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"CONCENTRATED STABLE NON-AQUEOUS
FABRIC SOFTENER COMPOSITION"

The present invention relates to fabric softener compositions adapted for use in the rinse cycle of a laundering process and in particular to highly concentrated fabric softener compositions which are easily dispersed in water when used, particularly in laundry machines having automatic dispensing mechanisms.

Compositions containing quaternary ammonium salts having at least one long chain hydrocarbyl group are commonly used to provide fabric softening benefits when employed in a laundry rinse operation; for example, see U.S. Patents 3,349,033; 3,644,203; 3,946,115; 3,997,453; 4,073,735 and 4,119,545.

For most aqueous softener compositions containing cationic quaternary ammonium compounds as active ingredients, the concentration of such cationics has, in general, been limited to the range of about 3 to 6% by weight (see U.S. Patent 3,904,533 and U.S. Patent 3,920,565). Such a low concentration is generally necessitated by the fact that cationics form gels in water systems at concentrations at above about 8%, and while the use of electrolytes to lower the viscosity of such compositions is known (see in particular U.S. Patent 4,199,545), such electrolytes are far from satisfactory. From a functional point of view, the electrolytes often do not perform as required particularly at concentrations of the cationics in the neighbourhood of about 12-15%. Further, while the performance of the electrolytes may

mitigate some of the gelling problem, their use is far from satisfactory in providing a highly concentrated aqueous system of cationics which does not gel or severely change in viscosity within the usual range of temperatures encountered in the handling thereof, for example 0°F (about -18°C) up to about 140°F (about 60°C) or in the dispensing from washing machines.

In the ordinary use of European household automatic washing machines, the user places the rinse cycle fabric softener in a dispensing unit (e.g. a dispensing drawer) of the machine. Then, in the operation of the machine, during the rinse cycle, the softener composition is subjected to a stream of cold water to transfer it to the drum. In winter, when the softener composition and the water fed to the dispenser may be especially cold, there can be problems in that some of the composition is not flushed completely off the dispenser during operation of the machine, and a deposit of the composition may build up with repeated wash cycles, so that it may become necessary for the user to flush the dispenser with hot water. This problem can be particularly severe for highly concentrated softener formulations because of the aforementioned gelling problem and also when a nonionic surfactant is present with the cationic softener since there is a tendency for the viscosity of the nonionic to increase when mixed with cold water forming a gel.

In British Application 2053249A published

4th February, 1981, there are disclosed cationic fabric softening compositions containing 15 to 60% by weight of cationic softener, 25 to 75% by weight of an aqueous medium and 0.5 to 40% by weight of a specified water soluble polymer.

In U.S. Patent 4,351,737 concentrated fabric softeners are described containing both cationic and non-ionic softeners and a non-ionic dispersing agent along with a solvent mixture of a C_1 to C_3 alkanol and a liquid glycol, polyglycol or an alkyl ether thereof. Hexylene glycol is not disclosed.

It has now been found that the dispersibility in cold water and the flow from automatic dispensers, even in highly concentrated, nonionic surfactant-containing liquid fabric softener compositions can be improved considerably by replacing part or all of the conventional liquid carrier of the non-aqueous liquid softener composition by hexylene glycol.

The present invention provides substantially non-aqueous stable, concentrated softener compositions based upon quaternary ammonium softening compounds and a carrier liquid comprising hexylene glycol and optionally a nonionic surfactant. The present invention also provides a method by which highly concentrated fabric softening compositions are produced and used.

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Thus according to the present invention a stable substantially non-aqueous, concentrated fabric softening composition comprises (A) a water dispersible quaternary ammonium compound cationic softener; (B) a non-aqueous carrier liquid comprising a mixture containing at least 25% by weight of hexylene glycol and the balance being a C₁ to C₃ alkanol, a C₂ to C₆ glycol, a C₁ to C₆ mono- or di-alkyl ether of the said glycol, or mixtures thereof, and (C) a non-ionic surfactant.

The invention also extends to a composition comprising (A) water dispersible quaternary ammonium compound cationic softener; (B) a non-aqueous carrier liquid comprising a mixture containing at least 25% by weight of hexylene glycol and the balance being a C₁ to C₃ alkanol, a C₂ to C₆ glycol, a C₁ to C₆ mono- or di-alkyl ether of the said glycol, or mixtures thereof, and (C) a nonionic surfactant which is a condensate of a C₈-C₂₀ aliphatic alcohol or a mono-, di-, or tri-alkyl phenol, with ethylene oxide or propylene oxide or a mixture thereof, each of the said alkyl groups in the alkyl phenol containing from 4 to 12 carbon atoms. Preferably A and B together constitute at least 91.9% of the composition.

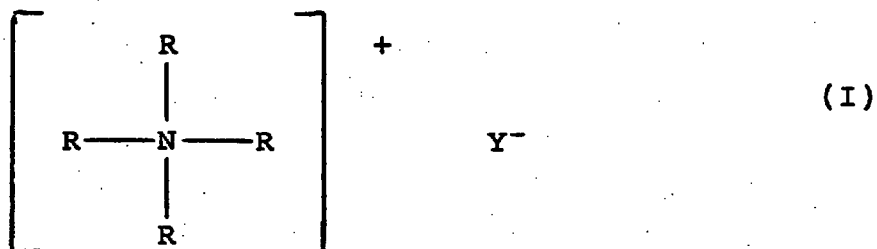
The compositions of the present invention are stable substantially non-aqueous compositions which contain a high concentration of the cationic fabric softener which is a water dispersible quaternary

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ammonium compound as hereinafter described and hexylene glycol as the, or as part of the, liquid carrier. Preferred compositions may also include non-ionic surfactant and electrolyte.

5. The compositions of the present invention contain at least about 20% cationic softener and up to about 80% thereof, preferably up to about 70% and more preferably up to about 60% said cationic softener having the general formula:

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- wherein R represents a C₁ to C₃₀ aliphatic group, preferably alkyl or alkenyl, or an aryl group (e.g. phenyl, tolyl or cumyl); or an aralkyl group (e.g. benzyl or phenethyl); or a halo, amido, hydroxy, or carboxy substituted version thereof; with the proviso that at least one R represents a C₁₄ to C₃₀ and preferably a C₁₄ to C₁₈ group, and the other R's are
20. lower alkyl groups, and more preferably at least two R's are C₁₄ to C₁₈ and the others are lower alkyl of C₁ to C₄ (and most preferably methyl or ethyl), or hydroxyalkyl (i.e. (CH₂-CH-O)_xH where x is 1 to 10,

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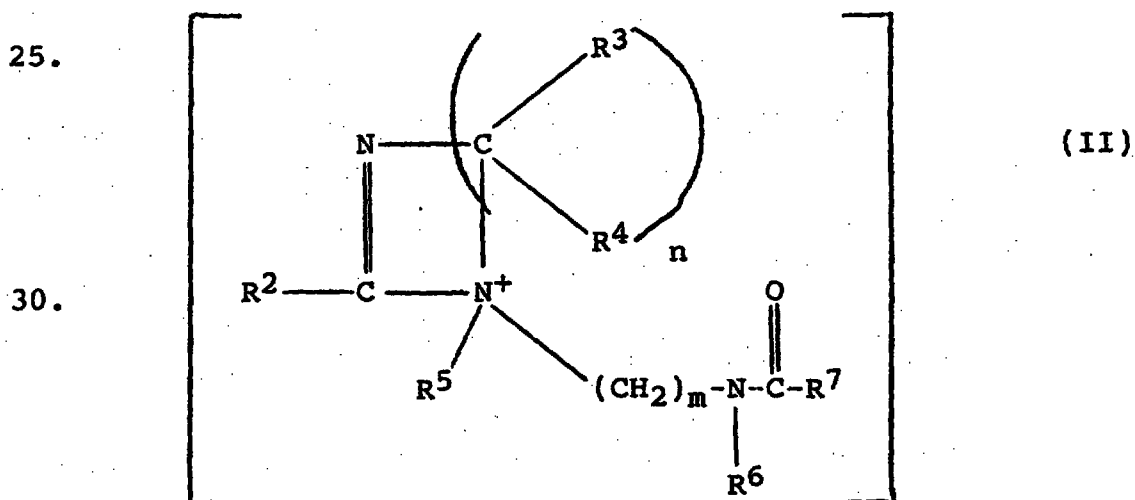
R¹

preferably 1 to 5, most preferably 1 or 2 and R^1 represents a hydrogen atom or a C_1 to C_4 alkyl group, and Y represents a water-solubilizing anion such as chloride, bromide, iodide, fluoride, sulphate, metho-
 5. sulphate, nitrite, nitrate, phosphate or carboxylate (e.g. acetate, adipate, propionate, phthalate, benzoate or oleate). Typical cationic compounds of Formula I include the following:

10. distearyl dimethyl ammonium chloride;
 ditallow dimethyl ammonium chloride;
 dihexadecyl dimethyl ammonium chloride;
 distearyl dimethyl ammonium bromide;
 di(hydrogenated tallow) dimethyl ammonium
 bromide;
 15. ditallow isopropyl methyl ammonium chloride;
 distearyl di(isopropyl) ammonium chloride;
 distearyl dimethyl ammonium methosulphate.

A highly preferred class of cationics is of Formula I wherein two of the R groups are C_{14} to C_{18} , one R is
 20. methyl, or ethyl and one R is methyl, ethyl, iso-
 propyl, n-propyl, hydroxyethyl or hydroxypropyl.

Other quaternary softeners which may be used include the imidazolinium compounds of the formula:



wherein R² represents a C₆ to C₃₀, preferably C₈ to C₁₈ alkyl or alkenyl group;

R³ and R⁴ each independently represent a hydrogen atom or a C₁ to C₄ alkyl group;

5 R⁵ represents a C₁ to C₄ alkyl group;

R⁶ represents a hydrogen atom or a C₁ to C₄ alkyl group;

R⁷ represents a C₆ to C₃₀, preferably C₈ to C₁₈, alkyl group;

10 n is 2 or 3; and

m is 2 or 3.

It will be appreciated that when n=2 the heterocyclic ring is five membered and when n=3 it is six membered.

15 Illustrative compounds of Formula II include:

1-methyl-1-((tallowylamido-) ethyl) -2-tallowyl 4,5 dihydro imidazolinium methyl sulphate; and

1-methyl-1-((palmitoylamido) ethyl)-2-octadecyl- 4,5 dihydroimidazolinium chloride.

20 Specifically preferred cationics are:

ditallow monomethyl monohydroxypropyl ammonium chloride;

distearyl dimethylammonium chloride;

ditallow dimethylammonium chloride; and

25 ditallow isopropyl methyl ammonium chloride.

The non-aqueous carrier may comprise from about 20% to about 80%, preferably from about 25% to about 60%, by weight of the composition.

30 The non-aqueous carrier of the composition of the present invention comprises at least about 25% and preferably at least 30% or 35% of hexylene glycol and the balance a C₁ to C₃ alkanol, preferably ethanol or propanol, a C₂ to C₆ glycol, preferably diethylene

glycol or propylene glycol, or a C₁ to C₆ mono- or di-alkyl ether of such glycols or mixtures thereof. The entire carrier may consist of hexylene glycol to the exclusion of the alcohols and/or glycols (and/or glycol ethers). Hexylene glycol is the commercial name for 2-methylpentane-2,4-diol which will be referred to as hexylene glycol herein.

Typical carriers may comprise (a) 25% hexylene glycol and 75% propylene glycol; (b) 31% hexylene glycol and 69% propylene glycol; (c) 31% isopropanol and 69% hexylene glycol; (d) 100% hexylene glycol.

The total amount by weight of the cationic fabric softener and the non-aqueous liquid carrier in the composition will preferably be at least about 80 and up to 100%, preferably from about 85% to 99% by weight, the balance including nonionic surfactant, optional electrolyte and/or optional conventional additives.

The optional non-ionic component of the present composition comprises from about 0 to about 5.5%, by weight of the composition and generally may vary with the cationic softener in a weight ratio of cationic to nonionic of from about 25:1 to about 3:1, preferably from about 12:1 to 6:1 and especially preferably about 10:1. Suitable non-ionic compounds include ethylene oxide and propylene oxide (and mixtures thereof) condensates of C₈ to C₂₀ aliphatic alcohols and mono-, di- and tri-alkyl (each C₄ to C₁₂) phenols. Generally, the non-ionics preferred herein are moderately hydro-philic in nature with a moderate hydrophilic group.

In the present invention, this group is preferably an oxyethyl chain generally of at least about 2 terminal oxyethyl groups (i.e. the oxyethyl groups are contiguous and terminate in an hydroxyl group) with

5. no more than about 20 such groups, although there may be as many as 200 moles oxyethylene per mole of the hydrophobic group.

Particularly suitable non-ionic surfactants include a C₉₋₁₁ aliphatic alcohol containing 8 moles

10. of condensed ethylene oxide; an ethoxylated nonyl phenol with 6 moles of ethylene oxide, an ethoxylated octyl phenol with 4.5 moles of ethylene oxide and a C₉ aliphatic alcohol with 7.5 moles ethylene oxide.

In addition to the foregoing components of the

15. softening compositions of the present invention, there may also be included numerous conventional, supplemental, and optional ingredients which do not adversely affect the stability and/or functional characteristics of the compositions of the present

20. invention. Thus, for example, there may be present the ubiquitous perfumes, dyes, pigments, opacifiers, germicides, optical brighteners, anti-corrosion agents (e.g. sodium silicate), polymers, anti-static agents and the like. Where used, each may comprise,

25. for example, from 0.01% to about 5% by weight of the composition.

It is, of course, recognized and understood that most available chemical materials and particularly those containing an hydrocarbyl moiety are generally

30. mixtures of closely related moieties. Thus, the long

chain alkyl substituents (R) in the cationics used in the present invention may not only be a single length carbon chain but are more likely a mixture. In this regard, a particularly useful quaternary salt,

5. wherein the alkyl groups are derived from tallow, may contain about 35% C₁₆ and 60% C₁₈ and minor amounts of C₁₄ and even others. Similarly, the aliphatic alcohol precursors for the non-ionics used herein may be of a single carbon chain length but more likely, again, they will and can be a mixture in any proportion of the operable chain length compounds.

- The fabric softening compositions of the present invention should have viscosities in the range of about 10 to 250 centipoises and preferably 25 to 150 centipoises in addition to their essential water-dispersibility in the rinse cycle (or any other form of dilution prior to use).

- The manner of combining the hexylene glycol, other optional carriers, cationic softener and optional nonionic surfactant, electrolyte, and other optional conventional additives is not particularly critical, nor is the order of addition. However, some degree of care should be taken to avoid excessive heating in order to limit solvent evaporation and, more importantly, to stay below the flash point temperature of the carrier and any other low boiling liquids that may be present or brought in as components of the softener and the like. For example, the flash point for hexylene glycol is 93°C. Generally, however, it is preferred to combine

all of the ingredients at room temperature, e.g. from about 15°C to 25°C, or lower.

A suitable procedure for preparing the compositions of the present invention involves dissolving any nonionic and other additives (e.g. brightener, colour, perfume, etc.) in the carrier and then mixing this mixture with the cationic e.g. by adding the mixture to the cationic which preferably is in liquid form, e.g. as a dispersion in the carrier or any component thereof (for example, as a dispersion in isopropanol), or adding the cationic to the mixture.

The invention extends to a method of imparting softness to fabrics which comprises adding a composition in accordance with the present invention to an aqueous medium containing the fabrics in an amount to provide a concentration of the cationic compound of from about 0.005% to 0.5% based on the weight of the fabrics. The invention also extends to a method which involves diluting the composition with from about 4 to 15 times as much water by volume as the volume of the said composition and then adding the diluted composition to the clothes.

The invention may be put into practice in various ways and a number of specific embodiments will be described to illustrate the invention with reference to the accompanying examples. Parts are by weight unless otherwise indicated.

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EXAMPLE 1

Following the procedure described above, 2 parts of an ethoxylated nonyl phenol (containing 6 moles of ethylene oxide) are dissolved in about 75 parts of hexylene glycol at a temperature of about 20°C. To this solution are slowly added 20 parts of distearyl dimethyl ammonium chloride (75% active in isopropanol) with stirring. A stable product results with a viscosity of about 100 cps.

EXAMPLES 2A to 2D

The procedure of Example 1 is repeated utilizing the following parts of (A) cationic (active), (B) surfactant; and (C) hexylene glycol given in Table 1 below:

TABLE 1

<u>Example</u>	<u>2A</u>	<u>2B</u>	<u>2C</u>	<u>2D</u>
Ingredients				
A	22	34	46	58
5. B	2.0	3.5	4.8	5.5
C	76.0	62.5 ¹	49.2	36.5

Notes on Table 1

¹ Hexylene glycol/ethylene glycol at 3:1 weight ratio.

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EXAMPLES 3A to 3F

Examples 1 and 2 are repeated utilizing in place of distearyl dimethyl ammonium chloride the following:

Example 3A - ditallow dimethyl ammonium chloride;

Example 3B - distearyl dimethyl ammonium metho-
sulphate;

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Example 3C - di(hydrogenated tallow) dimethyl ammonium
bromide;

Example 3D - di-hexadecyl dimethyl ammonium chloride;

Example 3E - distearyl diethyl ammonium chloride;

20. Example 3F - 1-methyl-1-((tallowylamido)ethyl)-2-
tallowyl-4,5-dihydroimidazolinium methyl
sulphate.

EXAMPLE 4

In order to demonstrate the improved dispensi-
25. bility of the hexylene glycol based liquid carrier
for the concentrated softening compositions the
following formulations Examples 4A to E are prepared
having the proportions set out in Table 2 below:

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TABLE 2

		<u>Weight %</u>
	Solvent (A-E)	31.9
	Cationic fabric softener ¹	60.0
5.	Nonionic ²	5.0
	Perfume, dye, minors	3.1

Notes on Table 2

- ¹ 75% active ingredient in isopropanol.
² Nonyl phenol ethoxylated with an average of 6 moles
10. ethylene oxide (C-9 phenol EO6:1).

The following are used as solvent:

- Example 4A - 10% isopropyl alcohol and 21.9% diethylene glycol (comparison);
15. Example 4B - 10% hexylene glycol and 21.9% propylene glycol;
Example 4C - 10% isopropyl alcohol and 21.9% hexylene glycol;
Example 4D - 31.9% hexylene glycol;
20. Example 4E - 31.9% propylene glycol (comparison).

- Each of the above softener formulations are added to the dispenser of a typical European washing machine, AEG-802, and after 3 cumulative up-to-boil cycles, (the dispenser being refilled for each
25. cycle), the amount of the formulation remaining in the dispenser is measured. The results are shown in the following Table 3 as a percent of the total amount used (i.e. a triple dose):

TABLE 3

<u>Example Solvent</u>		<u>Quantity Remaining (%)</u>
5.	4A 10% isopropanol	15
	21.9% diethylene glycol	
	4B 10% hexylene glycol	6.7
	21.9% propylene glycol	
	4C 10% isopropanol	3.6
	21.9% hexylene glycol	
	4D 31.9% hexylene glycol	2.8
10.	4E 31.9% propylene glycol	19

From the above results, it can be easily appreciated that the use of hexylene glycol in place of all or part of the conventional non-aqueous liquid carriers for rinse cycle added fabric softener formulations greatly improves the cold water dispersibility and flow (dispensibility), even of concentrated formulations containing nonionic surfactants.

Moreover, the concentrated liquid fabric softener compositions of the present invention are stable for long periods of time at both low, e.g. 4°C and high, e.g. 25°C, storage temperatures, including freeze-thaw cycles, over periods of 6 weeks and longer.

The compositions of the present invention have the further advantage that hexylene glycol is non-toxic and, therefore, the formulations are safe to use.

While the compositions of the present invention are intended primarily for direct use in the rinse cycle of automatic washing machines they can also be

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used in diluted form and for manual treatment, both in and out of the washing machine.

- Still further, it should be understood that as used herein, the term "non-aqueous" means that no
5. water is intentionally added to the system, although minor amounts of water, e.g. up to about 5%, can be present, based on addition of specific ingredients, e.g. dyes, as aqueous solutions, and the water contained in the softener, etc.

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CLAIMS

1. A stable substantially non-aqueous, concentrated fabric softening composition comprising (A) a water dispersible quaternary ammonium compound cationic softener; (B) a non-aqueous carrier liquid comprising a mixture containing at least 25% by weight of hexylene glycol and the balance being a C_1 to C_3 alkanol, a C_2 to C_6 glycol, a C_1 to C_6 mono- or di- alkyl ether of the said glycol, or mixtures thereof; and (C) a non-ionic surfactant.

2. A stable substantially non-aqueous, concentrated fabric softening composition comprising (A) water dispersible quaternary ammonium compound cationic softener; (B) a non-aqueous carrier liquid comprising a mixture containing at least 25% by weight of hexylene glycol and the balance being a C_1 to C_3 alkanol, a C_2 to C_6 glycol, a C_1 to C_6 mono- or di- alkyl ether of the said glycol, or mixtures thereof, and (C) a nonionic surfactant which is a condensate of a C_8 - C_{20} aliphatic alcohol or a mono-, di-, or tri-alkyl phenol, with ethylene oxide or propylene oxide or a mixture thereof, each of the said alkyl groups in the alkyl phenol containing from 4 to 12 carbon atoms.

3. A composition as claimed in Claim 1 or Claim 2 in which A and B together constitute at least 91.9% of the composition.



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4. A composition as claimed in Claim 1, 2 or 3 which comprises 20 to 80% by weight of (A) and 20 to 80% by weight of (B).

5. A composition as claimed in Claim 1, 2, 3 or 4 which comprises from 1 to 5.5% by weight of (C) nonionic surfactant.

6. A composition as claimed in anyone of Claims 1 to 5 in which the weight ratio of cationic fabric softener to nonionic surfactant is from about 25:1 to about 3:1.

7. A composition as claimed in Claim 6 in which the non-aqueous carrier (B) comprises at least 30% by weight of hexylene glycol.

8. A composition as claimed in Claim 7 in which the non-aqueous carrier consists of hexylene glycol.

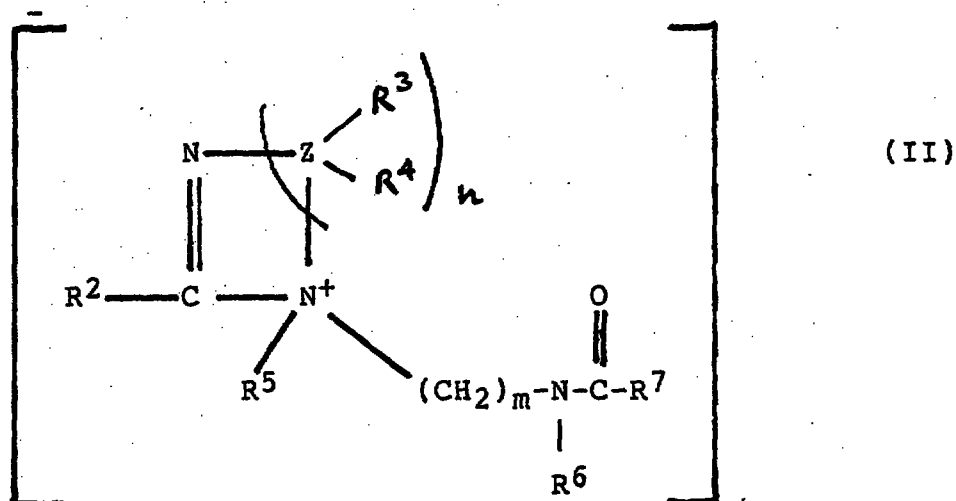
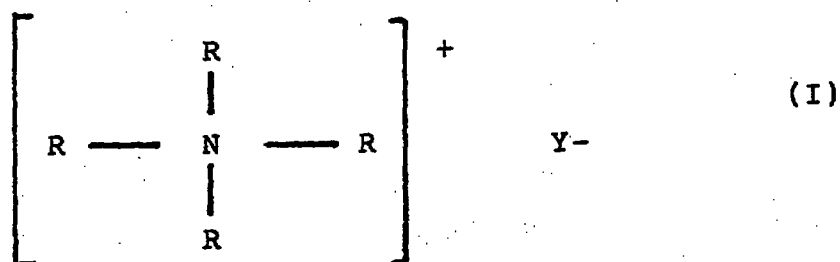
9. A composition as claimed in any one of Claims 1 to 8 in which the said non-aqueous carrier comprises (a) 25% hexylene glycol and 75% propylene glycol; (b) 31% hexylene glycol and 69% propylene glycol; (c) 31% isopropanol and 69% hexylene glycol; or (d) 100% hexylene glycol.

10. A composition as claimed in any one of Claims 1 to 9 in which the cationic softener is selected from the group consisting of compounds of



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the formula (I) or (II):



wherein R represents a C₁-C₃₀ aliphatic group, or an aryl group or an aralkyl group or a halo, amido, hydroxy or carboxy substituted version thereof with the proviso that at least one R represents a C₁₄ to C₃₀ group, and the other R's represent lower alkyl or (CH₂-CHR'-O)_xH groups where x is 1 to 10, and R' represents a hydrogen atom or a C₁ to C₄ alkyl group, Y represents a water-solubilizing anion; R² represents a C₆ to C₃₀ alkyl or alkenyl group; R³ and R⁴ each, independently, represent a hydrogen atom or



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a C₁ to C₄ alkyl group; R⁵ represents a C₁ to C₄ alkyl group; R⁶ represents a hydrogen atom or a C₁ to C₄ alkyl group; R⁷ represents a C₆ to C₃₀ alkyl group; m is 2 or 3; and n = 2 or 3.

11. A composition as claimed in Claim 10 in which the said condensate comprises from about 2 to about 20 moles of ethylene oxide per mole of alcohol or phenol.

12. A composition as claimed in Claim 1 substantially as specifically described herein with reference to the accompanying examples.

13. A method of forming a stable, concentrated non-aqueous fabric softening composition which comprises combining a water dispersible quaternary ammonium compound cationic fabric softening compound with a substantially non-aqueous liquid carrier comprising a mixture containing at least 25% by weight of hexylene glycol and the balance being a C₁ to C₃ alkanol, a C₂ to C₆ mono- or di- alkyl ether of the said glycol, or mixtures thereof, and a nonionic surfactant, at about room temperature, the amount of the cationic compound being at least about 20% by weight of the composition.

14. A method for imparting softness to fabrics which comprises adding a composition as claimed in any one of Claims 1 to 12 to an aqueous medium



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containing the fabrics in an amount to provide a concentration of the cationic compound of from about 0.005% to 0.5% based on the weight of the fabrics.

15. A method as claimed in Claim 14 in which the composition is added during the rinse cycle of an automatic washing machine.

16. A method as claimed in Claim 15 in which the composition is added to the aqueous medium by flowing a stream of cold water onto the composition whereby the composition is dispersed in the flowing stream and then transferred to the aqueous medium.

17. A method for imparting softeners to clothes which combines diluting the composition as claimed in any one of Claims 1 to 12 with from about 4 to 15 times as much water by volume as the volume of the said composition and then adding the diluted composition to the clothes.
