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(54) Titre : PROCEDE DE FABRICATION D'ASSOUPPLISSEURS DE TISSU FAISANT APPEL A UNE FAIBLE QUANTITE
DE SOLVANTS ET ELIMINANTS LES REACTIONS SECONDAIRES

(54) Title: PROCESS FOR MAKING HIGH SOLIDS FABRIC SOFTENERS USING LOW AMOUNTS OF SOLVENTS
AND ELIMINATING SIDE REACTIONS

(57) **Abrégé/Abstract:**

A process is disclosed for making substantially solvent free quaternary ammonium compounds using fatty acids, amines and quaternizing agents without side reactions that would produce esters of fatty acids. A relatively small amount of a water-alcohol solvent is used in the quaternizing reaction after which a fatty acid is added and the solvent removed by sparging and/or vacuum.



ABSTRACT OF THE DISCLOSURE

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A process is disclosed for making substantially solvent free quaternary ammonium compounds using fatty acids, amines and quaternizing agents without side reactions that would produce esters of fatty acids. A relatively small amount of a water-alcohol solvent is used in the quaternizing reaction after which a fatty acid is added and the solvent removed by sparging and/or vacuum.

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PROCESS FOR MAKING HIGH SOLIDS FABRIC SOFTENERS
USING LOW AMOUNTS OF SOLVENTS AND ELIMINATING SIDE REACTIONS

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The invention relates to a process for making substantially solvent free quaternary ammonium compounds using fatty acids, amines and quaternizing agents without
10 side reactions such as those reactions that would produce esters of fatty acids. A more fluid product is obtained than the ester-containing softener blends used for dryer softener sheets. Ordinarily quaternization of the amine takes place in the presence of the fatty acid and the quaternizing agent,
15 resulting in the formation of the ester of the fatty acid which makes the resultant product viscous and difficult to handle. By employing the method of the present invention, in which a relatively small amount of a water-alcohol solvent is used in the quaternizing reaction after which a fatty acid is
20 added and the solvent removed, a product is obtained which is ready to be flaked or powdered and can be used as a fabric softener, lubricant, hair conditioner, in the manufacture of aqueous dispersions and the like.

The preparation of quaternary ammonium compounds is
25 usually conducted in stainless steel or glass-lined equipment to which a tertiary amine is charged and a solvent.

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Flammable solvents such as isopropyl alcohol are generally
1 used although mixtures of isopropyl alcohol and water
sometimes are employed or water alone is used. Flammable
solvents are undesirable because they are a fire hazard and
special handling procedures are required when they are used.
5 In many applications, these solvents have to be stripped from
the mixture when the reaction is completed because the
ultimate use of the product is in a solventless or solid
form.

After the reactants are loaded into the reactor they
10 are heated to a temperature of about 50 to about 100°C after
which a quaternizing reagent is added. In some instances an
exotherm is produced as a result of the quaternizing reaction
and the reactor and its contents have to be cooled. The rate
of addition of the quaternizing agent can also be controlled
15 in order to minimize or eliminate the exotherm. As noted
previously, in some instances, the solvent, if any is
employed in the quaternizing reaction is stripped from the
quaternary ammonium compound obtained since some commercial
uses for the compounds are in solventless systems.
20 Additionally, bulk shipments of quaternary ammonium compounds
with solvents adds to transportation costs which is another
reason to remove the solvents.

It is also known in the prior art that when water
alone is used as the solvent a gelatinous sticky mass is
25 obtained from which the water is difficult to remove. When
alcohol is used as the solvent for the quaternizing reaction
the products obtained are not as difficult to handle as when
water is used, however, when used, it presents not only
a fire hazard but also an environmental problem if released
30 to the air. The combinations of water and alcohol have been
used for several years in quaternization reactions and this
solvent mixture generally is removed by either sparging

and/or by applying a vacuum to the quaternary ammonium
1 compound thus produced which contains the solvents. These
processes, although commercially employed are not entirely
satisfactory primarily because the mixture of solvents with
the quaternary ammonium compound and other reactants and
5 by-products is viscous to the point where solvent removal can
be difficult.

The largest use for quaternary ammonium compounds
is as a fabric softener and presently accounts for more than
about three quarters of the total market for these material.
10 Some fabric softeners are supplied as a liquid dispersion of
from about 3% to about 10% by weight of the quaternary
ammonium compound which is adapted to be added during the
rinse cycle of a commercial or home laundering operation.
Another significant fabric softening application is the
15 utilization of quaternary ammonium compounds in combination
with a substrate such as a nonwoven fabric or a polymeric
foam such as a polyurethane foam, this substrate so treated
being added to a fabric dryer such as a clothes dryer while
the fabric or clothes are still damp. The quaternary
20 ammonium compound is formulated usually with a fatty acid
or ester which promotes the transfer of the quaternary
ammonium compound from the nonwoven or porous polymeric
substrate to the fabric or clothes.

Quaternary ammonium compounds are now being added
25 to both solid and liquid laundry detergent compositions so
that the quaternary ammonium compound can be incorporated as
a fabric softener during the wash cycle of fabrics or
clothes. The most successfully utilized quaternary ammonium
compounds in this last respect are the dimethyl
30 (dihydrogenated tallow) ammonium chlorides or methyl
sulfates. Other quaternary ammonium compounds such as
imidazolines and amidoamine quaternaries are also used.

Quaternary ammonium compounds are also used to
1 manufacture organomodified clays which may be added to
drilling muds utilized in drilling oil wells, the
organomodified clay providing improved lubrication and
rheological properties of the drilling muds. These
5 organoclays are also employed as thixotropic agents in
plastisols, organosols, paints and other protective coatings,
grease additives, foundry additives, cosmetics, resins and
printing inks. The most common quaternary ammonium compounds
employed in this regard are methyldi(hydrogenated tallow)
10 benzylammonium chloride, dimethyldi(hydrogenated tallow)
ammonium chloride and dimethyl(hydrogenated tallow)
benzylammonium chloride.

Quaternary ammonium compounds are also employed as
disinfectants, e.g. bactericides or bacteristats, the most
15 common of which is the quaternary ammonium compound of
benzylchloride and a dimethylalkyl-amine, the alkyl group
having from about 12 to about 16 carbon atoms as well as
trimethyl alkyl ammonium chlorides where the alkyl group is a
long chain alkyl such as an octadecyl group. Additionally,
20 dimethyldicoconut-oil fatty ammonium chlorides are also
effective bactericides especially against anaerobic bacteria
which are sulfate reducers that are found in oil wells, these
bacteria causing severe corrosion problems and plugging of
formations which this type of quaternary ammonium compound
25 can minimize or eliminate. Additionally, these quaternary
ammonium compounds effective against anaerobic bacteria are
also effective in removing oil from sand stone formations in
oil wells and provide a two-fold effect of functioning not
only as a bactericide but also in promoting so-called
30 secondary recovery of oil.

An additional use of quaternary compounds is in hair
1 treatment because of the antistatic effects obtained with
such compounds, as well as the increased wetting which
promotes improvements in both wet and dry combing or brushing
and improves luster and feel. The most commonly used
5 quaternary ammonium compounds in this respect are
trimethylalkylammonium chloride, pentaethoxystearylammonium
chloride, dimethylstearylbenzylammonium chloride and
dimethyldialkylammonium chlorides.

The present invention relates to a method for
10 making a mixture of a quaternary ammonium compound and a
fatty acid which is substantially free of solvents, fatty
acid esters and amine salts by reacting an amine and a
quaternizing agent in the presence a relatively small amount
of a solvent based on an alcohol and water to obtain a first
15 mixture of a quaternary ammonium compound, said alcohol and
said water, adding a fatty acid to said first mixture to
obtain a second mixture that is relatively fluid and removing
said alcohol and said water as a vapor to thereby obtain
said quaternary ammonium compound and said fatty acid that is
20 substantially free of solvents, fatty acid esters and amine
salts.

It has been found that a highly functional mixture
of a quaternary ammonium compound, and a fatty acid can be
obtained that is substantially free of fatty acid esters,
25 amine salts and solvents by a quaternization reaction in
which an amine is quaternized by a quaternizing agent in the
presence of relatively small amounts of water and an alcohol
until the quaternization reaction is substantially complete.
A first mixture of a quaternary ammonium compound, alcohol
30 and water is obtained after which a fatty acid is added to
the first mixture to obtain a second mixture that is

relatively fluid. This is followed by removing the alcohol and water as a vapor from the second mixture to thereby obtain a quaternary ammonium compound in combination with such fatty acid, the combination being substantially free of fatty acid esters, amine salts and the water and alcohol.

5 The prior art method uses 3-5 wt% water and 2-3 wt% isopropanol to facilitate pumping and transportation. The process according to the invention uses less than 3% water and less than 2% alcohol (e.g. isopropanol). The fatty acid is added which thins the product whereby solvents may be removed and the product remains fluid at lower temperatures. This allows for lower storage temperatures than quaternaries alone or with the solvent mixtures of the prior art and also lower storage than quaternaries blended with the traditional esters used in the known prior art methods.

15 The tertiary amine employed according to the invention has the formula:



where R, R¹ and R² can be any of the following in combination
 (1) linear or branched chain saturated or unsaturated hydrocarbon groups having up to about 22 carbon atoms;

(2) a lower hydroxy alkyl group;

(3) an alkyl amido alkylene group of the formula:



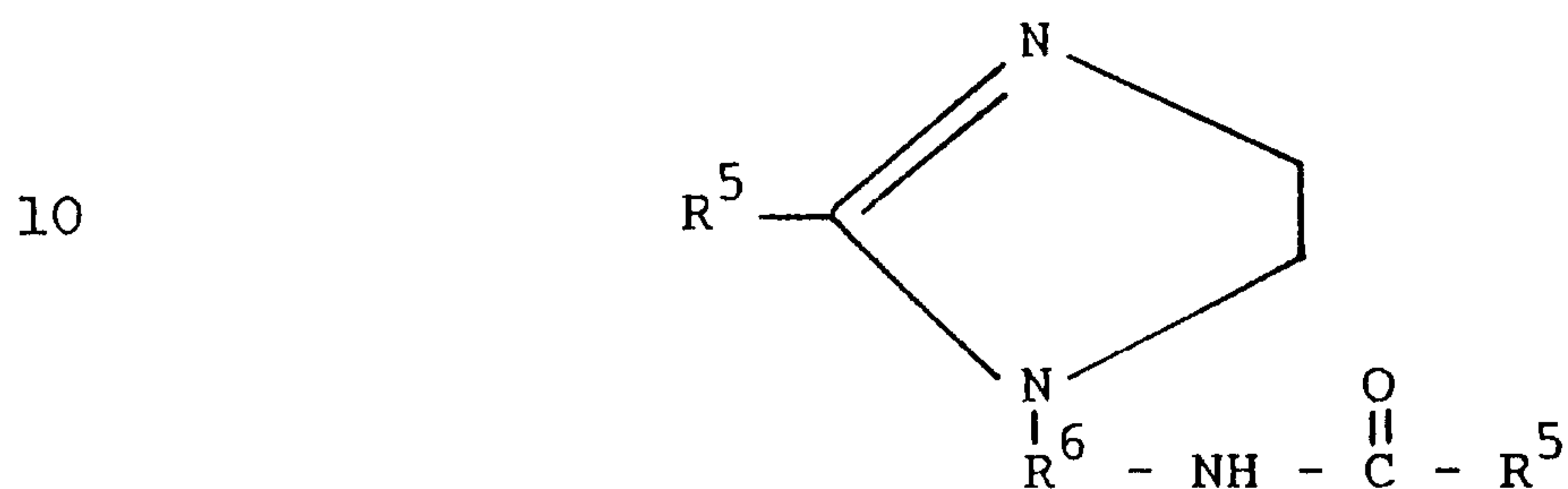
where R⁴ is lower alkylene and R³ is any of R, R¹ or R²;

(4) lower alkoxy group;

1 (5) poly(oxyloweralkylene) group;

so that at least one of R, R¹ or R² is one of said linear or branched chain aliphatic saturated or unsaturated hydrocarbon groups;

5 or said tertiary amine is an imidazoline of the formula:



15 where R⁵ is a linear or branched chain, aliphatic saturated or unsaturated hydrocarbon group having up to about 22 carbon atoms and R⁶ is a lower alkylene group;

said quaternizing agent being known in the art and which will produce a quaternary ammonium compound having an anion A⁻. Generally these anions are those having the formula R⁷_(a-b)X;

where R⁷ is a lower alkyl group or cyclo lower alkyl group such as benzyl, cyclohexylmethyl, tolyl, xylyl, naphthylmethyl, and X is chlorine, iodine, bromine, sulfate, methyl sulfate, carbonate, phosphate, borate group, where (a) is equal to the valence of X and (b) is from 1 to the valence of X and are based on quaternizing agents having the formula R⁷_aX.

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Examples of tertiary amines that can be employed
1 include:

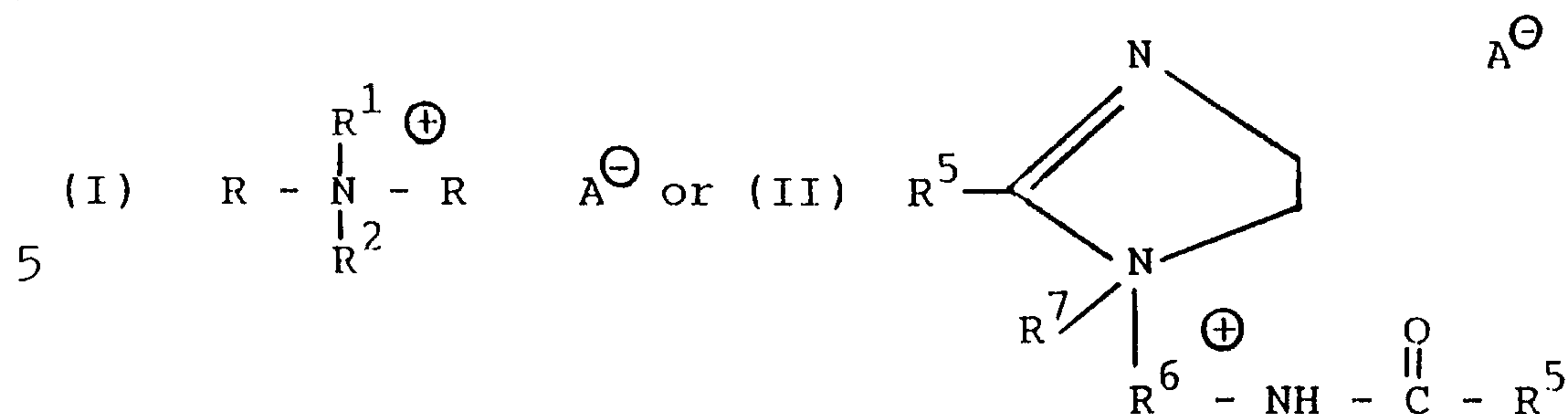
distearyl methyl amine,
dihydrogenated tallow methyl amine,
5 ditallow methyl amine,
dimethyl hydrogenated tallow amine,
dimethyl coco amine,
distearyl ethoxyethyl amine,
stearyl bis-hydroxyethyl amine,
10 stearyl bis (polyethoxy ethanol) amine,
bis (tallowamidoethyl) 2-hydroxyethyl amine,
bis (tallowamidoethyl) 2-hydroxypropyl amine,
1-hydrogenated tallow amido ethyl -2-hydrogenated
tallow imidazoline,
15 1-ethylene bis (2 tallow, 1 methyl, imidazolinium)
dimethyl amino propyl tallow amido-amine and
hydrogenated tallow hydroxyethyl imidazoline,

Examples of Quaternizing Agents that can be
20 employed include:

dimethyl sulfate,
diethyl sulfate,
methyl chloride,
25 methyl bromide and
benzyl chloride.

Said fatty acid is a linear or branched chain
aliphatic saturated or unsaturated fatty acid having from
30 about 12 to about 22 carbon atoms based on coconut oil,
vegetable oils, seed oils, animal fats and fish oils;

The quaternary ammonium compound thus obtained has the formula:



10 where A^- is an anion based on $R^7_{(a-b)X}$ or equivalent
anions known in the quaternary ammonium compound art;
and the fatty acid ester has the formula:



where R⁸ is a linear or a branched chain aliphatic saturated or unsaturated hydrocarbon group having from about 11 to about 21 carbon atoms.

20 As used throughout this specification the terms lower
alkoxy, lower alkylene and lower alkyl are intended to include
compounds having up to about 3 or about 4 carbon atoms
including the various isomeric configurations thereof e.g.
t-butyl, i-butyl, i-propyl and the like and the various
25 mixtures thereof whether such mixtures of such groups contain
components having one, or two, or three or four carbon atoms
or more and also where such groups individually or in
combination are in any of their isomeric forms.

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Equivalent amines (including imidazolines),
1 quaternizing agents, fatty acids and surface active agents
are disclosed in United States Patents to Marschner, U.S.
4,859,456; Caswell et al., U.S. 4,857,213; Demangeon et al.,
U.S. 4,851,141 and Mermelstein et al., U.S. 4,844,824.

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Sparging is generally conducted by passing an inert
gas such as nitrogen, (carbon dioxide etc.) through the
mixture. Combinations of inert gases may also be used.

One or more valved gas inlets are placed in the
10 reactor in which the quaternary ammonium compound is formed,
the openings being below the level of the reactants in the
reactor and preferably at the bottom of the reactor.
Similarly, a pipe or a plurality of pipes can be inserted
through the top of the reactor down to the reactants so that
15 the openings thereof are positioned below the surface of the
reactants preferably towards the bottom of the reactants and
the reactor. The sparging gas is then introduced at a
sufficient flow and pressure so that the sparging gas
introduced into the reactants can sweep the reacting mixture
20 and pull the water and alcohol from the reactants as the gas
exits the reactants at the surface in the form of bubbles.

The solvents may also be removed by vacuum, for
example under reduced pressures of from less than 760 mm to
about 2 mm of mercury and especially from about 200 mm to
25 about 10 mm of mercury either before, during or after
sparging. Any combination or sequences of sparging and
vacuum stripping of solvents may be employed.

The reactants in combination with the fatty acid,
during sparging and vacuum treatment may be held at a
30 temperature from about 50 to about 120°C and especially from
about 80 to about 100°C.

The amine, water and alcohol are added to the reactor and heated to about 140°F prior to the addition of the quaternization reaction and preferably a slight nitrogen purge is initiated at this time and prior to the addition of the quaternizing agent. In conducting the reaction, the amine is present in a slight stoichiometric excess e.g. from about 0.005 to about 5% excess and especially from about 0.01 to about 1% excess and the quaternizing agent is added incrementally so as to maintain a reaction temperature of from about 50 to about 100°C and especially from about 60 to about 90°C. It is standard practice in the art to add the quaternizing agent incrementally since the quaternization reaction is generally exothermic and the temperature of the reaction can be controlled by this type of addition. The progress of the reaction is followed so that when the amount of free amine has stabilized at from about 1 to about 2% by weight (that point in the reaction when the quaternization reaction has been substantially completed) the fatty acid is added. After addition of the fatty acid, the mixture is heated to anywhere from about 80 to about 110°C and especially from about 90 to about 100°C and the solvents (water and alcohol) are removed either by sparging, vacuum stripping or any combination of the two processes in any order, combination or sequence of sparging and vacuum stripping steps.

In conducting the quaternization reaction, sufficient alcohol is added so that there is from about 0.5 to about 5% and especially from 1 to about 3% by weight of alcohol present and water is similarly added so that there is from about 0.25 to about 5% and especially from about 1 to about 4% by weight of water present. The amount of water and

alcohol in terms of weight percent is calculated on the basis
1 of amine, quaternizing agent, water, alcohol and fatty acid
in the reaction mixture. The alcohol is a lower alkanol such
as those alcohols having up to about five carbon atoms,
especially the mono-hydroxy alcohols and includes the various
5 isomers thereof such as isopropyl alcohol, isobutyl alcohol,
t-butyl alcohol and various combinations thereof including
azeotropes of such alcohols or such alcohols and water.

The amount of quaternary ammonium compound and
fatty acid obtained in the mixture after the solvents have
10 been removed from the reaction are from about 10 to about 90
weight percent and especially from about 55 to about 75
weight percent of quaternary ammonium compound and from about
90 to about 10 weight percent and especially from about 25 to
about 45 weight percent by weight of fatty acid. The amine
15 and quaternizing agent are reacted in sufficient ratios and
the fatty acid is added afterwards to the quaternary ammonium
compound thus obtained in sufficient amounts so as to obtain
the foregoing ratios after the water and alcohol have been
removed.

20 The alcohol and water are sufficiently removed from
the quaternary ammonium compound mixed with the fatty acid
so that a mixture of quaternary ammonium compound and fatty
acid is obtained in amounts from about 95% to about 100% and
especially about 98% to about 100% i.e. substantially all of
25 the water and alcohol solvents are removed. Stated otherwise
the mixture may contain anywhere from about 2% to about 5% of
the water and alcohol solvent down to that point where
substantially all or all of the water and alcohol solvent is
removed.

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In one embodiment, the method of the invention is preferably practiced so that the quaternary ammonium compound that is obtained has the structural formula (I) wherein at least one of R, R¹ and R² is a branched chain or linear aliphatic saturated or unsaturated hydrocarbon group having from about 12 to about 22 carbon atoms, preferably saturated, and especially those based on hard tallow acids (i.e. hydrogenated tallow fatty acids) and the balance, if any of the aforesaid R, R¹ and R² groups is a lower alkyl group and R⁷ is a lower alkyl group. In another embodiment X is a sulfate group.

The invention also relates to a mixture of the various quaternary ammonium compounds as described above in combination with the fatty acids in the amounts and ratios as described previously.

15 The invention also relates to a fabric softening article of manufacture comprising a fabric softening amount of any of the mixtures as described herein operatively associated with a substrate that will release such mixture to a fabric under fabric softening conditions encountered in a fabric or clothes dryer and includes the use of any of the aforesaid mixtures in a fabric softening relationship with a nonwoven or woven fiber or a polymeric open-celled or substantially open-celled foam substrate, such combination being prepared in a manner well known in the art. The mixture is employed in an amount from about 0.1 to about 10 gms on a 9" x 11" sheet and especially about 1 to about 3 gms on a 9" x 11" sheet.

The various polymeric foams that are employed in this respect comprise polyurethane foams as well as any of the art known equivalent foams.

Additionally, the invention is directed to a fabric
1 cleaning composition comprising a detergent in combination
with a fabric softening amount of any of the mixtures
described herein. These detergents can be any of the art
known anionic, cationic, nonionic or amphoteric synthetic
5 detergents or wetting agents that are well known in the art
or a soap i.e. the reaction product of a fatty acid with a
alkaline hydroxide that is water soluble e.g. fatty acid
reaction products of sodium, potassium or ammonium hydroxides
or amines or the art known equivalents thereof.

10 Shampoos may also be formulated according to the
invention with such detergents and a hair conditioning amount
of the quaternary ammonium compound and fatty acid mixtures
of the present invention.

These surfactants are further described in Kirk-
15 Othmer, Encyclopedia of Chemical Technology, 3rd Edition
Vol. 22 pp. 332-432. Some specific detergents that
are especially suitable in this regard include:

20 alkyl benzene sulfate,
sodium lauryl ether sulfate,
nonyl phenyl ethoxylates and
alkyl alcohol ethoxylates.

25 The mixture of the invention is incorporated into
the detergents or soaps (whether solid or liquid) by blending
in a manner well known in the art. The amount of the mixture
employed is any where from about 1 to about 50 and especially
from about 2 to about 30 wt. % based on the quaternary
30 ammonium compound and the active components of the detergent
or soap i.e. the component of the detergent or soap that has
both organophilic and hydrophilic groups.

The following example is illustrative.

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The method of the invention as well as a mixture obtained according to this method is exemplified by the preparation of a mixture employing the following components 5 in the indicated amounts.

<u>Components</u>	<u>M.W.</u>	<u>Moles</u>	<u>% by Wt.</u>
Dihydrogentated Tallow-	523	1.00	53.52
10 Methyl-Amine			
Dimethyl Sulfate	126	0.98	12.98
Water			3.0
Ethanol			2.0
Stearic Acid			28.5

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The amine, water and alcohol are charged to a reactor and heated to 140°F. A nitrogen purge was used over the surface of the reactants in the reactor to minimize contact of the reactants with air. The dimethylsulfate was 20 charged at a rate to maintain the temperature of the reactants at 140-180°F. When the percentage of free amine has stabilized at 1.0-2.0 weight percent the stearic acid is added and mixed. The mixture thus obtained is heated to 200°F after which the water and ethanol are removed by means 25 of nitrogen sparging and/or vacuum stripping.

The foregoing method produced a reaction mixture which was analyzed by NMR analysis showing that it had the following components:

	<u>Weight Percent</u>
30 Quaternary Ammonium compound	67
Fatty Acid	33

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Although the invention has been described by reference
1 to some embodiments, it is not intended that the novel method
or the mixture obtained thereby as well as the fabric
softening article of manufacture and the detergent containing
a fabric softening amount of the mixture be limited thereby
5 but that certain embodiments are intended being included as
falling within the broad scope and spirit of the foregoing
disclosure and the following claims.

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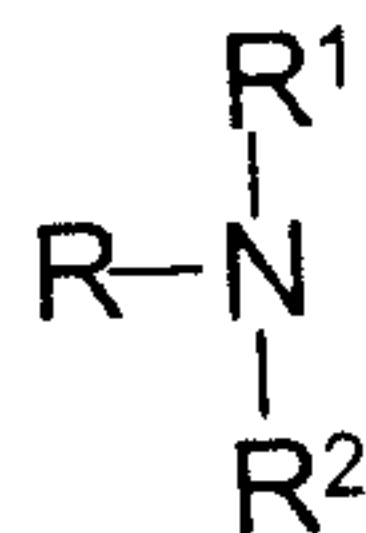
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THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A method for making a mixture of a quaternary ammonium compound and a fatty acid which is substantially free of solvents, fatty acid ester and amine salts by reacting an amine and a quaternizing agent in the presence of a relatively small amount of a solvent based on from about 0.5 to about 5%, by weight, alcohol and from about 0.25 to about 5%, by weight, water to obtain a first mixture of a quaternary ammonium compound, said alcohol and said water, adding a fatty acid to said first mixture to obtain a second mixture that is relatively fluid and removing said alcohol and said water as a vapor to thereby obtain said quaternary ammonium compound and said fatty acid that is substantially free of solvents, fatty acid esters and amine salts, wherein said amine has the formula:

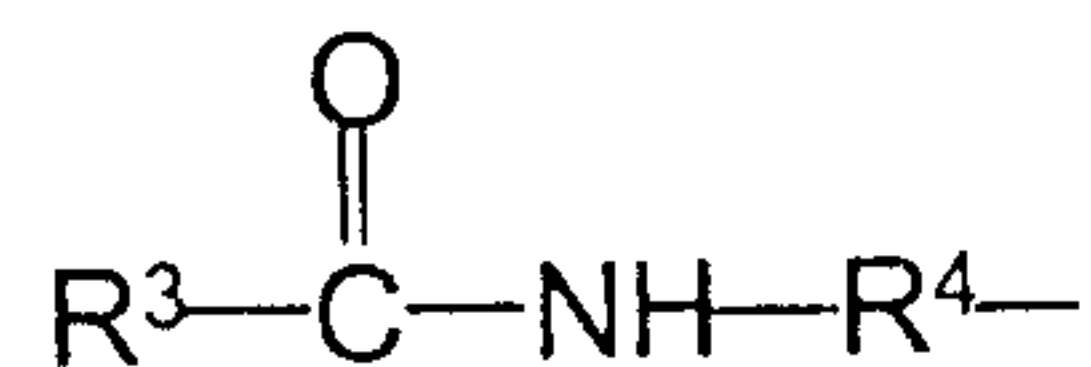


where R, R¹ and R² are any of:

(1) a linear or branched chain aliphatic saturated or unsaturated hydrocarbon group having up to about 22 carbon atoms;

(2) a hydroxy lower alkyl group;

(3) an alkyl amido alkylene group of the formula:



where R⁴ is lower alkylene and R³ is any of (1), (2), (4), or (5);

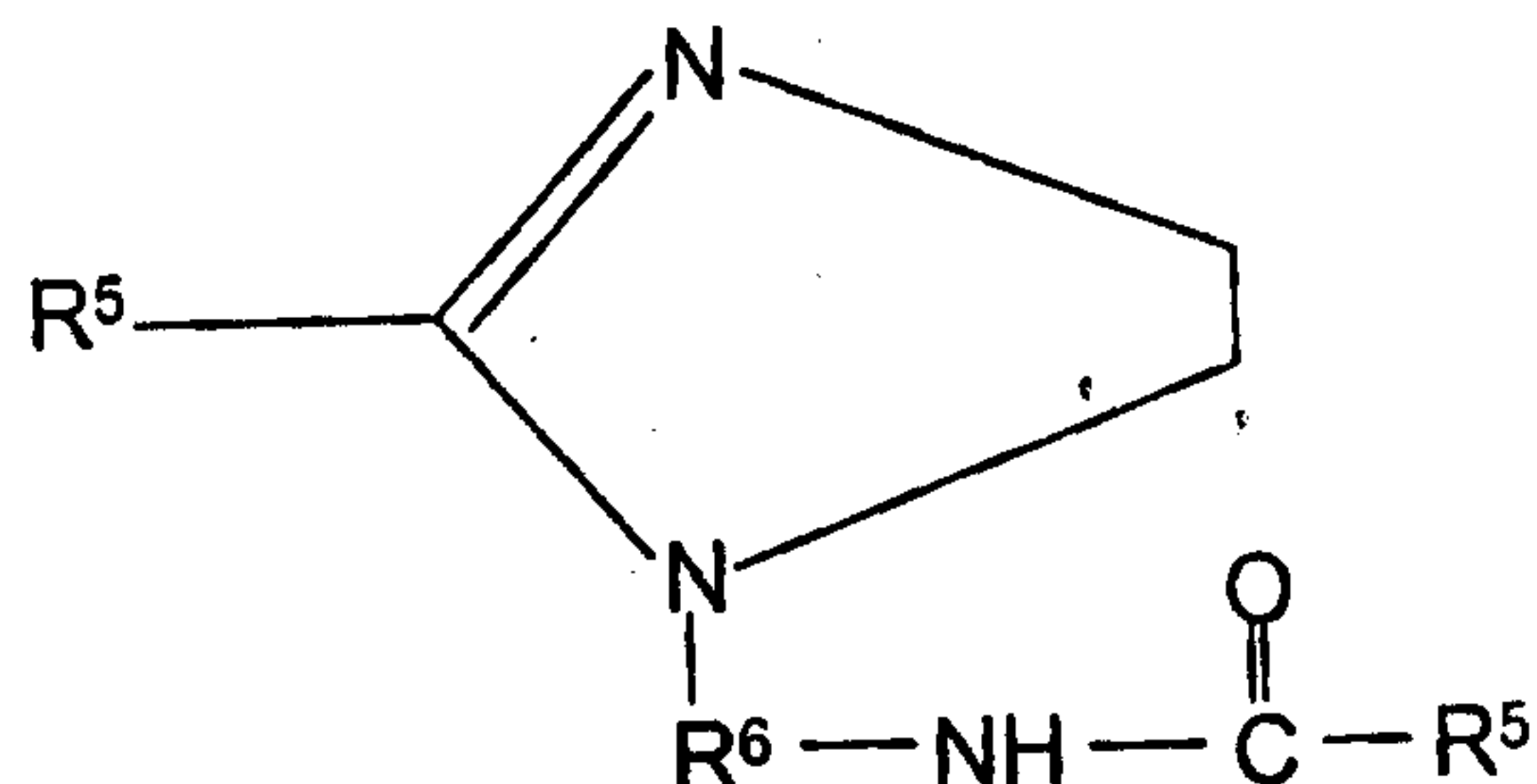
(4) a lower alkoxy group;

(5) a poly(oxyloweralkylene) group;

wherein at least one of R, R¹ or R² is one of said

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hydrocarbon groups or one of said hydroxy lower alkyl groups;
or said amine is an imidazoline of the formula:



where R^5 is a linear or branched chain aliphatic saturated or unsaturated hydrocarbon group having up to about 22 carbon atoms and R^6 is a lower alkylene group.

2. The method of claim 1, where said second mixture is subjected to sparging with a gas at temperatures of from about 80°C to about 100°C to remove any of said water or alcohol.

3. The method of claim 1 or 2, further comprising subjecting said second mixture to a vacuum to remove any of said water or alcohol.

4. The method of claim 1, 2 or 3, where said amine is present in a slight stoichiometric excess based on said quaternizing agent, and said amine and quaternizing agent being reacted in amounts sufficient to provide said quaternary ammonium compound in an amount from about 10% to about 90% by weight, said fatty acid being added in an amount to be present in an amount from about 90% to about 10% by weight, the weight percents of said quaternary ammonium compound and said fatty acid being based on the total amount of quaternary ammonium compound and fatty acid, said alcohol and water being sufficiently removed so that a mixture of quaternary ammonium compound and fatty acid is obtained having from about 95% to about 100% of said quaternary ammonium compound and fatty acid.

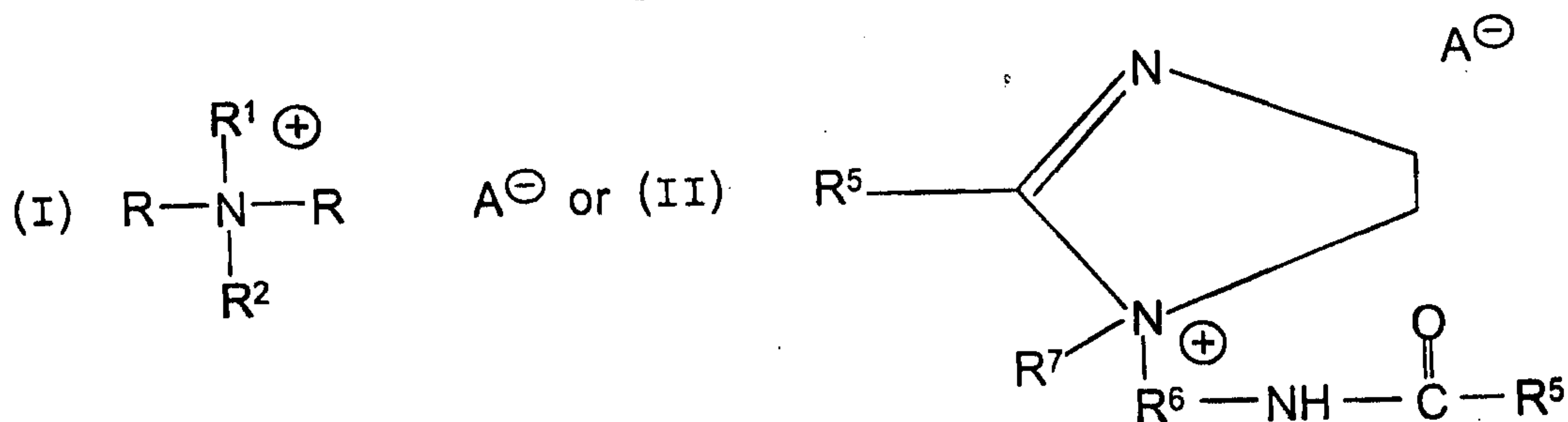
5. The method of claim 4, where

said quaternizing agent being capable of producing a quaternary ammonium compound having an anion A^{-} ;

- 19 -

said fatty acid is a linear or branched chain saturated or unsaturated fatty acid having from about 12 to about 22 carbon atoms;

said quaternary ammonium compound having the formula:



wherein R, R¹, R², R⁵, and R⁶ are as defined in claim 1 and R⁷ is a lower alkyl group or cyclo lower alkyl group; A⁻ is chlorine, iodine, bromide, sulfate, methyl sulfate, carbonate, phosphate, or a borate group.

6. The method of claim 5, where said quaternary ammonium compound is (I) and wherein at least one of said R, R¹ and R² groups is a branched chain or linear, aliphatic saturated or unsaturated hydrocarbon group having from about 12 to about 22 carbon atoms and the balance, if any, of said R, R¹ and R² groups is a lower alkyl group.

7. The method of claim 1, where said amine comprises a dihydrogenated tallow methyl amine and said quaternizing agent comprises a dialkyl sulfate and said fatty acid comprises stearic acid.

8. The method of any one claims 1 to 7, wherein said mixture is associated with a substrate in a softening amount under substrate softening conditions.

9. The method of any one of claims 1 to 7, wherein the mixture is combined with a cleaning composition.

10. The method of claim 9, wherein the cleaning composition is a detergent, soap or shampoo.

11. The method of claim 8, wherein the substrate is a fabric or a polymeric open cell foam.