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(5) Built liquid detergent compositions containing ternary active systems. (3) Priority: 06.04.81 US 251650 **1** Proprietor: **THE PROCTER & GAMBLE** COMPANY **301 East Sixth Street** 4 Date of publication of application: Cincinnati Ohio 45202 (US) 05.01.83 Bulletin 83/01 1 Inventor: Jacobsen, Ronald Lowell (4) Publication of the grant of the patent: 159 Burn Avenue 03.12.86 Bulletin 86/49 Wyoming, OH 45215 (US) Inventor: Hanley, Dean Charles 2944 Fairfield Avenue Cincinnati, OH 45201 (US) (A) Designated Contracting States: AT BE CH DE FR GB IT LI NL SE (74) Representative: Ernst, Hubert et al (58) References cited: **PROCTER & GAMBLE EUROPEAN TECHNICAL** EP-A-0 034 039 **CENTER Temselaan 100** FR-A-2 160 886 B-1820 Strombeek-Bever (BE) GB-A-1 091 517 GB-A-1 538 174 GB-A-2 078 246

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Courier Press, Learnington Spa, England.

#### Description

Background of the invention

- This invention relates to stable liquid detergent compositions having superior detergent properties. There has been considerable demand for liquid detergent compositions which provide superior 5 detergency under a wide variety of conditions including cool water conditions. In order to obtain superior detergency under a wide variety of conditions, a number of components are needed. The formulation of stable liquid detergent compositions is difficult when the components tend to separate into discrete phases.
- U.S. Patent 4,247,424, discloses liquid detergent compositions in the form of water-in-oil emulsions 10 which contain ethoxylated alcohol and amine oxide surfactants.
  - U.S. Patent 4,284,532, discloses liquid detergent compositions in isotropic form which contain ethoxylated alcohol and amine oxide surfactants.
- U.S. Patent 4,276,205, discloses detergent compositions containing ethoxylated alcohol and amine 15 oxide surfactants plus a polyalkylene glycol detergency improver such as polyethylene glycol.
  - British Patent 1,538,174 discloses a composition containing, in conjunction with casein, an alkylether sulfate and a nonionic detergent component which is selected from amine oxide and ethoxylated alcohols.
- British Patent 1,091,517, discloses a liquid detergent composition containing an alkyl-polyethoxysulfate, an amine oxide, a pyrophosphate and low levels of an alkyl monoethoxy-ether as a viscositycontrolling and foam-enhancing agent. 20

### Summary of the invention

The present invention encompasses a stable liquid detergent composition comprising:

(a) from 3% to 20% of an ethoxylated alcohol or ethoxylated alkyl phenol nonionic surfactant of the 25 formula R(OC<sub>2</sub>H<sub>4</sub>)<sub>n</sub>OH, wherein R is selected from the group consisting of aliphatic hydrocarbon radicals containing from 8 to 15 carbon atoms and alkyl phenyl radicals in which the alkyl group contains from 8 to 12 carbon atoms, n is from 3 to 9, and said nonionic surfactant has an HLB value of from 10 to 13; (b) from 2% to 15% of an amine oxide surfactant having the formula

 $R^{1}(OC_{2}H_{4})_{n}N$ -

- 35 wherein R<sup>1</sup> is an alkyl, hydroxyalkyl, alkoxyhydroxypropyl, alkoxyhydroxyethyl, alkyl amido or alkyl carboxylate radical in which the alkyl and alkoxy portions contain from 8 to 18 carbon atoms, R<sup>2</sup> and R<sup>3</sup> are selected from methyl, ethyl, propyl, isopropyl, 2-hydroxyethyl, 2-hydroxypropyl, 3-hydroxypropyl, or said groups joined together to form a cyclic structure in which the nitrogen is part of a heterocyclic ring, and n is 40 from 0 to 10;

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(c) from 1% to 10% of an alcohol polyethoxylate sulfate surfactant having the formula- $R^4O(C_2H_4O)_mSO_3M$ , wherein  $R^4$  is an alkyl or hydroxyalkyl radical containing from 10 to 18 carbon atoms, m is from 2 to 10 and M is a compatible cation;

- (d) from 5% to 25% of a water-soluble nitrilotriacetate or citrate detergency builder capable of sequestering calcium and magnesium ions in water solution; provided that components (a), (b), (c) and (d) 45 together represent less than 40% by weight of the composition;
  - (e) from 0% to 25% of a hydrotrope;
  - (f) up to 89% water; said liquid detergent composition being in isotropic form and having a pH of from 8 to 13 in a 0.2% water solution at 20°C.
- The compositions of the invention are single phase isotropic liquids which exhibit improved stability in 50 that they return, or substantially return, to a single isotropic phase after freezing and thawing. The compositions of the invention also avoid yellowing of fabrics which are regularly softened with
- Detailed description of the invention 55

conventional cationic fabric softeners.

- The liquid detergent compositions herein comprise five essential ingredients:
- (a) ethoxylated nonionic surfactant;
- (b) amine oxide surfactant;
- (c) alcohol polyethoxylate sulfate surfactant:
- (d) water-soluble detergency builder; and
- (e) water.

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The nonionic, amine oxide and polyethoxylate sulfate surfactants and the builders herein together represent less than 40% by weight of the composition. Compositions containing more than 40% by weight of such components tend to separate into a surfactant-rich soap phase and a salt-rich lye phase after

freezing and thawing. Preferred compositions herein contain less than 34%, and most preferably less than

28%, by weight of such components, and exhibit even greater stability in that they return to single phase isotropic liquids after being slowly frozen (e.g., over a period of days) and thawed. The compositions, particularly those with higher surfactant and builder levels within the above limits, also preferably contain the optional hydrotropes herein which help to solubilize the surfactants and salts in the water phase under

a wide variety of conditions. 5

#### Ethoxylated nonionic surfactant

The compositions of the present invention contain from 3% to 20% by weight of an ethoxylated nonionic surfactant of the formula  $R(OC_{2}H_{4})_{n}OH$ , wherein R is selected from the group consisting of aliphatic hydrocarbon radicals containing from 8 to 15 carbon atoms and alkyl phenyl radicals in which the 10 alkyl group contains from 8 to 12 carbon atoms, n is from 3 to 9, and said nonionic surfactant has an HLB value of from 10 to 13.

Suitable ethoxylated nonionic surfactants are the condensation products of alkyl phenols having an alkyl group containing from 8 to 12 carbon atoms, in either a straight chain or branched chain configuration, with ethylene oxide, the ethylene oxide being present in amounts equal to from 3 to 9 moles of ethylene oxide per mole of alkyl phenol. The alkyl substituent in such compounds can be derived, for example, from polymerized propylene or isobutylene, or from octene or nonene. Examples of compounds of this type include nonyl phenol condensed with 8 moles of ethylene oxide per mole of nonyl phenol and dodecyl phenol condensed with 9 moles of ethylene oxide per mole of dodecyl phenol. Commercially available nonionic surfactants of this type include Igepal® CO-530, CO-610, CO-630, CA-520, CA-620, and CA-630, marketed by the GAF Corporation.

Other useful nonionic surfactants herein are condensation products of primary or secondary aliphatic alcohols with from 3 to 9 moles of ethylene oxide per mole of alcohol. The alkyl chain of the aliphatic alcohol can either be straight or branched and contains from 8 to 15 carbon atoms. Examples of such ethoxylated alcohols include the condensation product of 5 moles of ethylene oxide with 1 mole of tridecanol, myristyl alcohol condensed with 8 moles of ethylene oxide per mole of myristyl alcohol, the condensation product of ethylene oxide with coconut fatty alcohol wherein the coconut alcohol is a mixture of fatty alcohols with alkyl chains varying from 10 to 14 carbon atoms and wherein the condensate contains 6 moles of ethylene oxide per mole of alcohol, and the condensation product of 9 moles of ethylene oxide

with coconut alcohol. Examples of commercially available nonionic surfactants of this type include 30 Tergitol® 15-S-7 marketed by the Union Carbide Corporation and Neodol® 23-6.5 marketed by the Shell Chemical Company. Whether the alcohol is derived from natural fats or produced by one of several petrochemical processes, a mixture of a carbon chain lengths is typical. The stated degree of ethoxylation is an average, the distribution being dependent on process conditions.

Ethoxylated alcohols are preferred because of their superior biodegradability relative to ethoxylated alkyl phenols. Particularly preferred are ethoxylated alcohols having an average of from 10 to 15 carbon atoms in the alcohol and an average degree of ethoxylation of from 3 to 8 moles of ethylene oxide per mole of alcohol.

The cloud point of a 1% aqueous solution of the ethoxylated nonionic surfactant is preferably below 75°C. and most preferably below 55°C.

The ethoxylated nonionic surfactants will have HLB (hydrophile-lipophile balance) values of from 10 to 13 and limited water solubility. The HLB value of surfactants and emulsifiers can be determined experimentally in a well-known fashion. The HLB value of compounds or mixtures of compounds in which the hydrophilic portion of the molecule is principally ethylene oxide can be estimated by the weight ratio of ethylene oxide portion to the lipophilic portion (e.g., the hydrocarbyl radical).

A preferred level of ethoxylated nonionic surfactants in the compositions of the invention is from 5% to 10%.

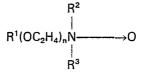
Optional ethoxylated nonionic surfactants include: (1) the condensation products of ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide and propylene glycol, and (2) the condensation products of ethylene oxide with the product resulting from the reaction of propylene oxide 50 and ethylenediamine. These surfactants are marketed by BASF Wyandotte under the tradenames Pluronic® and Tetronic® respectively.

#### Amide oxide surfactant

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The compositions of the present invention also contain from 2% to 15% by weight of an amine oxide surfactant having the formula

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wherein R<sup>1</sup> is an alkyl, hydroxyalkyl, alkoxyhydroxypropyl, alkoxyhydroxyethyl, alkyl amido or alkyl carboxylate radical in which the alkyl and alkoxy, respectively, contain from 8 to 18 carbon atoms, R<sup>2</sup> and R<sup>3</sup>

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are methyl, ethyl, propyl, isopropyl, 2-hydroxyethyl, 2-hydroxypropyl, 3-hydroxypropyl, or together are a heterocyclic, e.g., morpholino, structure in which the nitrogen is part of the heterocyclic ring, and n is from 0 to 10.

- Specific examples of amine oxide surfactants include: dimethyldodecylamine oxide, dimethyltetradecylamine oxide, ethylmethyltetradecylamine oxide, cetyldimethylamine oxide, dimethylstearyl-5 amine oxide, cetylethylpropylamine oxide, diethyldodecylamine oxide, diethyltetradecylamine oxide, dipropyldodecylamine oxide, bis - (2 - hydroxyethyl)dodecylamine oxide, bis(2 - hydroxyethyl) - 3 dodecoxy - 2 - hydroxypropylamine oxide, (2 - hydroxypropyl)methyltetradecylamine oxide, dimethyloleylamine oxide, dimethyl - (2 - hydroxydodecyl)amine oxide, C<sub>8-18</sub> alkyl alpha-dimethylamine oxide 10 carboxylates, and the corresponding decyl, hexadecyl and octadecyl homologs of the above compounds. A
- particularly preferred material is  $C_{12-16}$  alkyl dimethylamine oxide.

A preferred level of amine oxide surfactant in the compositions of the invention is from 3% to 10%. Preferred weight ratios of ethoxylated nonionic surfactant to amine oxide surfactant are from 1:1 to 4:1, more preferably from 1.5:1 to 3:1.

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#### The alcohol polyethoxylate sulfate surfactant

The present compositions contain from 1% to 10%, preferably from 2% to 6%, by weight of an alcohol polyethoxylate sulfate surfactant and of the formula  $R^4O(C_2H_4O)_mSO_3M$ , wherein  $R^4$  is an alkyl (preferred) or hydroxyalkyl radical containing from 10 to 18 carbon atoms, m is from 2 to 10 and M is a compatible cation.

The alcohol polyethoxylate sulfate surfactant is essential to the overall performance and stability of the present compositions. It has been found that if only the nonionic and amine oxide surfactants are present, fabrics which are regularly softened with conventional cationic fabric softening ingredients such as di-tallowdimethylammonium chloride will gradually become objectionably yellow. The reason for this effect is unclear, but in any event it can be controlled by the addition of an anionic surfactant to the

compositions. Surprisingly, of the anionic surfactants tested, only the alcohol polyethoxylate sulfates can be added to the compositions in sufficient quantity to provide the desired effect without forming a separating phase or requiring the use of uneconomical amounts of compatibilizing materials.

The specific alcohol polyethoxylate sulfate surfactants require at least about two ethoxy groups per molecule on the average to keep the composition single phase. Preferably the degree of ethoxylation is 30 from two to three.

Preferred alcohol polyethoxylate sulfate surfactants are C12-15 alkyl polyethoxylate (2.2) sulfate (C12-15E2.2S); C14-15E2.2S; C12-13E3S; C16-18E5S; C14-15E3S; and mixtures thereof. The sodium, potassium, and monoethanolammonium salts, and mixtures thereof, are preferred.

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#### Soap

A desirable and preferred additional ingredient is a C<sub>10-18</sub> fatty acid soap having the same cation or cations as the other anionic materials due to ion exchange. This ingredient, at a level of from 0.2% to 5%, , preferably from 0.5% to 1%, provides corrosion protection, suds control, and additional cleaning potential. Coconut and unsaturated C<sub>16-18</sub> soaps such as oleyl are preferred for solubility reasons.

#### Water-soluble detergency builder

The compositions herein also contain from 5% to 25%, preferably from 10% to 20%, by weight of a water-soluble nitrilotriacetate or citrate detergency builder capable of sequestering calcium or magnesium 45 ions in water solution.

The essential detergency builders of the present invention have the ability to sequester calcium or magnesium ions in water solution, and also maintain or assist in maintaining an alkaline pH in a washing solution. Sequestration is the formation of coordination complexes with metallic ions to prevent or inhibit precipitation or other interfering reactions. The phenomenon is also called chelation if certain structural

criteria are met by the coordination complex.

Examples of such builders are sodium and potassium nitrilotriacetate.

Other suitable polycarboxylate builders include the water-soluble salts, especially the sodium and potassium salts of citric acid.

Preferred builders for use in the present invention are sodium and potassium nitrilotriacetate, sodium 55 and potassium citrate, and mixtures thereof. Sodium nitrilotriacetate is particularly preferred.

It is to be understood that while the alkali metal, and particularly the sodium and potassium, salts of the foregoing detergency builder salts are preferred for use herein from economic and solubility standpoints, the ammonium, alkanolammonium, e.g., triethanolammonium, diethanolammonium, and the like, water-soluble salts of the foregoing builder anions are also useful herein.

#### Water

The compositions of this invention contain up to 89% water, and preferably contain from 35% to about 65% water. 65

Optional hydrotrope

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The liquid detergent compositions of this invention are stable and isotropic. Those containing lower levels of water are not necessarily true solutions. Many of the compositions herein appear to be microemulsions of an oil phase in water, the oil phase comprising inter alia the bulk of the ethoxylated nonionic surfactant.

The hydrotropes of the present invention are water soluble and preferably have an HLB value above 14. Suitable hydrotropes have shorter alkyl chain lengths than the corresponding surfactants used as the principal surfactant in detergent compositions. For example, the soluble salts, particularly sodium and potassium salts, of toluene sulfonate, xylene sulfonate, and cumene sulfonate are preferred hydrophilic

<sup>10</sup> stabilizing agents in the practice of the invention; a  $C_{11-15}$  alkylbenzene sulfonate typically used in household detergent compositions is not suitable. The cations are the same as or compatible with the anionic surfactants.

Phosphate esters, particularly those with a predominance of single alkyl groups and designated primary esters, can have the hydropholic characteristics necessary to assist in the formation of an isotropic

15 liquid detergent composition. Emphos® PS-413 and PS-236 (Witco Chemical Company) and Gafac® PE-510 (GAF Corporation) are commercially available phosphate materials suitable as the hydrotrope in the practice of the invention. Preferred phosphate esters will contain a high proportion of monoalkyl phosphate esters and can be of the type consisting of the condensation product of the reaction of R(OC<sub>2</sub>H<sub>4</sub>)<sub>x</sub>OH and a phosphoric or polyphosphoric acid, R being an alkyl or alkyl phenyl group, said alkyl containing from 4 to 18 carbon atoms and x being 0 to 20.

Ethoxylated nonionic surfactants with a relatively high degree of ethoxylation and a corresponding high HLB value can find use in the compositions of the present invention.

Mixtures of hydrotropes, especially mixtures of lower alkylbenzene sulfonates, such as toluene sulfonate, and phosphate esters, can be used, but preferably no phosphorus is present.

The types and levels of hydrotropes needed to produce an isotropic liquid detergent composition will be dependent on the type and level of other components, particularly the ethoxylated nonionic surfactant and its extent of water solubility. A preferred level of hydrotrope is from 5% to 16% by weight of the liquid detergent composition.

30 Other optional components

The following ingredients can be present, but desirably are not present, especially in substantial quantities. In some embodiments of the present invention the detergent compositions can contain up to 10%, preferably to 5%, of a fatty acid amide surfactant, such as ammonia amides, monoethanol amides, diethanol amides, and ethoxylated amides. Preferred amides are  $C_{8-20}$  monoethanol amides,  $C_{8-20}$  diethanol amides, and amides having the formula

O H  $\parallel \parallel$ R—C—N—CH<sub>2</sub>CH<sub>2</sub>OCH<sub>2</sub>CH<sub>2</sub>OH,

wherein R is a  $C_{8-20}$  alkyl group, and mixtures thereof. Particularly preferred amides are those where the alkyl group contains from 10 to 16 carbon atoms, such as coconut alkyl monoethanol or diethanol amide. Such compounds are commercially available under the tradenames Super-Amide® L-9 and GR, from Onyx Chemical Co., Jersey City, NJ, Super-Amide® F-3 from Ryco, Inc., Conshohocken, PA, and Gafamide® CDD-518, available from GAF Corp., New York, NY.

These amide components can be added to act as suds modifiers. They tend to boost the sudsing in an active system which exhibits relatively low sudsing and can depress the sudsing in systems which exhibit high sudsing.

The compositions of the present invention may also contain additional ingredients generally found in laundry detergent compositions, at their conventional art-established levels, as long as these ingredients are compatible with the components required herein. For example, the compositions can contain up to 15%, preferably no more than 5%, and most preferably from 0.001 to 2%, of one or more suds control components. Typical suds control agents useful in the compositions of the present invention include, but are not limited to, those described below.

55 Silicone suds control additives are described in U.S. Patent 3,933,672. The silicone material can be represented by alkylated polysiloxane materials such as silica aerogels and xerogels and hydrophobic silicas of various types. The silicone material can be described as a siloxane having the formula:

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65 wherein x is from 20 to 2,000, and R and R' are each alkyl or aryl groups, especially methyl, ethyl, propyl,

butyl, and phenyl. Polydimethylsiloxanes (R and R' are methyl, having a molecular weight within the range of from 200 to 200,000, and higher, are all useful as suds controlling agents. Additional suitable silicone materials wherein the side chain groups R and R' are alkyl, aryl, or mixed alkyl and aryl hydrocarbyl groups exhibit useful suds controlling properties. Examples of such ingredients include diethyl-, dipropyl-,

- dibutyl-, methyl-, ethyl-, phenylmethyl polysiloxanes and the like. Additional useful silicone suds control 5 agents can be represented by a mixture of an alkylated siloxane, as referred to hereinbefore, and solid silica. Such mixtures are prepared by affixing the silicone to the surface of the solid silica. A preferred silicone suds control agent is represented by a hydrophobic silanated silica having a particle size in the range from 10 to 20 nm and a specific surface area above 50 m<sup>2</sup>/gm intimately admixed with dimethyl
- silicone fluid having a molecular weight in the range from 500 to 200,000 at a weight ratio of silicone to 10 silanated silica of from 19:1 to 1:2. The silicone suds suppressing agent is advantageously releasably incorporated in a water-soluble or water-dispersible, substantially nonsurface-active, detergentimpermeable carrier.

Particularly useful suds control agents are the self-emulsifying silicone suds control agents described in U.S. Patent 4,075,118. An example of such a compound is DB-544®, commercially available from Dow

Corning, which contains a siloxane/glycol copolymer together with solid silica and a siloxane resin. Microcrystalline waxes having a melting point in the range from 35°C-115°C and a saponification value of less than 100 represent additional examples of preferred suds control components for use in the subject compositions, and are described in detail in U.S. Patent 4,056,481. The microcrystalline waxes are

- substantially water-insoluble, but are water-dispersible in the presence of organic surfactants. Preferred 20 microcrystalline waxes have a melting point from 65°C to 100°C, a molecular weight in the range from 400-1,000; and a penetration value of at least 6, measured at 25°C by ASTM-D1321. Suitable examples of the above waxes include: microcrystalline and oxidized microcrystalline petroleum waxes; Fischer-Tropsch and oxidized Fischer-Tropsch waxes; ozokerite; ceresin; montan wax; beeswax; candelilla; and carnauba wax.
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Alkyl phosphate esters represent an additional preferred suds control agent for use herein. These preferred phosphate esters are predominantly monostearyl phosphate which, in addition thereto, can contain di- and tristearyl phosphates and monooleyl phosphates, which can contain di- and trioleyl phosphates.

The alkyl phosphate esters frequently contain some trialkyl phosphate. Accordingly, a preferred 30 phosphate ester can contain, in addition to the monoalkyl ester, e.g., monostearyl phosphate, up to 50 mole percent of dialkyl phosphate and up to 5 mole percent of trialkyl phosphate.

Other adjunct components which can be included in the compositions of the present invention include anionic, zwitterionic, ampholytic and cationic surfactants; bleaching agents; bleach activators; soil release agents (particularly copolymers of ethylene terephthalate and polyethylene oxide terephthalate, such as 35 Milease T® sold by ICI, United States, as disclosed in U.S. Patent 4,132,680); soil suspending agents; corrosion inhibitors; dyes; fillers; optical brighteners; germicides; pH adjusting agents; alkalinity sources; enzymes; enzyme-stabilizing agents; perfumes, solvents such as ethyl alcohol; carriers; opacifiers; and the like. The required solution pH of from about 8 to about 13 can be obtained by the use of suitable alkaline

materials such as sodium hydroxide, sodium or potassium carbonate or bicarbonate, sodium or potassium 40 silicates, and the alkanolamines. Particularly preferred is monoethanol amine.

Preferably, the composition contains an optical brightening or whitening agent.

Suitable optical brightening agents include:

(1) The reaction product of about one mole of ethylene oxide and one mole of 1,2 bis(benzimidazolyl) ethylene, e.g., N - (2' - hydroxyethyl) - 1,2 - bis(benzimidazolyl) ethylene; (2) tetrasodium 4,4' - bis[(4'' - bis(2''' - hydroxyethyl) - amino - 6'' - (3''' - sulfophenyl)amino -45

1",3",5" - triazin - 2" - yl)amino] - 2,2' - stilbenedisulfonate;

- (3) N (2 hydroxyethyl 4,4' bis(benzimidazolyl)stilbene;
- (4) disodium 4 [6' sulfonaphtho(1',2' d)triazol 2 yl] 2 stilbenesulfonate;
- (5) disodium 4,4' bis[6 methyl ethanolamine) 3 anilino 1,3,5 triazin 2'' yl] 2,2' -50 stilbenedisulfide;

(6) disodium 4,4' - bis[(4'' - (2''' - hydroxyethoxy) - 6'' - anilino - 1'',3'',5'' - triazin - 2'' yl)amino] - 2,2' - stilbenedisulfonate;

(7) 1,2 - bis(5' - methyl - 2' - benzoxazolyl)ethylene;

- (8) 4 methyl 7 dimethylaminocoumarin;
  - (9) 2 styrylnaphth[1,2 d]oxazole;

(10) the reaction product of one mole of 4,4' - bis - (benzimidazoly!)stilbene with 0.5 mole of ethylene oxide and 0.5 mole of propylene oxide; and

(11) mixtures thereof.

60 These optical whitening agents are used in a level of from 0.03% to 0.8% and preferably at a level of 0.4% by weight.

Because of the outstanding performance characteristics of the present invention, surfactants additional to the essential components will not generally be necessary.

Examples of additional surfactant which can be used in the compositions of the present invention are 65

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found in U.S. Patent 3,717,630. However, these components should be used in an amount as to be certain that they will be compatible with the essential surfactant system.

All percentages, parts, and ratios used herein are by weight unless otherwise specified.

The following nonlimiting examples illustrate the compositions of the present invention.

5	EXAMPLES					
		1	11	111	IV	V
10	Sodium C <sub>14-15</sub> alkyl polyethoxy 2.25 sulfate	2.9	2.9			mana
	Sodium $C_{12-13}$ alkyl polyethoxy <sub>3</sub> sulfate	_	—	2.9		
15	Monoethanolammonium C <sub>12</sub> alkyl benzene sulfonate	_		_	2.6	
	Oleic fatty acid		0.4	—		1.6
20	Coconut fatty acid	0.5		0.5	0.5	_
	$C_{12-13}$ alcohol polyethoxylate <sub>6.5</sub>	6.4	6.4	6.4	6.4	4.7
25	$C_{12-16}$ alkyldimethyl amine oxide	3.3	3.3	3.3	3.3	3.3
	sodium nitrilotriacetate	18.2	18.2	18.2	18.2	21.5
	Potassium toluene sulfonate	9.0	9.0	9.5	11.5	9.0
30	Monoethanolamine	2.8	2.7	2.8	2.8	2.9
	Water and minors	41 <u>0</u> 4, <del>-</del>		Balance	<b>0</b>	
	Stability for one week at -1.1, 4.4, 10, 21.1 and 48.9°C	OK*	ОК	OK	ОК	ОК
35	Freeze at -17.8°C followed by thawing at -1.1°C	ŌK	OK	ОК	heavy ppt	ОК
40	thawing at 4.4°C	ОК	ОК	ОК	heavy ppt	white ppt
	thawing at 10°C <sup>.</sup>	OK	ОК	ОК	heavy ppt	white ppt
45	thawing at 21.1°C	ОК	ОК	ОК	ОК	ОК

\*OK means remains as a single phase isotropic liquid.

The above compositions were prepared by mixing the ingredients to form single phase isotropic liquids. The compositions remained as single phase isotropic liquids during the static testing for one week 50 at the indicated temperatures. However, when quickly frozen, only the compositions of the present invention (i.e., Examples I, II and III) returned to single phase isotropic liquids at all thawing temperatures tested.

When the level of sodium nitrilotriacetate in Examples I, II and III is reduced to 15%, thereby providing compositions containing a total of 27.6% by weight of the builder and surfactants herein, the compositions 55 exhibit even greater stability in that they also return to single phase isotropic liquids after being slowly frozen over a period of several days and thawed.

Other compositions of the present invention are obtained when the sodium nitriloacetate in Examples I, II and III is replaced with 15% of 20% of sodium or potassium citrate.

60 Claims

1. A stable liquid detergent composition comprising:

(a) from 3% to 20% by weight of an ethoxylated alcohol or ethoxylated alkyl phenol nonionic surfactant of the formula  $R(OC_2H_4)_nOH$ , wherein R is selected from the group consisting of aliphatic hydrocarbon 65

radicals containing from 8 to 15 carbon atoms and alkyl phenyl radicals in which the alkyl group contains from 8 to 12 carbon atoms, n is from 3 to 9, and said nonionic surfactant has an HLB value of from 10 to 13; (b) from 2% to 15% by weight of an amine oxide surfactant having the formula

> R<sup>2</sup> │ R<sup>1</sup>(OC₂H₄)<sub>n</sub>N-----→ │ B<sup>3</sup>

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wherein R<sup>1</sup> is an alkyl, hydroxyalkyl, alkoxyhydroxypropyl, alkoxyhydroxyethyl, alkyl amido or alkyl carboxylate radical in which the alkyl and alkoxy portions contain from 8 to 18 carbon atoms, R<sup>2</sup> and R<sup>3</sup> are selected from the group consisting of methyl, ethyl, propyl, isopropyl, 2-hydroxyethyl, 2-hydroxypropyl, 3-hydroxypropyl, or said groups joined together to form a cyclic structure in which the nitrogen is part of a heterocyclic ring, and n is from 0 to 10;

(c) from 1% to 10% by weight of an alcohol polyethoxylate sulfate surfactant having the formula

#### $R^4O(C_2H_4O)_mSO_3M$ ,

- 20 wherein R<sup>4</sup> is an alkyl or hydroxyalkyl radical containing from 10 to 18 carbon atoms, m is from 2 to 10 and M is a compatible cation;
  - (d) from 5% to 25% by weight of a water-soluble detergency builder;
  - (e) from 0% to 25% by weight of a hydrotrope;
  - (f) up to 89% by weight water;
- 25 characterized in that

- the water-soluble detergency builder is a nitrilotriacetate or a citrate, capable of sequestering calcium and magnesium ions in water solution;

- component (a), (b), (c) and (d) together represent less than 40% by weight of the composition;

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said liquid detergent composition is in isotropic form and has a pH of from 8 to 13 in a 0.2% water solution at 20°C.

2. The composition of Claim 1 comprising from 5% to 10% of an ethoxylated alcohol nonionic surfactant of the formula  $R(OC_2H_4)_nOH$  wherein R is a  $C_{10-15}$  alkyl group and n is from 3 to 8, from 3% to 10% of a  $C_{12-16}$  alkyl dimethylamine oxide, and from 2% to 6% of an alcohol polyethoxylate sulfate

surfactant of the formula  $R^4O(C_2H_4O)_mSO_3M$  wherein  $R^4$  is a  $C_{10-18}$  alkyl group, m is from 2 to 3 and M is sodium or potassium.

3. The composition of Claim 2 wherein the builder represents from 10% to 20% by weight of the composition.

4. The composition of Claim 3 wherein the nonionic, amine oxide and polyethoxylate sulfate surfactants and the builder together represent less than 28% by weight of the composition.

5. The composition of Claim 1 or 4 comprising from 5% to 16% of a hydrotrope selected from the group consisting of sodium and potassium toluene sulfonate, xylene sulfonate, cumene sulfonate, and mixtures thereof.

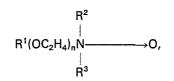
#### Patentansprüche

1. Eine stabile, flüssige Reinigungsmittelzusammensetzung, enthaltend:

(a) 3 Gew.-% bis 20 Gew.-% eines nichtionischen oberflächenaktiven Mittels, das ein ethoxylierter
 Alkohol oder ein ethoxyliertes Alkylphenol der Formel R(OC<sub>2</sub>H<sub>4</sub>)<sub>n</sub>OH ist, worin R aus der Gruppe ausgewählt ist, die aus aliphatischen Kohlenwasserstoffresten mit 8 bis 15 Kohlenstoffatomen und Alkylphenylresten, in welchen die Alkylgruppe 8 bis 12 Kohlenstoffatome enthält, besteht, n 3 bis 9 ist, und wobei das genannte nichtionische oberflächenaktive Mittel einen HLB-Wert von 10 bis 13 hat;

(b) 2 Gew.-% bis 15 Gew.-% eines Aminoxid-oberflächenaktiven Mittels der Formel





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worin R<sup>1</sup> ein Alkyl-, Hydroxyalkyl-, Alkoxyhydroxypropyl-, Alkoxyhydroxyethyl-, Alkylamido- oder Alkylcarboxylatrest ist, in welchem die Alkyl- und Alkoxyabschnitte 8 bis 18 Kohlenstoffatome enthalten, R<sup>2</sup> und R<sup>3</sup> aus der Gruppe ausgewählt sind, die aus Methyl, Ethyl, Propyl, Isopropyl, 2-Hydroxyethyl, 2-65 Hydroxypropyl und 3-Hydroxypropyl besteht, oder worin die genannten Gruppen miteinander unter

Bildung einer cyclischen Struktur verbunden sind, in welcher der Stickstoff Teil eines heterocyclischen Ringes ist, und worin n 0 bis 10 ist;

(c) 1 Gew.-% bis 10 Gew.-% eines Alkoholpolyethoxylatsulfat-oberflächenaktiven Mittels der Formel

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$$R^4O(C_2H_4O)_mSO_3M_1$$

worin R<sup>4</sup> ein Alkyl- oder Hydroxyalkylrest mit 10 bis 18 Kohlenstoffatomen ist, m 2 bis 10 ist, und M ein verträgliches Kation ist;

(d) 5 Gew.-% bis 25 Gew.-% eines wasserlöslichen Detergensgerüstoffes;

(e) 0 Gew.-% bis 25 Gew.-% eines Hydrotrops; und

(f) bis zu 89 Gew.-% Wasser;

dadurch gekennzeichnet, daß

der wasserlösliche Detergensgerüststoff ein Nitrilotriacetat oder ein Citrat ist, das in Wasserlösung zur
 Komplexbildung von Calcium- und Magnesiumionen befähigt ist;

--- die Komponenten (a), (b), (c) und (d) zusammengenommen weniger als 40 Gew.-% der Zusammensetzung darstellen;

und die genannte flüssige Detergenszusammensetzung in isotroper Form vorliegt und in einer 0,2 % igen 20 Wasserlösung bei 20°C einen pH von 8 bis 13 aufweist.

Die Zusammensetzung des Anspruchs 1, enthaltend 5 % bis 10 % eines nichtionischen oberflächenaktiven Mittels, das ein ethoxylierter Alkohol der Formel R(OC<sub>2</sub>H<sub>4</sub>)<sub>n</sub>OH ist, worin R eine C<sub>10-15</sub>-Alkylgruppe ist, und n 3 bis 8 ist, 3 % bis 10 % eines C<sub>12-16</sub>-Alkyldimethyaminoxids, und 2 % bis 6 % eines Alkoholpolyethoxylatsulfat - oberflächenaktiven Mittels der Formel R<sup>4</sup>O(C<sub>2</sub>H<sub>4</sub>O)<sub>m</sub>SO<sub>3</sub>M, worin R<sup>4</sup> eine C<sub>10-18</sub>-Alkylgruppe ist, m 2 bis 3 ist, und M Natrium oder Kalium ist.

3. Die Zusammensetzung des Anspruchs 2, worin der Gerüstoff 10 Gew.-% bis 20 Gew.-% der Zusammensetzung ausmacht.

4. Die Zusammensetzung des Anspruchs 3, worin das nichtionische Aminoxid-oberflächenaktive Mittel und das Polyethoxylatsulfat - oberflächenaktive Mittel sowie der Gerüstoff zusammen weniger als 20 30 Gew.-% der Zusammensetzung ausmachen.

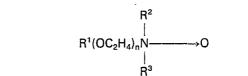
5. Die Zusammensetzung des Anspruchs 1 oder 4, enthaltend 5 % bis 16 % eines Hydrotrops, ausgewählt aus der Gruppe, die aus Natrium- und Kaliumtoluolsulfonat, -xylolsulfonat, -cumolsulfonat und Mischungen davon besteht.

#### 35 Revendications

1. Composition détergente liquide stable comprenant:

(a) de 3% à 20% en poids d'un tensioactif non ionique de type alcool éthoxylé ou alkylphénol éthoxylé de formule R(OC<sub>2</sub>H<sub>4</sub>)<sub>n</sub>OH, dans laquelle R est choisi dans le groupe constitué par les radicaux
40 hydrocarbonés aliphatiques contenant de 8 à 15 atomes de carbone et les radicaux alkylphényle dans lesquels le groupe alkyle contient de 8 à 12 atomes de carbone, et n est de 3 à 9, et le dit tensioactif non ionique a un rapport hydrophile-lipophile de 10 à 13;

(b) de 2% à 15% en poids d'un tensioactif de type oxyde d'amine de formule



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dans laquelle R<sup>1</sup> est un radical alkyle, hydroxyalkyle, alcoxyhydroxypropyle, alcoxyhydroxyéthyle, alkylamido ou alkylcarboxylate où les parties alkyle et alcoxy contiennent de 8 à 18 atomes de carbone, R<sup>2</sup> et R<sup>3</sup> sont choisis dans le groupe constitué par les radicaux méthyle, éthyle, propyle, isopropyle, 2-hydroxyéthyle, 2-hydroxypropyle, 3-hydroxypropyle, ou les dits groupes reliés l'un à l'autre pour former
une structure cyclique dans laquelle l'azote fait partie d'un noyau hétérocyclique, et n est de 0 à 10; (c) de 1% à 10% en poids d'un tensioactif de type alcoolsulfate polyéthoxylé de formule

#### $R^4O(C_2H_4O)_mSO_3M$ ,

- dans laquelle R<sup>4</sup> est un radical alkyle ou hydroxyalkyle contenant de 10 à 18 atomes de carbone, m est de 2 à 10 et M est un cation compatible;
  - (d) de 5% à 25% en poids d'un adjuvant de détergence hydrosoluble;
  - (e) de 0% à 25% en poids d'un hydrotrope;
  - (f) jusqu'à 89% en poids d'eau;

65 caractérisée en ce que

 – l'adjuvant de détergence hydrosoluble est un nitrilotriacétate ou un citrate, capable de séquestrer les ions calcium et magnésium en solution aqueuse;

- les constituants (a), (b), (c) et (d) ensemble représentent moins de 40% du poids de la composition;
- 5 la dite composition détergente liquide est sous une forme isotrope et a un pH de 8 à 13 en solution à 0,2% dans l'eau à 20°C.

2. Composition selon la revendication 1, comprenant de 5% à 10% d'un tensioactif non ionique de type alcool éthoxylé de formule R(OC<sub>2</sub>H<sub>4</sub>)<sub>n</sub>OH dans laquelle R est un groupe alkyle en C<sub>10-15</sub> et n est de 3 à 8, de 3% à 10% d'un oxyde d'alkyl (en C<sub>12-16</sub>) diméthylamine et de 2% à 6% d'un tensioactif de type alcoolsulfate
 10 polyéthoxylé de formule R<sup>4</sup>O(C<sub>2</sub>H<sub>4</sub>O)<sub>m</sub>SO<sub>3</sub>M dans laquelle R<sup>4</sup> est un groupe alkyle en C<sub>10-18</sub>, m est de 2 à 3

et M est le sodium ou le potassium.

3. Composition selon la revendication 2, dans laquelle l'adjuvant représente de 10% à 20% du poids de la composition.

4. Composition selon la revendication 3, dans laquelle les tensioactifs non ionique, de type oxyde 15 d'amine et de type sulfate polyéthoxylé et l'adjuvant représentent ensemble moins de 28% du poids de la composition.

5. Composition selon la revendication 1 ou 4, comprenant de 5% à 16% d'un hydrotrope choisi dans le groupe constitué par le toluène-sulfonate, le xylène-sulfonate, le cumène-sulfonate de sodium et de potassium et leurs mélanges.

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