

"HIGH FOAMING NONIONIC SURFACTANT BASED
LIQUID DETERGENT"

The present invention relates to novel light duty liquid detergent compositions with high foaming properties, containing a nonionic surfactant as the major active ingredient supplemented with lesser amounts of a specific group of anionic surfactants and even smaller amounts of a zwitterionic betaine surfactant and a fatty acid alkanolamide foam stabilizer in an aqueous medium.

Nonionic surfactants are in general chemically inert and stable toward pH change and are therefore well suited for mixing and formulation with other materials. The superior performance of nonionic surfactants on the removal of oily soil is well recognized. Nonionic surfactants are also known to be mild to human skin. However, as a class, nonionic surfactants are known to be low or moderate foamers. Consequently, for detergents which require copious and stable foam, the application of nonionic surfactants is limited. There has been substantial interest and efforts to develop a high foaming detergent with nonionic surfactants as the major ingredient. Yet, little has been achieved.

The prior art is replete with light duty liquid detergent compositions containing nonionic surfactants in combination with anionic and/or zwitterionic betaine surfactants wherein the nonionic detergent is not the major active surfactant, as shown in U.S. Patent No. 3,658,985 wherein an anionic based shampoo contains a minor amount of a fatty acid alkanolamide. U.S. Patent

- No. 3,769,398 discloses a betaine-based shampoo containing minor amounts of nonionic surfactants. This patent states that the low foaming properties of nonionic detergents renders its use in shampoo compositions non-preferred. U.S. Patent No. 5. 4,329,335 also discloses a shampoo containing a betaine surfactant as the major ingredient and minor amounts of a nonionic surfactant and of a fatty acid mono- or di-ethanolamide. U.S. Patent No. 4,259,204 10. discloses a shampoo comprising 0.8-20% by weight of an anionic phosphoric acid ester and one additional surfactant which may be either anionic, amphoteric, or nonionic. U.S. Patent No. 4,329,334 discloses an anionic-amphoteric based shampoo containing a major 15. amount of anionic surfactant and lesser amounts of a betaine and nonionic surfactants.

- U.S. Patent No. 3,935,129 discloses a liquid cleaning composition based on the alkali metal silicate content and containing five basic 20. ingredients, namely, urea, glycerin, triethanol-amine, an anionic detergent and a nonionic detergent. The silicate content determines the amount of anionic and/or nonionic detergent in the liquid cleaning composition. However, the foaming 25. property of these detergent compositions is not discussed therein.

- U.S. Patent No. 4,129,515 discloses a heavy duty liquid detergent for laundering fabrics comprising a mixture of substantially equal amounts 30. of anionic and nonionic surfactants, alkanolamines

and magnesium salts, and, optionally, zwitterionic surfactants as suds modifiers.

- U.S. Patent No. 4,224,195 discloses an aqueous detergent composition for laundering socks or
5. stockings comprising a specific group of nonionic detergents, namely, an ethylene oxide of a secondary alcohol, a specific group of anionic detergents, namely, a sulphuric ester salt of an ethylene oxide adduct of a secondary alcohol, and an amphoteric
10. surfactant which may be a betaine, wherein either the anionic or nonionic surfactant may be the major ingredient. The specific class of anionics utilized in this patent is the very same group of anionic detergents expressly excluded in the present
15. invention in order to eliminate the alkanol ethoxylate sulphation process and the potential dioxane toxicity problem. Furthermore, this patent finds heavily foaming detergents undesirable for the purpose of washing socks. Still further, this prior
20. detergent composition lacks a fatty acid alkanolamide foam stabilizer which is an essential ingredient in the light duty liquid detergent of the present invention.

- The prior art also discloses detergent compositions containing all nonionic surfactants as shown
25. in U.S. Patent No. 4,154,706 and U.S. Patent No. 4,329,336 wherein the shampoo compositions contain a plurality of particular nonionic surfactants in order to effect desirable foaming and deterative
30. properties despite the fact that nonionic surfactants are usually deficient in such properties.

U.S. Patent No. 4,013,787 discloses a piperazine based polymer in conditioning and shampoo compositions which may contain all nonionic surfactant or all anionic surfactant.

5. U.S. Patent No. 4,450,091 discloses high viscosity shampoo compositions containing a blend of an amphoteric betaine surfactant, a polyoxybutylene-polyoxyethylene nonionic detergent, an anionic surfactant, a fatty acid alkanolamide and a polyoxy-
10. alkylene glycol fatty ester. But, none of the exemplified compositions contains an active ingredient mixture wherein the nonionic detergent is present in major proportion, probably due to the low foaming properties of the polyoxybutylene polyoxy-
15. ethylene nonionic detergent.

- However, none of the above-cited patents discloses a high foaming, nonionic based, liquid detergent composition containing a nonionic surfactant as the major active ingredient and minor
20. amounts of a supplementary high foaming anionic sulphate or sulphonate surfactant excluding ethoxylated alcohol ether sulphates, a supplementary foaming zwitterionic surfactant selected from the betaine group and a fatty acid alkanolamide foam
25. stabilizer, as the four essential ingredients, the said nonionic ingredient constituting more than 50% of the total surfactant content.

- We have found that a high foaming liquid detergent can be formulated with a nonionic
30. surfactant as the major active ingredient which has

desirable cleaning properties, mildness to the human skin and avoids the dioxane toxicity problem associated with the sulphation process of manufacturing anionic ethoxylated alcohol ether sulphates.

5 The invention aims to provide novel, high foaming, nonionic based, light duty liquid detergent compositions containing a nonionic surfactant as the major active ingredient, i.e. in an amount exceeding 50% of the total surfactant content.

10 This invention also aims to provide novel, nonionic based, liquid detergent compositions containing a major amount of nonionic surfactant supplemented with lesser amounts of an anionic surfactant, a zwitterionic betaine surfactant and a
15 fatty acid alkanolamide foam stabilizer.

 This invention further aims to provide a novel, nonionic based, liquid detergent with desirable high foaming and cleaning properties which is mild to the human skin.

20 This invention also aims to provide a novel, nonionic based liquid detergent containing a supplemental anionic surfactant excluding the ethoxylated alkyl ether sulphates which eliminates the alkanol ethoxylate sulphation process and the
25 potential dioxane toxicity problem.

 According to the present invention a nonionic based, light duty liquid detergent consists essentially of as essential ingredients;

30 A) 8 to 30% of a water soluble, ethoxylated, nonionic surfactant as the major detergent ingredient;

B) 1 to 10% of a foaming anionic surfactant selected from the group consisting of water soluble organic sulphates and organic sulphonates, excluding the ethoxylated C₈-C₁₈ alkyl ether polyethenoxy sulphates;

C) 0.5 to 8% of a foaming, water soluble, C₈-C₁₈ alkyl amido propyl dimethyl betaine; and

D) 0.5 to 8% of a C₁₀-C₁₈ fatty acid monoethanolamide foam stabilizer;

dissolved in

E) an aqueous vehicle, the sum of A)-D) being from 10% to 55% by weight of the composition and the said nonionic detergent being in excess of 50% by weight of the said sum.

In a preferred aspect of the present invention a nonionic surfactant based, light duty liquid detergent consists;

A) 13% to 25% of a water soluble nonionic surfactant selected from the group consisting of primary and secondary C₈-C₁₈ alkanol condensates with 5 to 30 moles of ethylene oxide, condensates of C₈-C₁₈ alkylphenol with 5 to 30 moles of ethylene oxide, condensates of C₈-C₂₀ alkanol with a heteric mixture of ethylene oxide and propylene oxide having a weight ratio of ethylene oxide to propylene oxide from 2.5:1 to 4:1 and a total alkylene oxide content of 60% to 85% by weight and condensates of 2 to 30 moles of ethylene oxide with sorbitan mono- and tri-C₁₀-C₂₀ alkanolic acid esters having an HLB of 8 to 15;

B) 2% to 8% of a water-soluble anionic detergent selected from the group consisting of C₈-C₁₈ alkyl sulphates, C₈-C₁₆ alkylbenzene sulphonates, C₁₀-C₂₀ paraffin sulphonates, C₁₀-C₂₄ alpha olefin sulphonates, C₈-C₁₈ alkyl sulphoacetates; C₈-C₁₈ alkyl sulphosuccinate esters, C₈-C₁₈ acyl isethionates and C₈-C₁₈ acyl taurates;

C) 2% to 6% of a foaming, water soluble, C₈-C₁₈ alkyl amido propyl dimethyl betaine;

D) 2% to 6% of a C₁₂-C₁₄ fatty acid monoethanolamide foam stabilizer; solubilized in

E) an aqueous medium; the sum of A-D being from 20% to 40% by weight of the composition and the said nonionic detergent being in excess of 50% by weight of the said sum.

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This particular combination of four ingredients in the proportions, by weight, of more than 50% nonionic surfactant to less than 50% of the sum of anionic surfactant, betaine and fatty acid alkanol-
5 amide, is critical to the high foaming and desirable cleansing properties of present liquid detergent and the retention of the mildness to the skin property. The total amount of surfactants may constitute about 10%-55%, preferably about 20%-40%, most preferably
10 25%-35%, by weight of the liquid composition.

The nonionic surfactant which constitutes the major ingredient in the liquid detergent composition of the present invention is present in amounts of about 8%-30%, preferably 13%-25%, most preferably
15 16%-22%, by weight of the composition and provides superior performance in the removal of oily soil and mildness to human skin.

The water soluble nonionic surfactants utilized in the present invention are commercially well known
20 and include the primary aliphatic alcohol ethoxylates, secondary aliphatic alcohol ethoxylates, alkylphenol ethoxylates and ethylene-oxide-propylene oxide condensates of primary alkanols, such as Plurafac (Registered Trade Mark) (Wyandotte) and
25 condensates of ethylene oxide with sorbitan fatty acid esters such as the Tween (Registered Trade Mark) (ICI). The nonionic synthetic organic detergents generally are the condensation products of an organic aliphatic or alkyl aromatic hydro-
30 phobic compound and hydrophilic ethylene oxide groups. Practically any hydrophobic compound having

- a carboxy, hydroxy, amido, or amino group with a free hydrogen attached to the nitrogen can be condensed with ethylene oxide or with the polyhydration product thereof, polyethylene glycol, to form a water-soluble nonionic detergent. Further, the length of the polyethenoxy chain can be adjusted to achieve the desired balance between the hydrophobic and hydrophilic elements.

- The nonionic detergent class includes the
10. condensation products of a higher alcohol (e.g. an alkanol containing about 8 to 18 carbon atoms in a straight or branched chain configuration) condensed with about 5 to 30 moles of ethylene oxide, for example, lauryl-myristyl alcohol condensed with
 15. about 16 moles of ethylene oxide (EO), tridecanol condensed with about 6 moles of EO, myristyl alcohol condensed with about 10 moles of EO per mole of myristyl alcohol, the condensation product of EO with a heart-cut of coconut fatty alcohol containing
 20. a mixture of fatty alcohols with alkyl chains varying from 10 to about 14 carbon atoms in length and wherein the condensate contains either about 6 moles of EO per mole of total alcohol or about 9 moles of EO per mole of alcohol and tallow alcohol ethoxylates
 25. containing 6 EO to 11 EO per mole of alcohol.

- A preferred group of the foregoing nonionic surfactants are the Neodol ethoxylates (Shell Co.), which are higher aliphatic, primary alcohol ethoxylates having about 5 to 20 ethyleneoxy groups per
30. mole of aliphatic primary alcohol containing about

9-15 carbon atoms, such as C₉-C₁₁ alkanol condensed with 8 moles of ethylene oxide (Neodol 91-8), C₁₂-13 alkanol condensed with 6.5 moles ethylene oxide (Neodol 23-6.5), C₁₂-15 alkanol condensed with 12 moles ethylene oxide (Neodol 25-12), C₁₄-15 alkanol condensed with 13 moles ethylene oxide (Neodol 45-13), and the like. Such ethoxamers have an HLB (hydrophobic lipophilic balance) value of about 8-15 and give good O/W (oil-in-water) emulsification, whereas ethoxamers with HLB values below 8 contain less than 5 ethyleneoxy groups and tend to be poor emulsifiers and poor detergents.

Additional satisfactory water soluble alcohol ethylene oxide condensates are the condensation products of a secondary aliphatic alcohol containing 8 to 18 carbon atoms in a straight or branched chain configuration condensed with 5 to 30 moles of ethylene oxide. Examples of commercially available nonionic detergents of the foregoing type are C₁₁-C₁₅ secondary alkanols condensed with either 9 EO (Tergitol (Registered Trade Mark) 15-S-9) or 12 EO (Tergitol 15-S-12) marketed by Union Carbide.

Other suitable nonionic detergents include the polyethylene oxide condensates of one mole of alkyl phenol containing from about 8 to 18 carbon atoms in a straight- or branched chain alkyl group with about 5 to 30 moles of ethylene oxide. Specific examples of alkyl phenol ethoxylates include nonyl condensed with about 9.5 moles of EO per mole of nonyl phenol, dodecylphenol condensed with about 12 moles of EO per

- mole of phenol, dinonyl phenol condensed with about 15 moles of EO per mole of phenol and di-isooctyl-phenol condensed with about 15 moles of EO per mole of phenol. Commercially available nonionic
5. surfactants of this type include Igepal CO-630 (nonyl phenol ethoxylate) marketed by the GAF Corporation.

- Also among the satisfactory nonionic detergents are the water-soluble condensation products of a
10. C_8 - C_{20} alkanol with a heteric mixture of ethylene oxide and propylene oxide wherein the weight ratio of ethylene oxide to propylene oxide is from 2.5:1 to 4:1, preferably 2.8:1-3.3:1, with the total of the ethylene oxide and propylene oxide (including
 15. the terminal ethanol or propanol group) being from 60-85%, preferably 70-80%, by weight. Such detergents are commercially available from BASF-Wyandotte and a particularly preferred detergent is a C_{10} - C_{16} alkanol condensate with
 20. ethylene oxide and propylene oxide, the weight ratio of ethylene oxide to propylene oxide being 3:1 and the total alkoxy content being about 75% by weight.

- Condensates of 2 to 30 moles of ethylene oxide with sorbitan mono- and tri- C_{10} - C_{20} alkanolic acid
25. esters having an HLB of 8 to 15 also may be employed as the nonionic detergent ingredient in the described shampoo. These surfactants are well known and are available from Imperial Chemical Industries under the Tween trade name. Suitable surfactants
 30. include polyoxyethylene (4) sorbitan monolaurate,

polyoxyethylene (4) sorbitan monostearate, polyoxyethylene (20) sorbitan trioleate and polyoxyethylene (20) sorbitan tristearate.

Other suitable water-soluble nonionic
5 detergents which are less preferred are marketed under the trade name "Pluronic" (Registered Trade Mark). The compounds are formed by condensing ethylene oxide with a hydro-
phobic base formed by the condensation of propylene
10 oxide with propylene glycol. The molecular weight of the hydrophobic portion of the molecule is of the order of 950 to 4,000 and preferably 1,200 to 2,500. The addition of polyoxyethylene radicals to the hydrophobic portion tends to increase the solubility
15 of the molecule as a whole so as to make the surfactant water-soluble. The molecular weight of the block polymers varies from 1,000 to 15,000, and the polyethylene oxide content may comprise 20% to 80% by weight. Preferably, these surfactants will
20 be in liquid form and satisfactory surfactants are available as grades L 62 and L 64.

The anionic surfactant, which is an essential ingredient of the liquid detergent compositions of the present invention constitutes about 1% to 10%,
25 preferably 2%-8%, most preferably 3%-6%, by weight thereof and provides good foaming properties. However, preferably reduced amounts are utilized in order to enhance the mildness to the skin property desired in the compositions of the present
30 invention, and thus, the weight ratio of nonionic detergent to anionic should exceed about 3:1. In

addition, the particular group of anionic surfactants utilized excludes the C₈-C₁₈ alkyl polyethenoxy ether sulphate surfactants in order to avoid the dioxane toxicity associated with the process of sulphation of ethoxylated alcohols. Thus, the said ethoxylated alcohol ether sulphates are expressly excluded from the specific group of anionic surfactants utilized.

The anionic surfactants which may be used in the nonionic based, liquid detergent of the present invention are water soluble and include the sodium, potassium, ammonium and ethanolammonium salts of C₈-C₁₈ alkyl sulphates such as lauryl sulphate, myristyl sulphate and the like; linear C₈-C₁₆ alkyl benzene sulphonates; C₁₀-C₂₀ paraffin sulphonates; alpha olefin sulphonates containing about 10-24 carbon atoms; C₈-C₁₈ alkyl sulphoacetates; C₈-C₁₈ alkyl sulphosuccinate esters; C₈-C₁₈ acyl isethionates; and C₈-C₁₈ acyl taurates. Preferred anionic surfactants are the water soluble C₁₂-C₁₆ alkyl sulphates, the C₁₀-C₁₅ alkylbenzene sulphonates, the C₁₃-C₁₇ paraffin sulphonates and the alpha C₁₂-C₁₈ olefin sulphonates.

The fourth essential ingredient of the nonionic based liquid detergents according to the present invention is a fatty acid monoethanolamide which functions as a foam stabilizer in amounts of about 5 0.5-8%, preferably 2%-6%, most preferably 3%-5%, by weight of the composition. Useful compounds in this group include monoethanolamides of higher fatty acids having about 10-18 carbon atoms. Specific examples of suitable monoethanolamides include 10 cocomonethanolamide, cocodi-ethanolamide, lauric monoethanolamide, and lauric-myristic monoethanolamide (a C₁₂-C₁₄ fatty acid monoethanolamide), with the latter being particularly preferred.

15 All of the aforesaid four ingredients in this light duty liquid detergent are water soluble or water dispersible and remain so during storage.

The four essential ingredients discussed above are solubilized in an aqueous medium comprising 20 water and, optionally, solubilizing ingredients such as C₂-C₃ mono- and di-hydroxy alkanols, e.g. ethanol, isopropanol and propylene glycol, or water soluble salts of C₁-C₃ alkyl substituted benzene sulphonate hydrotropes, e.g. sodium xylene 25 sulphonate, sodium cumene sulphonate and potassium toluene sulphonate, or mixtures of the said C₂-C₃ alkanols and the said C₁-C₃ substituted benzene

- sulphonates. Suitable water soluble hydrotropic salts include sodium, potassium, ammonium and mono-, di- and tri-ethanolammonium salts. While the aqueous medium is primarily water, preferably the
5. said solubilizing agents are included in order to control the viscosity of the liquid composition and to control low temperature cloud-clear properties. Usually, it is desirable to maintain clarity to a
10. the proportion of solubilizer generally will be from about 1%-15%, preferably 2%-12%, most preferably 3%-8%, by weight of the detergent composition with the proportion of ethanol, when present, being 5% by weight or less in order to provide a composition
15. having a flash point above about 46°C. Preferably the solubilizing ingredient will be a mixture of ethanol and either sodium xylene sulphonate or sodium cumene sulphonate or a mixture of the said sulphonates.
20. The foregoing solubilizing ingredients also facilitate the manufacture of the compositions of the present invention because they tend to inhibit gel formation.
25. Another preferred optional ingredient in the compositions of the present invention is a water soluble sodium, potassium or triethanolammonium formate. The formate salts tend to inhibit irreversible gel formation in the final liquid composition when the temperature is reduced to about
30. 5°C to 8°C. Generally the concentration of the

formate will be from 0.5%-6%, preferably 1%-5%, by weight of the nonionic liquid detergent composition.

In addition to the previously mentioned essential and optional constituents of the light duty liquid detergent, one may also employ normal and conventional adjuvants, provided they do not adversely affect the properties of the detergent. Thus, there may be used various colouring agents and perfumes; ultraviolet light absorbers such as the Uvinul (Registered Trade Mark), which are products of GAF Corporation; sequestering agents such as ethylene diamine tetra-acetates; magnesium sulphate heptahydrate; preservatives such as formaldehyde or hydrogen peroxide; pearlescing agents and opacifiers; pH modifiers; etc. The proportion of such adjuvant materials, in total, will normally not exceed 15% by weight of the detergent composition, and the percentages of most of such individual components will be a maximum of 5% by weight and preferably less than about 2% by weight.

The present nonionic based light duty liquid detergents such as dishwashing liquids are readily made by simple mixing methods from readily available components which, on storage, do not adversely affect the entire composition. However, it is preferred that the nonionic surfactant be mixed with the solubilizing ingredients, e.g. ethanol and sodium xylene sulphonate, if present, prior to the addition of the water to prevent possible gelation. The nonionic based surfactant system is prepared by

- sequentially adding with agitation the anionic surfactant, the betaine and ethanolamide to the nonionic surfactant which has been previously mixed with a solubilizing agent such as ethyl alcohol
5. and/or sodium xylene sulphonate to assist in solubilizing the said surfactants, and then adding with agitation the formula amount of water to form an aqueous solution of the nonionic based surfactant system. The high level of fatty acid alkanolamide
10. may cause clouding of the compositions below room temperature, which can be rectified by increasing the level of the sodium xylene sulphonate and/or the ethyl alcohol content. The use of mild heating (up to 100°C) assists in the solubilization of the
15. surfactants. The viscosities are adjustable by changing the total percentage of active ingredients. Usually, no thickening agent is added, but thickeners may be added if higher viscosity liquids are desired. In all such cases the product
20. made will be pourable from a relatively narrow mouth bottle (1.5 cm diameter) or opening, and the viscosity of the detergent formulation will not be so low as to be like water. The viscosity of the detergent desirably will be at least 100 centipoises
25. (cps) at room temperature, but may be up to about 1,000 centipoises as measured with a Brookfield Viscometer using a number 1 spindle rotating at 12 rpm. Its viscosity may approximate those of commercially acceptable detergents now on the
30. market. The detergent viscosity and the detergent

itself remain stable on storage for lengthy periods of time, without colour changes or settling out of any insoluble materials. The pH of this formation is substantially neutral, e.g. about 6 to 8 and preferably about 7.5.

These products have unexpectedly desirable properties. For example, the foam quality and deterative property is equal to or better than standard light duty liquid detergents while using a nonionic surfactant as the primary surfactant and minimal amounts of anionic surfactant, thereby achieving a mild, non-irritating liquid detergent.

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.

In the examples and elsewhere throughout the specification and claims all parts and percentages are by weight unless otherwise indicated.

EXAMPLE 1-12

Ingredient

<u>Example</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
Neodol 91-8	19	19	-	-	-	-	-	-	-	-	-	-
Neodol 91-6	-	-	19	-	-	-	-	-	-	-	-	-
Neodol 23-6.5	-	-	-	19	16	-	-	-	-	-	-	-
Neodol 25-12	-	-	-	-	-	19	-	-	-	-	-	-
Neodol 45-11	-	-	-	-	-	-	19	-	-	-	-	-
Tergitol 15-S-9	-	-	-	-	-	-	-	19	-	-	-	-
Tergitol 15-S-12	-	-	-	-	-	-	-	-	19	-	-	-
Igepal CO-630	-	-	-	-	-	-	-	-	-	19	-	-
Plurafac B-26	-	-	-	-	-	-	-	-	-	-	19	-
Ultrawet N*	-	-	-	-	-	-	-	-	-	-	-	19

* see note 8 to Table 4.

<u>Ingredient</u>		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
<u>Example</u>													
Ammonium lauryl	(C12-C16)	6	2	6	6	6	6	6	6	6	6	6	6
5.	sulphate												
	Cocoamidopropyl-	4	4	4	4	4	4	4	4	4	4	4	4
	dimethyl												
	betaine												
	Lauric-myristic	5	5	5	5	5	5	5	5	5	5	5	5
10.	monoethanolamide												
	Ethanol	5	5	5	5	5	5	5	5	5	5	5	5
	Sodium xylene	7	7	7	7	7	7	7	7	7	7	7	7
	sulphonate												
	Water	Balance											

15.

The nonionic surfactant, ethanol and sodium xylene sulphonate are mixed together until homogeneous and clear, at room temperature or at slightly elevated temperatures (a maximum of

20. 100°C). The ammonium lauryl sulphate, the betaine and the ethanolamide are then added with agitation, followed by the addition of the water, while agitating the mixture to form an aqueous solution of the nonionic based surfactant system. If clouding
25. occurs, additional ethanol and/or sodium xylene sulphonate is added which clarifies the solution immediately.

- These nonionic surfactant-based detergent formulations exhibit high foaming properties and are
30. effective in cleaning. Hand dishwashing evaluation

indicated that, these formulations are equivalent to or better than two leading dishwashing liquids when used in cleaning dishes soiled with both Crisco (Registered Trade Mark) shortening and Ragu spaghetti sauce soils as shown in Table 1.

TABLE 1

	<u>Composition</u>	<u>Number of plates washed</u>	
		<u>Crisco Soil</u>	<u>Ragu Spaghetti Sauce Soil</u>
10	Example 1	20	41
	Example 2	17	27
	Example 3	20	38
	Example 4	17	32
	Example 5	16	27
15	Example 6	17	34
	Example 7	15	32
	Example 8	16	33
	Example 9	16	32
	Example 10	18	37
20	Example 11	17	31
	Example 12	19	38
	Leading anionic Brand A ^a	16	33
	Leading anionic Brand B ^b	19	29

Notes on Table 1

25 a) b) See notes on Table 6.

Other satisfactory liquid nonionic detergent compositions are set forth in Examples 13-16 which follow, with the composition of Example 13 being a particularly preferred composition.

EXAMPLE 13A, 13B and 13CExample 13A

<u>Composition</u>	<u>% by weight</u>
Neodol 91-8	19
5. Ammonium C ₁₂ -C ₁₆ alkyl sulphate	6
Cocoamidopropyl dimethyl betaine	4
Lauric-myristic monoethanolamide	4
Sodium formate	2
Sodium xylene sulphonate	2.4
10. Sodium cumene sulphonate	0.5
Ethanol	1.2
Magnesium sulphate heptahydrate	1.0
Water, perfume, salts	<u>q.s.</u>
	100.0

15.

The composition of Example 13A exhibits a viscosity of 225 cps at 24°C and has a cloud point below 5°C and a clear point below 16°C.

- When the ethanol and sodium cumene sulphonate are omitted (Example 13B) from the composition of Example 13A, the viscosity increases to 300 cps at 24°C. On the other hand, when an additional 1% of ethanol (Example 13C) is included in the composition of Example 13A, the viscosity is reduced to 115 cps.

25.

EXAMPLES 14-16

<u>Composition</u>	<u>% by weight</u>		
<u>Example</u>	<u>14</u>	<u>15</u>	<u>16</u>
Neodol 91-8	14	16	8
Ammonium C ₁₂ -C ₁₆ alkyl sulphate	2	4	1
30. Cocoamidopropyl dimethyl betaine	2	3	0.5
Lauric-myristic monoethanolamide	2	3	0.5
Sodium xylene sulphonate	1.2	1.9	0.3
Water, salt	<u>q.s.</u>	<u>q.s.</u>	<u>q.s.</u>
	100.0	100.0	100.0

Variations in the above formulations may be made. For example, other anionic surfactants may be substituted for the ammonium lauryl sulphate such as sodium lauryl sulphate, potassium lauryl sulphate, linear dodecyl benzene sulphonate and the like. Similarly, other ethanolamides may be substituted for the lauric/myristic monoethanolamides such as cocomonethanolamide, cocodiethanolamide, lauric myristic diethanolamide and the like.

In addition, the amounts of each of the ingredients may be varied within the parameters set forth herein.

The omission of one or more ingredients adversely affects foaming as shown in Tables 2 and 3 using the well known Ross-Miles foam test described in "Oil and Soap", 18, pages 99-102, (1941). 0.1% test solutions of detergent compositions are run in a Ross-Miles foam column, and the foam height is recorded.

TABLE 2
Foam Generation (room temperature approx. 20°C)

5.	Ex.	Detergent Composition	Foam Height (mm)		
			Oppm	Tap Water (@ 100ppm)	300 ppm
	17	N91-8 ¹ (30% AI)	60	68	60
	18	16/6			
		N91-8/ALS ²	112	110	105
	19	16/6/4			
10.		N91-8/ALS/Betaine ³	140	143	120
	20	16/6/4/3			
		N91-8/ALS/Betaine/ LMMEA ⁴	143	152	142
	21	Leading Brand of	122	146	103
15.		Commercial LDLD ⁵			

Notes on Table 2

- ¹ Neodol ethoxylate (Shell Co.) containing 8 ethylene oxide groups per mole of C₉-C₁₁ aliphatic alcohol.
- ² Ammonium lauryl sulphate (65% C₁₂ alkyl, 28% C₁₄ alkyl and 7% C₁₆ alkyl by weight).
- ³ Cocoamidopropyl dimethyl betaine.
- ⁴ Lauryl myristyl monoethanolamide.
- ⁵ Containing 30% by weight of anionic surfactants and 4% by weight of fatty acid alkanolamide.

25.

TABLE 3
Foam Generation (50°C)

		<u>Foam Height (mm)</u>		
		<u>Tap Water</u>		
		<u>(@ 100ppm) 300 ppm</u>		
5.	<u>Ex. Composition</u>	<u>Oppm</u>		
	17 N91-8 (30% AI)	75	95	70
	18 16/6			
	N91-8/ALS	126	132	125
	19 16/6/4			
10.	N91-8/ALS/Betaine	134	145	141
	20 16/6/4/3			
	N91-8/ALS/Betaine/ LMMEA	163	170	148
	21 Leading Brand of	149	174	135
15.	Commercial LDLD			

These results clearly show that the omission of one or more of the ingredients from the nonionic based detergent composition of the present invention greatly reduces the foaming and fails to afford satisfactory foaming. It is also noted that the compositions of the present invention exhibit better foaming than a light duty liquid detergent presently on the market which contains about 30% by weight of a mixture of anionic alkyl benzene sulphonate and anionic alkyl polyethenoxy ether sulphate detergents and about 4% by weight of a fatty acid alkanolamide.

This particular combination of anionic surfactant and betaine surfactant, together with the fatty acid alkanolamide foam stabilizer, provides a

- detergent system which coacts with the nonionic surfactant to produce a liquid detergent composition with desirable foaming, foam stability, deterative properties and mildness to human skin. Surprisingly,
5. the resultant homogeneous liquid detergent exhibits the same or better foam performance, both as to initial foam volume and stability of foam in the presence of soils, and cleaning efficacy as an anionic based light duty liquid detergent (LDLD) as
10. shown in the following Table 4 based upon the Hand Dishwashing test. In this test, ceramic dinner plates having a diameter of nine and one-half inches soiled with about 4 grams of Crisco(R) or about 15 grams of Ragu(R) spaghetti sauce soil are washed at
15. thirty second intervals in a dish pan containing either six grams (0.1%) or 12 grams (0.2%) of liquid composition dissolved in six litres of water of a selected hardness at a temperature of about 46°C. (Six grams of detergent are employed when each plate
20. is soiled with Ragu(R) spaghetti sauce and twelve grams are employed when each plate is soiled with Crisco at the beginning of the test.) A layer of foam is generated by allowing the six litres of water to fall from a separatory funnel mounted
25. sixteen inches (40.6 cms) above the bottom of the dish pan into a Petri dish containing the liquid composition to be tested which is located in the centre of the dish pan. The Petri dish is removed carefully and the foam height is measured prior to
30. the start of the test. A soiled dish is placed in

- the solution every thirty seconds and is washed by the operator for 10 to 15 seconds while holding it about half in and half out of the solution. Washing continues until about one half of the surface of the dish pan is covered with foam. Usually, a control is run at the same time as the test product in order to eliminate any differences due to different operators. Results are reproducible and a difference of two plates is considered to be significant.

TABLE 4
Performance Evaluation of Various Nonionic Surfactants

15.	<u>Nonionic based System</u>		<u>Hand Dishwashing Performance (No. of plates washed)</u>	
	<u>Ex.</u>	<u>19/6/4/5 X/ALS/Betaine/LMMEA</u>	<u>Crisco</u>	<u>Ragu Sauce</u>
	22	X = Neodol 91-8	20	41
	23	X = Neodol 91-6	20	38
20.	24	X = Neodol 23-6.5	17	32
	25	X = Neodol 25-12	17	34
	26	X = Neodol 45-11	15	32
	27	X = Tergitol 15-S-9	16	33
	28	X = Tergitol 15-S-12	16	32
25.	29	X = Igepal CO-630 ⁶	18	37
	30	X = Plurafac B-26 ⁷	17	31
	31	X = Ultrawet N ⁸	19	38
	<u>Anionic Based System</u>			
	32	17/13/4		
30.		LDBS ⁹ /AEOS(3) ¹⁰ /LMMEA	15	34
	33	16/6/4/3		
		AEOS (6.5)/ALS/Betaine/LMMEA	17	33

Notes on Table 4

Neodol (Shell): Primary alcohol ethoxylates.

Tergitol (Union Carbide): Secondary alcohol ethoxylates.

5. 6 Ethoxylated (9.5) nonylphenol.
- 7 Condensate of ethylene oxide and propylene oxide on C₁-C₁₆ alkanol (EO/PrO = 3:1 and total alkylene oxide = 75%).
- 8 Modified alkanol ethoxylate.
10. 9 Sodium linear dodecyl benzene sulphonate.
- 10 Ammonium C₁₂-C₁₅ alkyl (C₂H₄O)₃ sulphate.

- The performance results with the hand dish-washing evaluation summarized in Table 4 clearly
15. show that the nonionic surfactant-based formulations perform equivalent to or better than the anionic based systems.

- It has been found that satisfactory performance can also be achieved with the present nonionic based
20. liquid detergent at reduced levels of anionic surfactant in order to obtain better mildness; and at lower levels of ethanolamides in order to avoid clouding of the composition, as shown in Table 5.

25.

TABLE 5

			Hand Dishwashing Performance (No. of plates washed)	
	<u>Ex.</u>	<u>Ingredients</u>	<u>Crisco</u>	<u>Ragu Sauce</u>
5.	22	19/6/4/5 Neodol 91-8/ALS/Betaine/ LMMEA	20	41
	34	19/4/4/5 Neodol 91-8/ALS/Betaine/ LMMEA	17	37
10.	35	19/2/4/5 Neodol 91-8/ALS/Betaine/ LMMEA	17	32
	36	19/6/4/4 Neodol 91-8/ALS/Betaine/ LMMEA	17	36
	37	19/6/4/3 Neodol 91-8/ALS/Betaine/ LMMEA	16	32
15.	38	34 Neodol 91-8	3	7

Amounts as low as 2% anionic surfactant (Example 35), exhibit good performance and amounts as low as 3% ethanolamide (Example 37) exhibit good performance, particularly as compared with a composition containing 34% by weight of Neodol 91-8 nonionic detergent.

The nonionic surfactant-based formulation offers the following benefits in addition to similar or better performance:

- Eliminates the potential dioxane toxicity problem associated with manufacture of alkanol ethoxamer-sulphates;
- Leaves more possibilities for further improvement in mildness;

Leaves more room for cost-saving.

Soil removal performance, as an evaluation of cleaning efficiency for dishwashing or general application, is superior to known products presently

5. on the market as shown in Table 6, using a static soaking test. A soil-containing plate (0.5 g of Crisco^R soil) which has been aged for 1 1/2 minutes is soaked for 30 seconds in a warm (50°C) aqueous test solution of 150 ppm hardness and 100 ppm
10. alkalinity which contains 0.1% detergent and is immediately transferred to an ice-water bath to stop the soil removing process. The unremoved soil is solidified on the plate which is air dried and % SR (Soil Removal) is calculated as:

15.

$$\% \text{ SR} = \frac{\text{Amount of Soil Removed}}{\text{Original Amount of Soil}} \times 100\%$$

20.

TABLE 6
Static Soak Test

<u>Ex.</u>	<u>Detergent</u>	<u>% of Soil Removal</u>
39	Commercial LDLD Leading Brand A ^a	33
25. 40	Commercial LDLD Leading Brand B ^b	54
41	Neodol 91-8 (30% AI)	2.5
42	Neodol 23-6.5 (30% AI)	19
43	16/6/4/3	
30. 44	N 91-8/ALS/BEC ^c /LMMEA	73
	16/6/4/3	
	N 91-8/ALS/BEC ^c /LMDEA	56
45	16/10/3	
	N 91-8/AEOS(3)/LMMEA	50

Notes on Table 6

- a) Contains 17% of sodium dodecylbenzene sulphonate, 13% of ammonium C₁₂-C₁₅ alkyl triethenoxy ether sulphate and 4% of fatty acid alkanolamide.
- 5 b) Contains approximately 15% of C₁₂-C₁₄ polyethenoxy (12) ether sulphate, 8% of C₁₂-C₁₄ ethenoxy (1) ether sulphate, 3% ammonium lauryl sulphate, 5% lauryl dimethyl amine oxide, 1% alkyl glyceryl ether sulphonate and 1% of nonionic.

- 10 c) BE - C₈-C₁₈ alkylamidopropyl dimethyl betaine.

It is also noted that comparative Example 45 which contains the ethoxylated alkyl ether sulphates exhibits poorer soil removal results than Examples 43 and 44 which are representative of the present invention.

15

The present invention is also suitable for laundry application when high foaming is desirable such as in the case of hand washing.

Oily soil detergency performance, as an evaluation of comparative cleaning efficiency for laundry application, is shown in Table 7. A number of dacron/cotton swatches of 65/35 fabric content are soiled with 3 drops of the following soils on separate swatches and labelled accordingly:

20

- 25
1. O/T/E - oleic acid/Triolene/Eicogene
 2. DMO - dirty motor oil
 3. Sebum - Spanglers sebum
 4. Nujol (Registered Trade Mark) - Mineral oil.

The swatches are washed in a targetometer bucket, containing 0.96 gms detergent per litre tap water at

30

- room temperature, for ten minutes. The swatches are removed from the bucket and rinsed with cold water, dried in a dryer, and the reflectance, R_d , of each swatch is read, using a Macbeth or Radio Shack computer. The higher R_d value indicates better detergency and generally a difference of about 2 R_d units is significant because it can be perceived by the eye.

TABLE 7
Oil Soil Detergency

	<u>Ex.</u>	<u>Detergent</u>	<u>Rd</u>			
			<u>Sebum</u>	<u>DMO</u>	<u>O/T/E</u>	<u>Nujol</u>
10.	46	Typical Laundry Liquid Brand A 32/7 N25-711/LDBS ¹²	76.1	41.1	61.6	69.2
15.	47	Typical Laundry Liquid Brand B 18/16/1 AEOS(2)/LDBS/CDEA ¹³	73.7	43.5	62.0	67.6
20.	48	16/6/4/3 N23-6.5/ALS/BE/LMMEA	73.3	40.03	65.7	70.0
	49	16/6/4/3 N91-8/ALS/BE/LMMEA	74.4	41.4	65.4	70.0
	50	16/10/3 N91-8/LDBS/LMMEA	72.5	42.3	62.0	67.9
25.	51	16/10/3 N91-8/LTBS/LMMEA	72.2	43.7	62.0	68.4
	52	16/6/4/3 N91-8/ALS/BE/LMDEA	72.7	39.3	64.7	68.3
30.	53	16/10/3 N91-8/AEOS(3)/LMMEA	71.8	38.8	61.1	68.2

Notes on Table 7

- 11 Neodol ethoxylate containing 7 ethylene oxide groups per mole of C₁₂-C₁₅ aliphatic alcohol.
- 12 Linear sodium dodecyl benzene sulphate.
- 5. 13 Cocodiethanolamide.

- This Table 7 clearly shows the superior detergency of Examples 48, 49 and 52 representing the present invention against O/T/E and Nujol soil, as well as the comparable detergency against Sebum and DMO soils despite a lesser total active detergent concentration than commercially available detergents (i.e. 29% in Examples 48, 49 and 52 vs. 39% and 33% in Examples 46 and 47). It is also noted that the omission of the betaine from compositions otherwise in accordance with the present invention (Examples 50 and 51) gives poorer detergency results with reference to Sebum, O/T/E and Nujol soils. The use of the ethoxylated ether sulphates which are excluded from the group of anionic surfactants utilized in the present invention yield poorer detergent compositions than the present compositions.
- 10.
 - 15.
 - 20.

- The mildness property of nonionic based detergents of the present invention is clearly shown in Tables 8 and 9, using the in vivo skin irritation test on guinea pigs.
- 25.

- In the guinea pig test, the abdomen is shaved one day prior to the initiation of the test, an appropriate concentration of the product in water selected from the range of about 0.5%-20% is selected
- 30.

- for testing and 1 cc of the test solution is applied to the two separate areas about one inch square on the shaved abdomen of the test animal. The said area is covered with a patch which is removed after
5. four hours. The foregoing procedure is repeated on the second and third days using different sites on the animal's abdomen. On the sixth day, any hair which has grown is removed with a commercial hair removing product and the test animal is thoroughly
 10. rinsed with water and dried. Four hours later each of the test sites is rated by a skilled observer for irritation, i.e. scaling, redness, cracking and visible sores, on a scale of 0-4. A rating of 0 corresponds to no irritation and a rating of 4
 15. indicates visible sores and cracking. The ultimate irritation value represents the average of six ratings. A difference in rating of 0.5 is considered to be significant. The aqueous solutions of detergents utilized in this test contain 5% ethyl
 20. alcohol and 7% sodium xylene sulphonate (SXS) in the nonionic based formula and 3% SXS in the anionic based formula.

TABLE 8
Guinea Pig Dermal Irritation

25.	<u>Ex.</u>	<u>Ingredients</u>	<u>Mean Irritation</u>	
			<u>Scores</u>	
			<u>2%</u>	<u>3%</u>
	54	19/6/4/4.5		
		Neodol 91-8/ALS/Betaine/LMMEA	1.3*	2.2*
30.	55	Commercial LDLD ^b Leading Mild-	2.9	3.8
		ness Brand		

Note on Table 8

* Significantly different from Leading brand of mild LDLD.

TABLE 9Guinea Pig Dermal Irritation

5

<u>Ex.</u>	<u>Ingredients</u>	<u>Mean Irritation</u>	
		<u>Scores</u>	
		<u>2% conc.</u>	<u>3% conc.</u>
56	19/6/4/4.5		
10	N91-8/ALS/Betaine/LMMEA	2.0	2.0**
57	19/2/4/5	0.66**	2.0**
	N-91-8/ALS/Betaine/LMMEA		
58	Mild Anionic based detergent ¹⁴	2.0	2.8

15

Notes on Table 8

¹⁴ 16/6/4/3 AES-6.5 EO/ALS/Betaine/LMMEA

** Significantly different from the mild anionic based detergent (14).

20

The results of this test clearly show that all the nonionic based detergents are significantly less irritating than the commercial brand leading in mildness and the anionic based detergent, with Example 57 (Table 9) containing only 2% anionic surfactant being the least irritating product at 2% concentration.

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CLAIMS

1. A nonionic based, light duty liquid detergent consisting essentially of as essential ingredients;

5 A) 8 to 30% of a water soluble, ethoxylated, nonionic surfactant as the major detergent ingredient;

10 B) 1 to 10% of a foaming anionic surfactant selected from the group consisting of water soluble organic sulphates and organic sulphonates, excluding the ethoxylated C₈-C₁₈ alkyl ether polyethenoxy sulphates;

C) 0.5 to 8% of a foaming, water soluble, C₈-C₁₈ alkyl amido propyl dimethyl betaine; and

15 D) 0.5 to 8% of a C₁₀-C₁₈ fatty acid mono-ethanolamide foam stabilizer; dissolved in

20 E) an aqueous vehicle, the sum of A)-D) being from 10% to 55% by weight of the composition and the said nonionic detergent being in excess of 50% by weight of the said sum.

2. A nonionic surfactant based, light duty, liquid detergent consisting essentially of by weight

25 (A) 13% to 25% of a water soluble nonionic surfactant selected from the group consisting of primary and secondary C₈-C₁₈ alkanol condensates with 5 to 30 moles of ethylene oxide, condensates of C₈-C₁₈ alkylphenol with 5 to 30 moles of ethylene oxide, condensates of C₈-C₂₀ alkanol with a heteric mixture of ethylene oxide and propylene oxide having

30



a weight ratio of ethylene oxide to propylene oxide from 2.5:1 to 4:1 and a total alkylene oxide content of 60% to 85% by weight and condensates of 2 to 30 moles of ethylene oxide with sorbitan mono- and tri-
5 C₁₀-C₂₀ alkanolic acid esters having an HLB of 8 to 15;

(B) 2% to 8% of a water-soluble anionic detergent selected from the group consisting of C₈-C₁₈ alkyl sulphates, C₈-C₁₆ alkylbenzene sulphonates,
10 C₁₀-C₂₀ paraffin sulphonates, C₁₀-C₂₄ alpha olefin sulphonates, C₈-C₁₈ alkyl sulphoacetates; C₈-C₁₈ alkyl sulphosuccinate esters, C₈-C₁₈ acyl isethionates and C₈-C₁₈ acyl taurates;

(C) 2% to 6% of a foaming, water soluble,
15 C₈-C₁₈ alkyl amido propyl dimethyl betaine;

(D) 2% to 6% of a C₁₂-C₁₄ fatty acid mono-ethanolamide foam stabilizer;
solubilized in

(E) an aqueous medium; the sum of A-D being
20 from 20% to 40% by weight of the composition and the said nonionic detergent being in excess of 50% by weight of the said sum.

3. A liquid detergent composition as claimed
25 in Claim 1 or Claim 2 which includes, in addition, 1% to 15% by weight of a solubilizing agent selected from the group consisting of C₂-C₃ mono- and di-hydroxy alkanols, water soluble salts of C₁-C₃ alkyl substituted benzene sulphonate hydrotropes and
30 mixtures thereof.

4. A liquid detergent composition as claimed in Claim 1, 2 or 3 in which ethanol is present in the amount of 5% by weight or less.



5. A liquid detergent composition as claimed in Claim 1, 2, 3 or 4 in which the said nonionic surfactant is a condensate of a primary C₈-C₁₈ alkanol with 5-30 moles of ethylene oxide.
- 5.
6. A liquid detergent composition as claimed in any one of Claims 1 to 5 in which the said anionic detergent is selected from the group consisting of C₁₂-C₁₆ alkyl sulphates, C₁₀-C₁₅ alkyl-
10. benzene sulphonates, C₁₃-C₁₇ paraffin sulphonates and C₁₂-C₁₈ alpha olefin sulphonates.
7. A liquid detergent composition as claimed in any one of Claims 1 to 6 in which the nonionic
15. surfactant is present in an amount of 16% to 22% by weight, the said anionic detergent is present in an amount of 3% to 6% by weight and each of the said betaine and the said fatty acid alkanolamide are present in an amount of 2% to 6% by weight.
- 20.
8. A liquid detergent composition as claimed in any one of Claims 1 to 7 which includes, in addition, 1% to 5% by weight of sodium, potassium or triethanolammonium formate.
- 25.
9. A liquid detergent composition as claimed in any one of Claims 1 to 8 in which the said nonionic detergent is a condensate of a primary C₈-C₁₈ alkanol with 5-30 moles of ethylene oxide, the
30. said anionic detergent is a C₁₂-C₁₆ alkyl sulphate.



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

5. 11. A method of preparing the liquid detergent as claimed in any one of the preceding claims which comprises the steps of first mixing the said nonionic surfactant with the solubilizing agent, sequentially adding with agitation the said anionic surfactant, the said betaine and the said fatty acid alkanolamide, and lastly adding with agitation, the formula amount of water to form an aqueous solution of the nonionic based surfactant composition.
- 10.
15. 12. A liquid detergent as claimed in any one of Claims 1 to 10 when made by a method as claimed in Claim 11.
-

20.