominantly linear $C_8$-$C_{28}$ fatty alcohols, esters of predominantly linear $C_8$-$_{28}$ fatty acids with polyhydric alcohols;

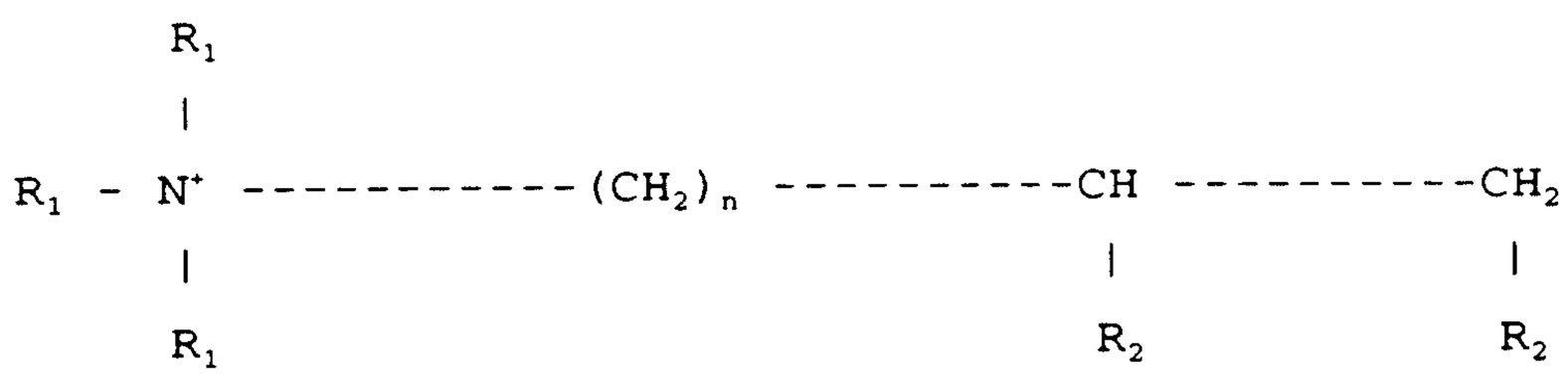
B

(c) mixtures thereof

said composition being obtainable by heating a mixture comprising the materials (i) and (ii) to a temperature of above 40°C, followed by dispersing the mix into water.

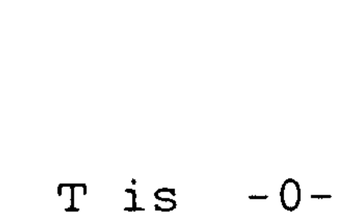
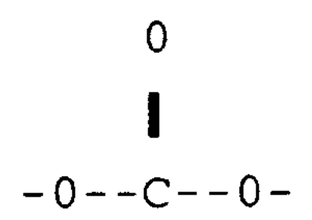
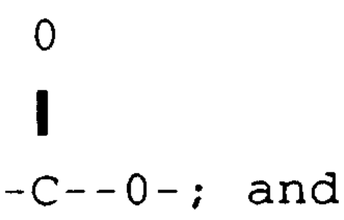
2. A fabric softening composition which comprises a dispersion in water of:

(i) at least 1% by weight of an ester-linked quaternary ammonium material of the formula:



wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hydroxyalkyl groups;

each $R_2$ group is independently selected from $C_{8-28}$ alkyl or alkenyl groups;

T is  or  or  ; and

n is an integer from 0-5; and

(ii) at least 0.1% by weight of an extender material selected from:

(a) a tertary or quaternary ammonium compounds, comprising one $C_{8-28}$ alkyl or alkenyl group;

(b) predominantly linear nonionic materials selected from predominantly linear ester materials, predominantly linear $C_8$-$C_{28}$ fatty alcohol, esters of predominantly linear $C_{8-28}$ fatty acids with polyhydric alcohols;

(c) mixtures thereof;

said dispersion having mean particle diameter of less than 10 μm.

3. A fabric softening composition as claimed in claim 1 comprising at least 3% by weight of the ester-linked quaternary ammonium material of formula (I).

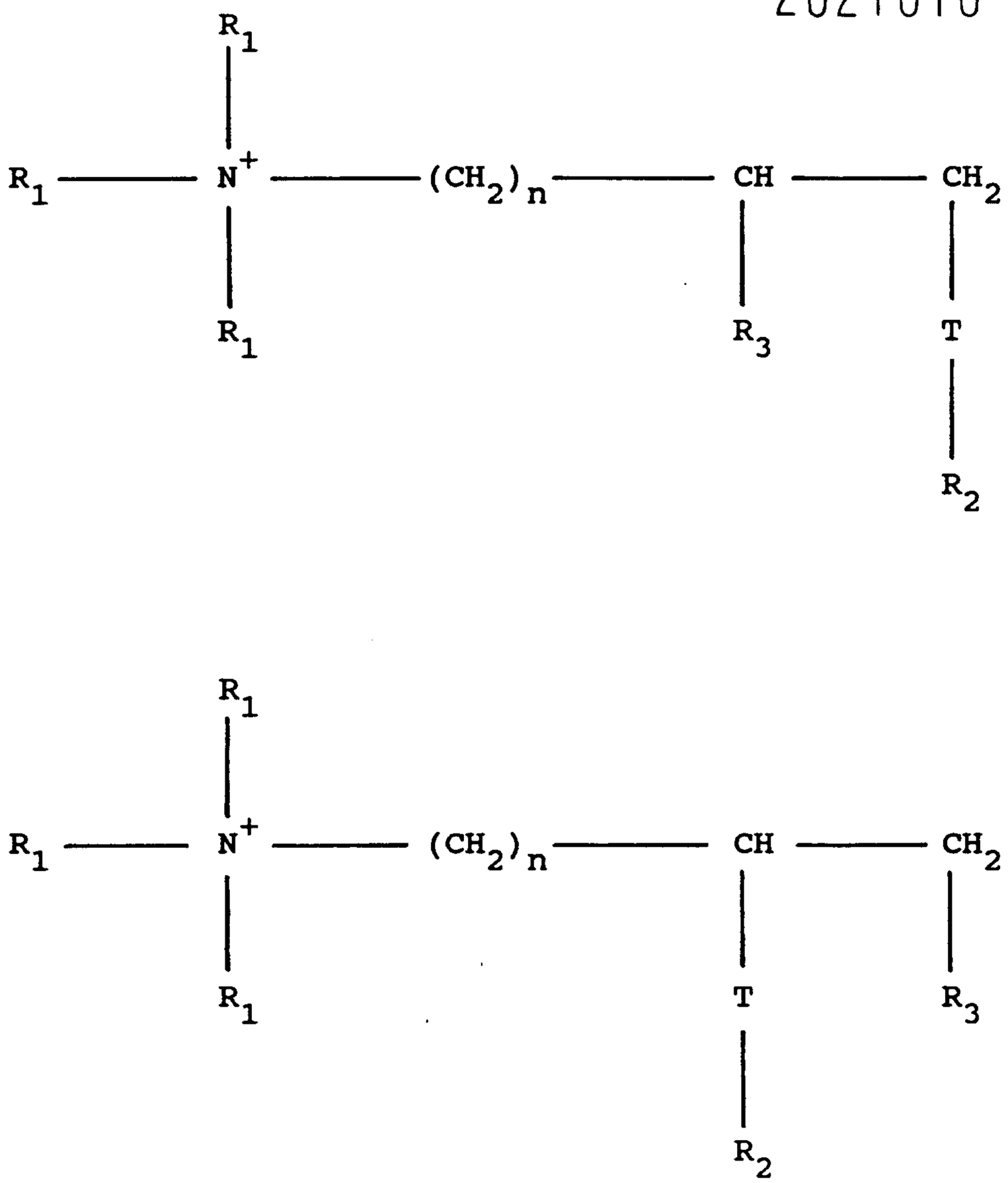
4. A fabric softening composition as claimed in claim 1 comprising at least 8% by weight of the ester-linked quaternary ammonium material of formula (I).

5. A fabric softening composition as claimed in claim 1 comprising from 3 to 30% by weight of an ester-linked quaternary ammonium material of formula (I).

6. A fabric softening composition as claimed in any preceding claim comprising as an extender material selected from predominantly linear ester materials, predominantly linear $C_8$-$C_{28}$ fatty alcohol, esters of predominantly linear $C_{8-28}$ fatty acids with polyhydric alcohols; a quaternary ammonium compound comprising one long alkyl or alkenyl group connected via an ester link.

7. A fabric softening composition as claimed in any preceding claim comprising as an extender material an ester-linked quaternary ammonium material selected from materials of formula (II) and (III) as follows:

C



(II)



(III)

wherein each $R_1$ group is independently selected from $C_{1-4}$ alkyl, alkenyl or hydroxyalkyl groups; each $R_2$ group is independently selected from $C_{8-28}$ alkyl or alkenyl groups;

T is $-\overset{O}{\overset{|}{O}}-C-$, $O-\overset{O}{\overset{|}{C}}-O$ or $-C-\overset{O}{\overset{|}{O}}-$; and

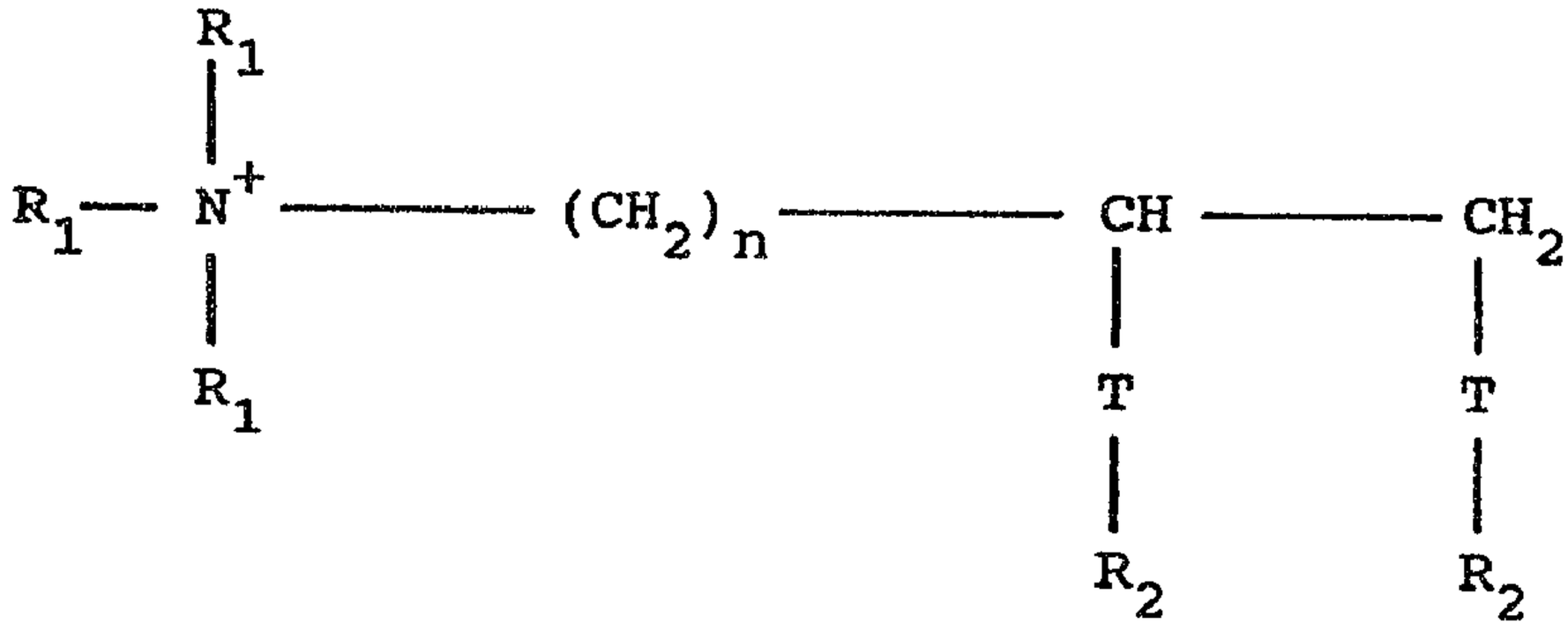
n is an integer from 0-5; and

wherein $R_3$ is a hydrogen or hydroxy group.

8. A fabric softening composition as claimed in any preceding claim comprising at least 0.5% of the extender material.

9. A fabric softening composition as claimed in any preceding claim comprising from 0.5% to 10% of the extender material.

10. A fabric softening composition as claimed in any preceding claim having a pH in the range from 2 to 8.



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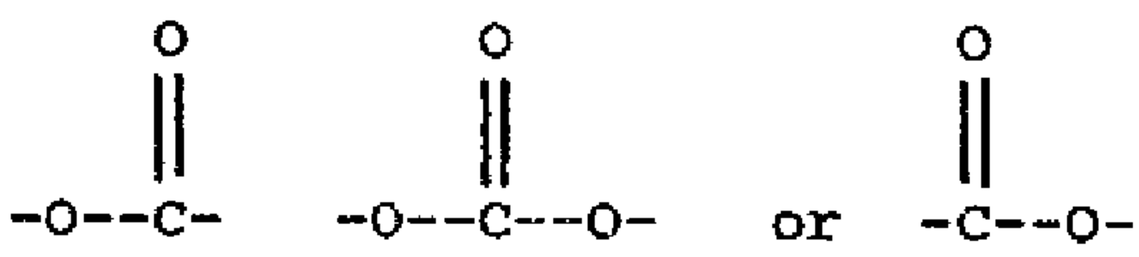

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