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(54) LIQUID DETERGENT COMPOSITIONS

(71) We, COLGATE-PALMOLIVE COMPANY, a Corporation organised under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to liquid detergent compositions and provides such a composition which is substantially builder-free and biodegradable yet which functions comparably to conventional phosphate-built or nitrilotriacetate-built heavy duty laundry detergents for laundering soiled clothing and other cloth or fabric articles.

In recent years there has been a sharply increasing awareness of possible disruptive effects on the ecology attending discharge of various pollutants into ground waters, streams, rivers and lakes. Particularly undesirable is the large scale discharge of compounds which persist in the environment and adversely affect the quality of such waters. As a result of governmental and individual concern, manufacturers of detergent compositions are attempting to eliminate non-biodegradable synthetic organic detergent materials, e.g. branched chain alkyl benzene sulphonates, as the organic surface active agents in commercially available household laundry detergents. Along with such research, there are extensive efforts to replace the polyphosphate builder constituents in such detergent compositions so that the wash water discharges from household and commercial laundries will not cause accumulations of phosphates in rivers, lakes and other waters.

The elimination of polyphosphates from commercial and household detergents has not been without its attendant difficulties, since these materials have very desirable building, sequestering and soil-suspending activities and the removal of these materials from the detergent compositions may result in significant losses in cleaning power. A detergent composition comprising only active synthetic organic detergents does not produce the same cleaning effect as a combination of these detergents and polyphosphates. At the present time, the only substitute for the polyphosphates which appears to be approximately equal with regard to building and soil-suspending effects are nitrilotriacetic acid and its acetate salts, often referred to generically as NTA.

According to the present invention a liquid detergent composition which is suitable for heavy duty laundering and is clear, non-gelling and substantially devoid of phosphate and nitrogenous builders comprises:—

(a) a detergent mixture of nonionic detergent and anionic detergent in a weight ratio in the range from 15:1 to 1:1, the nonionic detergent comprising from 100 to 50% by weight based on the weight of the nonionic detergent of a C₁₀—C₁₅ fatty alcohol ethoxylate containing from 6 to 11 moles of condensed ethylene oxide per mole of fatty alcohol, with the proviso that the number of moles of condensed ethylene oxide is from 2/5 to 8/11 of the average number of carbon atoms in the fatty alcohol, and from 0 to 50% by weight of a C₁₄—C₂₀ fatty alcohol ethoxylate containing from 10 to 14 moles of condensed ethylene oxide per mole of condensed fatty alcohol, with the proviso that the number of moles of condensed ethylene oxide is from 7/10 to 1 times the number of carbon atoms in the fatty alcohol, and the anionic detergent comprising a sulphated ethoxylated C₁₆—C₂₀ fatty alcohol containing 2 to 6 moles of condensed ethylene oxide per mole of condensed fatty alcohol;

(b) sufficient lower alkanol (as hereinafter defined) to solubilize the detergent mixture; and

(c) water;

the liquid detergent composition being substantially neutral in water.

The present liquid detergent compositions are based on a combination of an ethoxamer sulphate as the anionic detergent component, and a nonionic detergent both of which are readily biodegradable. However, the use of nonionic detergents which are biodegradable is not without its attendant drawbacks. Thus, the use of nonionic detergents in liquid detergent compositions must be balanced against the tendency of these materials to gel upon standing in open contact with the atmosphere. Furthermore, some nonionic detergents, subsequent to prolonged contact with the atmosphere, are not readily dispersible in water and will gel upon contact with water. The present invention is based on the discovery that by utilizing certain specific nonionic detergents this gelling phenomenon is reduced or eliminated altogether, thereby permitting nonionic biodegradable detergents having deterative properties similar to prior art alkaline built detergent compositions to be prepared in the absence of polyphosphates and other inorganic builder salts or alkaline materials. Although the composition of the present invention may include a small amount of NTA, or the like, acting as a metal sequestrant, to prevent discoloration of the composition and the laundry washed, such an amount of NTA and the like is too small to have a significant building effect on the composition.

In the clear liquid composition, various constituents aid in solubilizing other components so that the product is clear and concentrated yet readily pourable. Furthermore, repeated test washings indicate that the cleaning power of the compositions of the invention is approximately equivalent to that of other commercial built detergent compositions in cleaning action against realistic body soil and actual laundering tests in home laundry washing machines and against mixed loads under various conditions.

At least 50% by weight of the nonionic detergent components of the compositions of the present invention is a low molecular weight nonionic having the formula



wherein R is an alkyl group, preferably a straight chain alkyl group, having from 10 to 15 carbon atoms, and may comprise either one specific chain length or a mixture of up to all six, n is from 6 to 11 with the proviso that n is from 2/5 to 8/11 of the average number of carbon atoms in R. The remainder of the nonionic detergent comprises a higher molecular weight nonionic having the formula



wherein R' is an alkyl group, preferably a straight chain alkyl group, averaging from 14 to 20 carbon atoms and m is from 10 to 14 with the proviso that m is from 7/10 to 1 times the number of carbon atoms in R'.

The anionic component of the composition is an anionic ethoxamer sulphate, i.e. a polyethenoxy sulphate of the formula



wherein R'' is a fatty alkyl group of from 10 to 20 carbon atoms, p is from 2 to 6 and preferably from 1/5 to 1/3 of the number of carbon atoms in R'', and M is a salt-forming ion, usually selected from alkali metals, ammonium, lower alkylamino and lower alkanolamino.

The composition may include a minor proportion of a fluorescent brightener system that is normally at least partially water-soluble, the proportions of the non-ionic and anionic detergents, the fluorescent brightener system, the water and the alcohol preferably being such that the fluorescent brightener system is substantially in solubilized form.

The compositions of the invention preferably comprise from 25% to 75% weight of nonionic detergent, from 5 to 25% by weight of anionic detergent, from 0.5 to 5% by weight of fluorescent brightener system, from 5 to 35% by weight of water and 5 to 35% by weight of the lower alkanol.

The nonionic detergent which provides the composition with its non-gelling characteristics is the low molecular weight nonionic condensation product of fatty alcohol with ethylene oxide or ethylene glycol optionally containing a small proportion of propylene glycol. Normally, this condensation will be with ethylene oxide which is cheaper and does not require the removal of by-product water. Methods for

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a of tergent tergents use of inst the osphere. with the h water. specific together, operties absence though TA, or tion and a signi-	5 10 15 20 25 30 35 40 45 50 55	5 10 15 20 25 30 35 40 45 50 60	the manufacture of such compounds are well-known and these materials have been previously employed in detergent compositions, although generally their use has been limited to being part of the entire active organic detergent portion of light duty liquid detergent composition. Generally, due to the methods of manufacture, mixed alkyls are employed in the low molecular weight nonionic component, and for the composition to have the desired non-gelling characteristics it is preferred to utilize mixed alkyl chains wherein the chain length averages approximately 12. Furthermore, the ethylene oxide which reacts with these mixed alkyls may also vary somewhat within the above-noted range of from 6 to 11. The ethylene oxide content can vary within this range as long as the molar amount of ethylene oxide is from 2/5 to 8/11 of the average number of carbon atoms in the alkyl group. Since it is critical to maintain the proper balance of fatty alkyl chain length and ethylene oxide chain length within the above-noted ranges in order to obtain good deterative properties together with the non-gelling property and compatibility with other materials, it is preferred to utilize a nonionic detergent of the formula $RO(C_2H_4O)_nH$ wherein R is a mixed straight chain alkyl having from 10 to 15 carbon atoms, the average number of carbon atoms being approximately 12, and n is from 6 to 9 with the proviso that n is from 2/5 to 3/5 of the average number of carbon atoms in R. Preferred nonionics which have a minimum of gel-forming characteristics include "Neodol 25-7" (R=mixed 12-15 carbon atom alkyl, n=11 average value) made by Shell Chemical Company, United States of America, and "Plurafac B-26" (Plurafac is a trade mark), the reaction product of a linear alcohol with a mixture of ethylene and propylene oxides, made by Wyandotte Chemical Company, United States of America. The nonionic detergent portion of the heavy duty liquid detergent of the present invention may also contain up to 50% by weight of the aforesaid high molecular weight nonionic detergent. As with the lower molecular weight nonionics which impart the non-gelling characteristics to the composition of the present invention, these higher molecular weight nonionics are also produced by reacting mixed alkyls with ethylene oxide chains which include different chain lengths within the 10 to 14 ethylene oxide radical range. Within this range, as noted above, there is a desirable hydrophilic-lipophilic balance which is necessary in order to obtain good deterative properties together with other desirable properties and compatibility with other detergent composition ingredients. Generally, the alkyl group will be from 14 to 16 carbon atoms and usually the average carbon content of the alkyl groups of preferred compounds are from 14 to 15, and in the most preferred higher molecular weight nonionic detergent compositions for use with the lower molecular weight nonionics, the alkyl groups will be over 80% 14 to 15 carbon atom chain lengths. Similarly, it is preferable to have from 10 to 12 ethylene oxide radicals per chain and in a most preferred embodiment these will average about, and very preferably be equal to, 11 ethylene oxide units per chain. A preferred higher molecular weight nonionic is "Neodol 45-11" (R'=mixed 14 to 15 carbon atom alkyls, m=11 average value) made by Shell Chemical Company, United States of America. The desired hydrophilic-lipophilic balance is maintained by keeping m equal to about 7/10 to 1 times R'. In addition to the chain lengths of the hydrophilic-lipophilic portions of the high molecular weight and low molecular weight nonionic detergents described above, for best detergency and biodegradability it is important that such portions be of certain configurations. Of necessity, the ethylene oxide chain will be linear and terminated by a free hydroxyl group. Also, as in the case of "Plurafac B-26", some mixed chains of ethylene oxide and propylene oxide may be utilized, although pure ethylene oxide chains are generally preferred. The alkyl groups also most preferably will be linear, although a minor degree of branching such as at a carbon next to or two carbons removed from the terminal carbon of a straight chain and away from the ethoxy chain may be tolerated, provided that such a branch is of no more than 3 carbon atoms in length. Usually, the number of carbon atoms in such branched configuration will be only a small proportion, rarely being more than 20 or 10%, of the total carbon atom content of the alkyl groups. Also, although linear alkyls which are terminally joined to the ethylene oxide chains are highly preferred and result in best detergency, biodegradability and other important properties including non-gelling characteristics, medial or secondary joiner to the ethylene oxide in the chain may occur in a minor proportion of such alkyls, but generally such proportion will be less than 20% and preferably less than 10%. With regard to the inclusion	5 10 15 20 25 30 35 40 45 50 55 60

of minor quantities of propylene oxide in the ethylene oxide chain, it is preferred that the ethylene oxide chain consists predominantly of ethylene oxide, generally over 80% and preferably over 90% thereof.

The anionic detergent component of the compositions of the present invention, namely the sulphated ethoxylated higher fatty alcohol of the formula



is most readily biodegradable and of better detergency when the fatty alkyl is terminally joined to the polyoxyethylene chain, which, of course, is terminally joined to the sulphur atom of the sulphate group. Again, although slight branching of the alkyl chain may be tolerated to the extent of not more than 10% of the carbon atom content, as noted above with regard to the nonionic detergents, it is even more important to minimise deviation from the linear structure in the case of the anionic detergents. Furthermore, medial joinder of the alkyl group to the polyoxyethylene chain should be minimal, i.e. less than 10%, and even such joinder then should be preferably concentrated near the end of the alkyl chain. Within the 10 to 20 carbon atom alkyl groups, the preferred alkyls are of 12 to 15 carbon atoms and those which are most preferred are mixed alkyls containing 12, 13, 14 and 15 carbon atoms chains. The mixture is preferably one containing at least 10% of each chain length and no more than 50% of any one chain length.

The ethylene oxide content of the anionic detergent is such that p is from 2 to 6 and is preferably from 2 to 4, generally averaging about 3, especially when R'' is a mixed 12 to 15 carbon atom alkyl group. To maintain a desired hydrophilic-lipophilic balance, when the carbon content of the alkyl chain R'' is in the lower portion of the 10 to 20 range, the ethylene oxide content may be reduced so that p is about 2, whereas when R'' is of 16 to 18 carbon atoms, p may be from 4 to 6.

The salt-forming cation may be any suitable solubilizing metal or radical but will most frequently be alkali metal or ammonium. If alkylamine or lower alkanolamine groups are present, alkyls and alkanols thereof will usually contain 1 to 4 carbon atoms, and the amines and alkanolamines may be mono-, di- or tri-substituted, e.g. monoethanolamine, diisopropanolamine or trimethylamine.

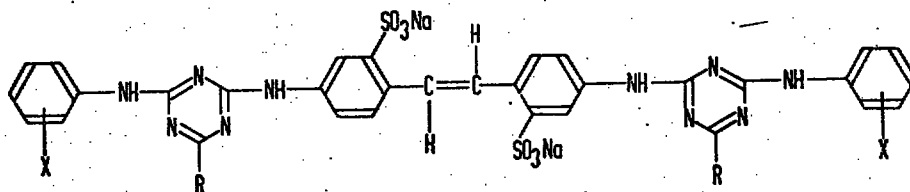
The importance of using the correct anionic detergent in the present compositions is shown by the failure of corresponding alcohol sulphates in otherwise similar liquid detergent compositions to wash as well as the present compositions containing the desired higher alcohol-ethylene oxide sulphates. For example, a higher alcohol sulphate in which the alcohol is a mixed 12 to 15 carbon atoms alcohol, exhibits a significantly poorer detergency in liquid compositions otherwise like those of the present invention, compared to the corresponding ethoxylated sulphate in the corresponding composition. Even within the preferred range of alcohol-ethylene oxide sulphates, an improvement in detergency is noted for the compositions which include a mixed 12 to 15 carbon atom alcohol-ethylene oxide sulphate, when compared to other higher alcohol-ethylene oxide sulphates, such as a mixed 14 to 15 carbon atom alcohol-ethylene oxide sulphate of the same ethylene chain length. The preferred detergent is available from Shell Chemical Company, United States of America, and is identified by them as "Neodol 25-3S", the sodium salt, normally, sold as a 60% active material, including about 40% of aqueous solvent medium, of which a minor proportion is ethanol. Although "Neodol 25-3S" is the sodium salt, the potassium salt and other suitable soluble salts may also be used either in partial or complete substitution for that of sodium.

Examples of higher alcohol polyethenoxy sulphates which may be used as the whole or a part of the anionic detergent constituent of the present liquid composition include: mixed C_{12} - C_{15} normal primary alkyl triethenoxy sulphate, sodium salt; myristyl triethenoxy sulphate, potassium salt; n-decyl diethenoxy sulphate, diethanolamine salt; lauryl diethenoxy sulphate, ammonium salt; palmityl tetraethenoxy sulphate, sodium salt; mixed C_{14} - C_{15} normal primary alkyl mixed tri- and tetraethenoxy sulphate, sodium salt; stearyl pentaethenoxy sulphate, trimethylamine salt; and mixed C_{10} - C_{18} normal primary alkyl triethenoxy sulphate, potassium salt. Minor proportions of the corresponding branched chain and medially alkoxyated detergents, such as those described above but modified to have ethoxylation at a medial carbon atom, e.g. one located 4 carbons from the end of the chain, may be employed, but the carbon atom content of the higher alkyl will be the same. Similarly, the joinder of a normal alkyl may be at a secondary carbon one or two carbon atoms from the end of the chain. In either case, only the minor proportions previously mentioned will be present.

For most of the heavy duty liquid detergents it will be unnecessary and undesirable to utilize active detergent ingredients other than the high and low molecular weight nonionics and anionic combination described above. However, for some applications minor proportions of supplementary detergents may be used. These will generally be of the nonionic type although in some compositions other anionic, cationic, amphoteric or ampholytic detergents may be employed. For example, there may be used higher fatty acid esters of polyethylene glycols; block copolymers of ethylene oxide and propylene oxide; higher alkyl-di-lower alkyl amine oxides; the sodium salts of the sulphuric acid derivatives of other higher fatty alcohol condensation products with ethylene oxide; triethanolamine lauryl sulphate; straight chain alkyl sulphonates; sodium lauroyl sarcoside; cetyltrimethylammonium bromide; benzethonium chloride; dimethyl dibenzyl ammonium chloride; N-higher alkyl N, N-di-lower alkyl aminopropane sulphonates; amidosulphobetaines; betaines and amidobetaines. Descriptions of such additional detergents may be found in the text book *Synthetic Detergents*, by Schwartz, Perry and Berch, published in 1958 by Interscience Publishers, New York, United States of America, (see pages 25 to 143). It should be kept in mind that such materials will be employed only for specific purposes and in small proportions compared to the detergent combination of the low and high molecular weight higher fatty alcohol-ethylene oxide nonionics and the alkyl-ethylene oxide sulphate anionic, and will usually be used when a specific cleaning property thereof is desirable for a particular application.

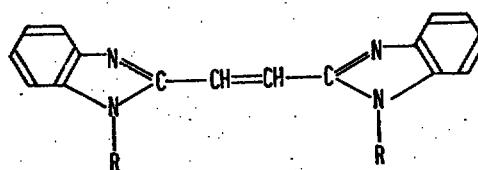
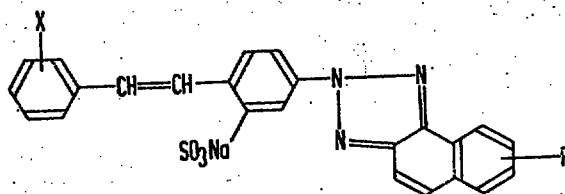
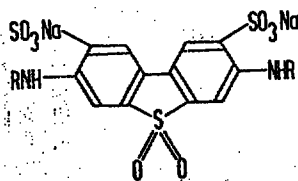
The fluorescent or optical brighteners or whiteners optionally employed are important constituents of modern detergent compositions. They give washed laundry a bright appearance so that the laundry is not only clean but also looks clean. Due to the variety of synthetic fibres incorporated in the textiles which are made into clothing and other items of laundry, and the importance of substantivity of the brightener compound to the fibres, many different fluorescent brightening compounds have been made, which may be incorporated in the present detergent compositions, often in mixtures. The term "brightener system" is used herein to mean the brightener compound or mixture of brightener compounds used in the compositions. Of course, brighteners suitable for use on cotton, and brighteners which are stable in the presence of oxidizing bleaches are also of importance. Therefore, although it is possible to utilize only a single brightener for a specific intended purpose, in the compositions of the present invention it is generally desirable to utilize a mixture of brighteners which will have good brightening effects on cotton, nylons, polyester, and blends of such materials and which, additionally, are bleach stable. A description of such types of optical brighteners is given in the articles, *Optical Brighteners and Their Evaluation*, by Per S. Stensby, reprint of articles published in *Soap and Chemical Specialties* in April, May, July, August and September, 1967, especially at pages 3-5 thereof.

The cotton brighteners, frequently referred to as CC/DAS brighteners because of their derivation from the reaction product of cyanuric chloride and the disodium salt of diamino-stilbene disulphonic acid in molar proportion of 1:2, are bistriazinyl derivatives of 4,4'-diaminostilbene-2,2'-disulphonic acid. In most cases, the CC/DAS is reacted with two moles of aniline or sulphanilic or metanilic acid and the product is additionally substituted on the triazine rings. Such compounds are of the general formula:—



wherein X may be either hydrogen or SO_3Na and the R's may be those derived from aniline, morpholine, N-methylethanolamine or diethanolamine.

Because some heavy duty laundry detergents will be employed in conjunction with bleach, a bleach-stable brightener, usually a benzidine sulphone disulphonic acid, a naphthotriazolylstilbene sulphonc acid or a benzimidazolyl derivative will be used. These compounds are respectively of the formulae:—

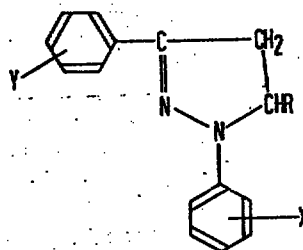
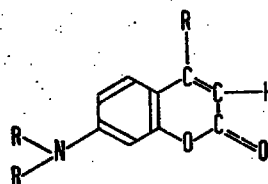


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wherein R may be hydrogen or a substituent such as lower alkyl, lower alkanol, lower aminoalkanol, anilino or morpholino.

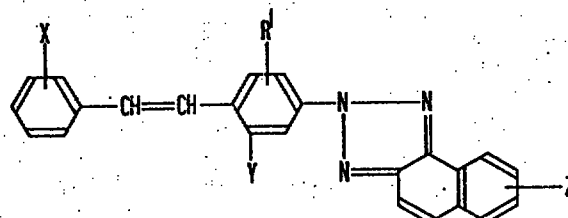
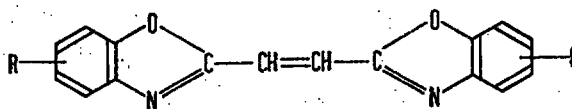
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The polyamide brighteners, especially good for nylons, are usually either amino-courmarin or diphenyl pyrazoline derivatives. Unfortunately, neither of these is adequately stable in the presence of chlorine bleaches. The compounds are of the formulae given below and the R groups thereof may be those previously listed as substituents on the optical brighteners discussed earlier, while X and Y may be substituents such as halogens and lower alkyls.



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The polyester brighteners, which are becoming of more importance as polyesters are being used to a greater extent in an increasing proportion of textiles, are usually of one of the following formulae, wherein R, X and Y have the meaning previously given in relation to the optical brightener formulae and Z may be selected from the X and Y substituents. These brighteners also usually serve to whiten polyamides.



5 The brighteners may be used in their acid forms or as salts. They may be em-
ployed as solids or in solutions and may be diluted with a carrier powder. Although
the chemical and physical forms of the brighteners can affect their brightening actions,
if the compounds are used in soluble forms the brightening activities for the same
compounds on an active ingredient basis will be equivalent. In the present composi-
tions and in the wash waters resulting, the brighteners are maintained sufficiently
soluble to the effective and uniformly substantive to the materials of the laundry being
washed.

10 Among the brighteners that may be used in brightener systems for the present
compositions are :— "Calcoflour White ALF" (American Cyanamid); "ALF-N"
(American Cyanamid); "SOF A-2001" (CIBA); "CDW" (Hilton-Davis); "Phorwite
RKH" (Verona); "CSL, powder, acid" (American Cyanamid); "FB 766" (Verona);
15 "Blancophor PD" (GAF); "UNPA" (Geigy); "Tinopal RBS" (Geigy); and "RBS
200" (Geigy). The acid or "nonionic" forms of the brighteners tend to be solubilized
by the alcohol of the present compositions, while the salts tend to be water-soluble.
Thus, a combination of such solvents and the detergent combination serve to keep
the fluorescent brighteners dissolved.

20 The lower alkanol employed is a C₂ or C₃ monohydric alcohol, or a C₂ or C₃
dihydric or trihydric alcohol, or a C₁ to C₄ alkyl ether of such dihydric or trihydric
alcohol. Ethanol and isopropanol are the preferred lower alkanols, and of these two,
ethanol is the more preferred because of a slightly greater solubilizing power and more
pleasant odour. If ethanol is used, it will normally be denatured, and of the denatured
25 alcohols those identified as "SD40" or "3A" are preferred. However, other denatured
alcohols may also be used. The alcohols need not be anhydrous and the small propor-
tions of water normally present with them are considered to be parts of the water
components of the liquid detergent composition. Some or all of the monohydric
alcohols may be replaced with said dihydric or trihydric lower alcohols which, in
30 addition to having solubilizing powers and reducing the flash points of the product,
also can act as anti-freezing constituents and may improve compatibilities of the
solvent system with particular product components. Among these compounds, the
most preferred are ethylene glycol, propylene glycol and glycerol, but the lower
alkyl (C₁—C₄) etheric derivatives of such compounds known as "Cellosolves" (Cello-
35 solve is a trade mark), may also be employed, generally to only a minor extent.

The water component of the compositions is preferably deionized so that it will
be low in content of ions which can form insoluble compounds. However, ordinary
tap water can be used provided that the hardness thereof is sufficiently low so that in
the formula employed there is no detrimental precipitation out of salts on stand-
40 ing. When sequestrants are present, hardness will be less important and in such cases
even waters with hardnesses over 300 ppm (parts per million) equivalent calcium
carbonate can be acceptable. Generally however, the water hardness should be less
than 150 ppm and most preferably less than 50 ppm.

45 The sequestering agent, when used, may be any suitable compound, including
the aminopolycarboxylic acids and hydroxycarboxylic acids. Thus, ethylene diamine
tetraacetic acid, nitrilotriacetic acid, hydroxyalkyl derivatives thereof in which the
hydroxyalkyl group replaces one or more acetic acid groups, gluconic acid, ascorbic
acid, glucono-delta-lactone (which is converted to gluconic acid), citric acid, lactic
acid and salts thereof, especially the water-soluble alkali metal salts, e.g. sodium or

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potassium. Ammonium, alkanolamine and amine salts may also be used. Other sequestering agents or water-softening agents of the inorganic type, such as certain phosphates, may be used in very small amounts if desired, but should not be present in amount sufficient to provide a builder function.

Adjuvants may be present in the liquid detergent compositions to give them additional properties, either functional or aesthetic. Thus there may be present: soil suspending or anti-redeposition agents, such as polyvinyl alcohol, sodium carboxymethyl cellulose and hydroxypropylmethyl cellulose; enzymes, e.g. protease amylases; thickeners, e.g. gums, alginates and agar agar; hydrotropes, e.g. sodium xylene sulphonate and ammonium benzene sulphonate; foam improvers, e.g. lauric myristic diethanolamide; foam destroyers, e.g. silicones; bactericides, e.g. tribromosalicylanilide and hexachlorophene; fungicides; dyes, pigments (water-dispersible); preservatives; ultraviolet absorbers; fabric softeners; pearlescing agents; opacifying agents, e.g. behenic acid and polystyrene suspensions; and perfume. Of course, such materials will be selected for the properties desired in the composition and to be compatible with the other constituents of the composition.

The pH of the heavy duty liquid detergent composition will normally be substantially neutral or only slightly alkaline. The composition of the present invention contains a minimum of alkaline material *per se*, since the ability of the particular nonionic and anionic detergents to remove particulate soil is enhanced in approximately neutral solutions. However, for various reasons, such as solubilizing and neutralizing the various brighteners used, a small amount of an alkaline material may be included. Suitable alkaline materials include mono-, di- and trialkanolamines, alkylamines, and ammonium and alkali metal hydroxides. The preferred alkaline materials are alkanolamines, especially trialkanolamines and most especially triethanolamine. A pH reading of the liquid detergent composition using a glass electrode and a reference calomel electrode, generally indicates a maximum pH of about 8. However, because the detergent system is essentially non-aqueous despite the presence of a minor proportion of water, the pH reading obtained may be false. A better indication is obtained by measuring the pH of a 25% solution in water. This should usually be in the range of about 6.8 to 8.8. In water containing items to be laundered the pH will normally be within the same range.

The proportion of the various components of the present heavy duty liquid detergents are important to the obtainment of a uniform product and acceptable heavy duty laundering action. In the absence of a significant builder content, it is very important that the product contain a significant proportion of detergents. So as to promote solubility of the fluorescent brighteners and other constituents and make a clear, homogeneous and readily pourable liquid product, generally from 25 to 75% by weight of the total liquid detergent concentrate should be nonionic fatty alcohol-ethylene oxide condensation products in a mixture of from 100% to 50% by weight of the nonionics of low molecular weight nonionic and from 0 to 50% by weight of high molecular weight nonionic as described above. The preferred range is from 30 to 60% of total nonionics and in the most preferred embodiment of the invention approximately 40% is employed. It is preferred that the nonionic mixture comprises at least a major proportion of low molecular weight nonionic since the non-gelling properties of this material are more effective at higher concentrations. Accordingly, it is preferred to utilize a nonionic mixture of from 100% to 75% by weight of the nonionics of the low molecular weight nonionic and from 0 to 25% by weight of the nonionic of high molecular weight nonionic, and it is most preferred to use all low molecular weight nonionic. For the greatest effectiveness, the quantity of the anionic ethoxamer sulphate detergent should be in the range of 5 to 20% by weight of the composition and preferably about 25% of the total nonionic content, i.e. about 10% of the whole composition. Suitable weight ratios of total nonionic to anionic are in the range from 15:1 to 1:1, with 8:1 to 2:1 being preferred and 5:1 to 3:1 most preferred.

The concentration of fluorescent brightener system in the composition will generally be from about 0.5 to 5% on an active ingredient basis, preferably about 1 to 3% and most preferably about 2%. Such quantities are readily solubilized in the clear liquid product and contribute substantially to brightening of fabrics in the laundry. Generally, 25% and preferably at least 50% of the brightener content will be a brightener for cotton. It is preferred to use 51 to 90% of a cotton brightener, with the balance being one or more of polyamide brighteners, polyester brighteners and bleach-stable brighteners.

The lower alcohol will generally be present in a sufficient proportion to aid in stabilizing or dissolving various constituents of the product, such as the brightener

Other certain be
ve them present: m carb- se amyl- a xylene ristic di- ssalicyl- reserva- ants, e.g. rials will ble with
be sub- vention articular approxi- ing and rial may s, alkyl- materials mine. A eference because or pro- a is ob- ally be the pH
y liquid ceptable nt, it is nts. So d make i 25 to ic fatty to 50% 50% by d range t of the mixture ince the trations. to 75% to 25% ost pre- ss, the of 5 to ionionic tal non- referred
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system. The proportion employed will generally be from 5 to 35%, preferably from 5 to 25%. When used without a supplementing diol or triol, the quantity of alcohol present will normally be from 5 to 20%, but when a supplementing polyol is used the content of alcohol may be reduced to the range from 5 to 15%, preferably from 5 to 10%. In such circumstances, the proportion of polyol will generally be from 5 to 15%, most preferably 10%.

The percentage of water utilized will also generally be from 5 to 35%, and a preferred range is from 5 to 25%, a most preferred range being from 15 to 20%. Similar consideration prevail in determining the proportion of water to be employed as in the case of the alcohols.

The non-building proportion of metal sequestrant, when used, may range from 0.2 to 3%, preferably from 0.5 to 1.5%, and most preferably approximately 0.8%.

Considering the wide range of adjuvants which may be utilized, for widely different purposes, the proportions employed may vary widely. Generally, however, the total thereof should not exceed 10% and will preferably be less than 5% and more preferably less than 3%. Individual components should usually not exceed 5%, preferably 3% and more preferably 1% of the product. The use of large amounts of such adjuvants will often significantly change the properties of the liquid detergent and, therefore, is to be avoided.

The heavy duty liquid detergent compositions of the present invention can be made by simple manufacturing techniques which do not require any complicated equipment or expensive operations. In a typical manufacturing method, the optical brighteners may be slurried in the monohydric alcohol or mixture of monohydric and polyhydric alcohol. If initially slurried in the monohydric alcohol, subsequently the polyhydric alcohol, if used, may be added to this. Then water and, if necessary, a small amount of a base such as triethanolamine is added, which helps partially to dissolve the previously suspended material but not yield a clear solution. Addition of the detergent combination causes the remainder of the brightener to dissolve to make a clear solution. Then the sequestrant, if used, may be added as the acid or salt (preferably the sodium, potassium or amine salt) and agitation is continued until the solution becomes clarified, which will normally take about 5 to 10 minutes. At this point perfume and dye may be added to give the product its final desired appearance and odour. All of the operations may be effected at room temperature, although suitable temperatures within the range of 10 to 80°C may be employed, as desired. Additions of adjuvants may be effected at suitable points in the process but for the most part these will be added last. The product obtained will usually have a pH within the range of from 6.5 to 8, e.g. 7.0 and a density within the range of from 0.9 to 1.1, preferably from 0.95 to 1.05 and most preferably from 0.98 to 1.02. The viscosity of the product at 25°C will usually be from 20 to 200 centipoises, preferably from 50 to 150 cps., and will be in the higher part of this range if a polyhydric alcohol is used in replacement of some lower monohydric alcohol.

Use of the present compositions is simple and efficient. Compared to present heavy duty laundry detergent powder, much smaller volumes of the present liquids may be employed for cleaning soiled laundry. For example, with a typical and preferred composition of this invention, containing about 40% of the fatty alcohol-ethylene oxide condensate and 10% ethoxamer sulphate, only about 2 ounces or 1/4 cup of liquid need to be used for a full top-loading automatic machine tub of wash, in which the water volume might be from 15 to 18 U.S. gallons. Thus, the concentration of liquid detergent in the wash water is of the order of 0.1%, 1 gram per litre or 1,000 parts per million. Generally, the proportion employed will be from 0.7 to 1.5 g/l, which may correspond to 0.3 to 0.6 g/l of the mixture of fatty alcohol-ethylene oxide condensation products, from 0.075 to 0.15 g/l of ethoxamer sulphate, from 0.01 to 0.05 g/l of the brightener system, from 0.05 to 0.4 g/l of lower monohydric alcohol and from 0.005 to 0.03 g/l of organic sequestrant. The proportions of other constituents of the liquid compositions may vary accordingly. Of course, equivalent results can be obtained by using larger proportions of a more dilute liquid detergent but the greater quantity needed will require additional packaging and shipping space and will be less convenient for the consumer to use. However, such more dilute products are within the present invention. By preliminary diluting the liquid detergent composition with water the same end result is obtained because the wash water also serves to dilute the detergent composition down to a use concentration.

Although it is preferred to employ wash water of low hardness and at an elevated temperature, the present invention is also useful in laundering clothes and other items in hard waters and in extremely soft waters, as well as in waters at room tempera-

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ture or below. Thus, water hardnesses may range from 0 to over 300 ppm as calcium carbonate and washing temperatures may be from 10°C to 80°C. Preferably, the temperatures will be from room temperature, 20 to 25°C, to 70°C. Also, although washing will ordinarily be effected in an automatic washing machine, with the washing followed by rinse and spin or draining or wringing operations, the detergent composition may also be used for hand washing of laundry. In such cases, the concentration in water of the liquid detergent composition will often be increased and sometimes it may be used full strength to assist in washing out soils or stains which are otherwise difficult to remove. After completion of the washing and spinning operations, it will generally be the practice to dry the laundry e.g. soon thereafter, in an automatic dryer, but that particular type of drying operation is not essential.

When the liquid detergent composition is added to water, whether that water is hot or cold, the composition immediately dissolves uniformly throughout the wash water, even in the absence of significant agitation. The washing and brightening agents are carried into contact with all the laundry and there are no localized over concentrations of either of these materials. The clothing washed, following normal methods, is exceptionally clean, and in comparative tests the product has been rated as good as some of the best commercial heavy duty detergent compositions on the market. Although it is a low- and non-foaming detergent composition and thus very suitable for side-loading washing machines, excellent washing is also obtained in top-loading machines in which foaming detergents are normally employed. Repeated testing of soiled and re-soiled laundry items, using the present compositions and larger quantities of commercial heavy duty detergent compositions built with phosphate or NTA, show that the soilings are repeatedly removed and no objectionable build-up thereof occurs. For the most part, users do not not any really significant differences between the washing properties of the present composition and the commercial compositions tested. In fact, there has been a significant preference for the present product.

If the low molecular weight nonionic detergents are omitted from the composition and replaced by higher molecular weight nonionic detergents having a similar hydrophilic-lipophilic balance, the liquid composition gels within a short period of time when exposed to the atmosphere. Furthermore, liquid detergent compositions without sufficient low molecular weight nonionic, i.e. at least 50% of the total nonionic detergents, to retard or eliminate this gelling do not disperse properly in water after exposure to the atmosphere. If the liquid detergent composition gels and does not disperse properly in water, this causes the brighteners to be concentrated in small areas, causing uneven and blotchy brightening.

The discovery of the present compositions and their exceptionally good properties were unexpected because those skilled in the art have not expected to be able to make a heavy duty laundry detergent composition without the use of inorganic or organic builder salts. This was especially surprising in view of the previous use of the organic detergent components of the present compositions only as light duty cleaning agents, since their ability to remove soil from laundry in heavy duty applications was unappreciated. Also, it was a conclusion contrary to that formerly reached by many other research workers in the field of detergency. The use of the compositions of the present invention without NTA has no adverse effect on the substantivity of the optical brighteners, whereas sequestrants of the NTA or polyphosphate types can interfere with the even deposition of brighteners on laundry. Surprisingly, outstanding whiteness values are obtained by the present combination of nonionics and anionics. The detergent components do not react with hypochlorite bleaches whereas NTA can undergo such reactions. Also, the various detergent constituents, as was previously mentioned, help mutually to solubilize one another and form a flowable clear liquid. The alcohols and water solubilize the brightener system and the detergent components aids in producing good solubility and a clear product. The low molecular weight nonionic prevents the liquid detergent from gelling. Gel prevention means stain prevention, because otherwise staining of the wash could occur due to deposits of gel holding the brightener to only some parts of the laundry. This liquid detergent composition is easily biodegradable and does not add large quantities of phosphate or NTA builder to the wash water.

The following Examples illustrate the invention. In the Examples and elsewhere in the specification all parts and proportions, such as percentages, are by weight unless otherwise noted.

EXAMPLE 1

A clear, liquid detergent having the following formula is prepared by slurring

the mixture of optical brighteners in "SD-40" alcohol followed by the addition of water and triethanolamine with stirring. Subsequently, the "Neodol 25-7" and the "Neodol 25-3S" are added, and after a few minutes of agitation at moderate speed the solution becomes clear:—

5	RO(C ₂ H ₄ O) ₇ H ("Neodol 25-7", R=mixed 12, 13, 14 and 15 carbon atoms primary alkyl)	40.00	5
10	R''O(C ₂ H ₄ O) ₃ SO ₃ Na (Neodol "25-3S", R''=mixed 12, 13, 14 and 15 carbon atoms primary alkyl)	10.00	
10	"SD-40" Denatured alcohol	14.00	10
15	Triethanolamine	1.5	
15	Optical Brightener A*	1.0	
	Optical Brightener B**	1.0	
	Optical Brightener C***	0.17	
	Optical Brightener D****	0.033	
	Perfume	0.35	15
	Dye	0.002	
	Water Q.S.		
		100.0	

*A=4,4' - Bis[(4 - anilino - 6 - methylamino - S - triazine - 2 - yl)amino] - 2,2'-stilbenedisulphonic acid, disodium salt.

**B=4,4' - Bis[(4 - anilino - 6 - [Bis(2 - hydroxyethyl)amino] - S - triazine - 2-yl)amino] - 2,2' - stilbenedisulphonic acid, disodium salt.

***C=4-naphthotriazolyl-2-stilbenesulphonic acid, sodium salt.

****D=2-(1,3-isindazole)-2'-(naphth[1,2-d]oxazole)stilbene.

The pH of a 25% solution of the above composition in water is about 7.0. This liquid detergent composition, which is a sparkling, clear, free-flowing liquid, is packed in polyethylene bottles of substantially cubic shape and of one U.S. quart capacity and is ready for use. Storage tests indicate that this product will be stable for several years without precipitation of insoluble salts, settling into a plurality of phases, clouding or other disadvantageous effects on appearance or function.

Furthermore, if the above composition is allowed to stand in contact with the atmosphere, it will not gel even after 18 hours exposure to the air. Further, after exposure to the air, when poured into hot or cold water it is readily dispersed therein.

The composition of Example 1 cleans better at a lower concentration than commercially available heavy duty biodegradable phosphate-free laundry detergent compositions. This composition can be utilized in a concentration which corresponds to approximately $\frac{1}{4}$ cup per 16 U.S. gallon automatic washing machine tub and at such concentration the composition compares favourably with the said commercially available compositions for which concentrations corresponding to 1- $\frac{1}{4}$ cups must be used. Also subjective evaluations of the composition of Example 1 in consumer tests using a panel of housewives showed a significant preference for this liquid detergent composition.

Since no builder is utilized, it might be thought that particulate soil could accumulate on washed items. However, subsection of cloths to repeated soilings and washings shows that the composition of Example 1 compares favourably with phosphate-containing detergent compositions with regard to particulate soil removal.

EXAMPLE 2

A series of liquid detergent compositions are produced utilizing a formula similar to Example 1 by wherein the "Neodol 25-7" is replaced by the following nonionic detergents:—

- (A) C₁₂—C₁₃ alcohol + 6.5 moles of ethylene oxide
- (B) C₁₂—C₁₅ alcohol + 9 moles of ethylene oxide
- (C) C₁₀—C₁₄ alcohol + 6 moles of ethylene oxide
- (D) C₁₁ alcohol + 8 moles of ethylene oxide

- (E) "Plurafac B-26"*
 (F) "Surfonic J-4"* (Jefferson Chemical Company, United States of America)
 (G) C_{14} — C_{18} alcohol + 11 moles of ethylene oxide
 (H) C_{14} — C_{18} alcohol + 11 moles of ethylene oxide
 (I) C_{16} — C_{18} alcohol + 11 moles of ethylene oxide
 (J) C_{12} — C_{18} alcohol + 12 moles of ethylene
 (K) 1:1 mixture of C_{14} — C_{18} alcohol + 11 moles of ethylene oxide and "Plurafac B-26"
 (L) 1:1 mixture of C_{16} — C_{18} alcohol + 11 moles of ethylene oxide and C_{12} — C_{18} alcohol + 6.5 moles of ethylene oxide.
 *linear alcohol + a mixture of ethylene oxide and propylene oxide.

When each of the above detergent compositions is tested in accordance with the open-beaker gel test, i.e. the liquid detergent composition is left in an uncovered beaker and is observed for gel formulation, compositions (A), (B), (C), (D), (E), (F), (K), and (L) do not gel after exposure to the atmosphere for 18 hours and are readily dispersible in water. However, compositions (G), (H), (I) and (J), all containing 100% higher molecular weight nonionic detergents, gel within 5 minutes exposure to the atmosphere, and because they are gelled they are not easily dispersed in water. In ungelled form, however, all the above compositions exhibit adequate cleaning and deterative characteristics when compared both to phosphate-built detergent compositions and commercial biodegradable phosphate-free detergent compositions.

EXAMPLE 3

Examples 2(K) and (L) are repeated except that the ratio of the detergent ingredients is varied from the 1:1 ratio to the following ratios of high molecular weight nonionic detergent, i.e. C_{14} — C_{18} alcohol + 11 moles of ethylene oxide or C_{16} — C_{18} alcohol + 11 moles of ethylene oxide, and low molecular weight detergent, i.e. "Plurafac B-26" and C_{12} — C_{18} alcohol + 6.5 moles of ethylene oxide, is varied as follows:—

(A)	0.1:1
(B)	0.25:1
(C)	0.5:1
(D)	0.75:1
(E)	1.25:1
(F)	1.5:1

When each of the above samples is subjected to the open-beaker gel test, Samples (A) to (D) do not gel within 18 hours and are dispersed readily upon pouring into both hot and cold water. Samples 3(E) and (F), however, become gelled within 5 minutes of exposure to the atmosphere and are not readily dispersible in hot or cold water.

EXAMPLE 4

Examples 2(A) to (F) and (K) and (L) are repeated, but varying the total amount of nonionic detergent from 40% to the following amounts, with a concomitant increase or decrease in the total amount of water and alcohol:—

(A)	20%
(B)	25%
(C)	30%
(D)	45%
(E)	50%
(F)	60%

Each of the above detergent compositions exhibited adequate deterative properties, although these did not clean quite as well as compositions wherein the amount of nonionic materials was approximately 40%.

EXAMPLE 5

Examples 2(A) to (F), (K) and (L) are repeated except that the following anionic detergent materials are substituted for "Neodol 25-3S":—

- (A) C_{10} ether sulphate containing 2 moles of condensed ethylene oxide;
 (B) C_{13} ether sulphate containing 2 moles of condensed ethylene oxide;
 (C) C_{16} ether sulphate containing 5 moles of condensed ethylene oxide;

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- (D) C_{10} ether sulphate containing 3 moles of condensed ethylene oxide;
(E) C_{14} ether sulphate containing 6 moles of condensed ethylene oxide.

Each of the above detergent compositions exhibited excellent deterative properties and was non-gelling upon standing when tested by the open-beaker gel test.

WHAT WE CLAIM IS:—

1. A liquid detergent composition which is suitable for heavy duty laundering and is clear, non-gelling and substantially devoid of phosphate and nitrogenous builders comprising:—

(a) a detergent mixture of nonionic detergent and anionic detergent in a weight ratio in the range from 15:1 to 1:1, the nonionic detergent comprising from 100 to 50% by weight based on the weight of the nonionic detergent of $C_{10}-C_{15}$ fatty alcohol ethoxylate containing from 6 to 11 moles of condensed ethylene oxide per mole of fatty alcohol, with the proviso that the number of moles of condensed ethylene oxide is from 2/5 to 8/11 of the average number of carbon atoms in the fatty alcohol, and from 0 to 50% by weight of a $C_{14}-C_{20}$ fatty alcohol ethoxylate containing from 10 to 14 moles of condensed ethylene oxide per mole of condensed fatty alcohol, with the proviso that the number of moles of condensed ethylene oxide is from 7/10 to 1 times the number of carbon atoms in the fatty alcohol, and the anionic detergent comprising a sulphated ethoxylated $C_{10}-C_{20}$ fatty alcohol containing 2 to 6 moles of condensed ethylene oxide per mole of condensed fatty alcohol;

(b) sufficient lower alkanol (as hereinbefore defined) to solubilize the detergent mixture; and

(c) water;

the liquid detergent composition being substantially neutral in water.

2. A detergent composition as claimed in Claim 1 wherein the nonionic detergent comprises a nonionic $C_{10}-C_{15}$ fatty alcohol ethoxylate containing from about 6 to 9 moles of condensed ethylene oxide per mole of condensed fatty alcohol, with the proviso that the number of moles of ethylene oxide is from 2/5 to 3/5 of the average number of carbon atoms in the fatty alcohol.

3. A composition as claimed in Claim 1 wherein the nonionic detergent comprises a mixed $C_{12}-C_{15}$ fatty alcohol ethoxylate containing about 7 moles of condensed ethylene oxide per mole of condensed fatty alcohol.

4. A detergent composition as claimed in any of the preceding Claims wherein nonionic detergent constitutes from 25 to 75% by weight of the composition; anionic detergent constitutes from 5 to 25% by weight of the composition; lower alkanol constitutes from 5 to 35% by weight of the composition and water constitutes from 5 to 35% by weight of the composition.

5. A detergent composition as claimed in Claim 4 wherein nonionic detergent constitutes approximately 40% by weight of the composition and anionic detergent constitutes approximately 10% by weight of the composition.

6. A detergent composition as claimed in any of the preceding Claims which also includes a brightener system.

7. A detergent composition as claimed in Claim 6 including from 0.5 to 5% by weight of the brightener system.

8. The detergent composition as claimed in any of the preceding Claims which also includes a metal sequestrant.

9. A detergent composition as claimed in Claim 8 which includes from 0.2 to 3% by weight of a metal sequestrant.

10. A detergent composition as claimed in any of the preceding Claims the pH of a 25% aqueous solution of which is in the range from 6.8 to 8.8.

11. A detergent composition as claimed in Claim 1 and substantially as described in any of the Examples.

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Reference has been directed in pursuance of section 9, subsection (1) of the Patents Act 1949, to patent No. 1,335,017.

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- (D) C_{10} ether sulphate containing 3 moles of condensed ethylene oxide;
 (E) C_{11} ether sulphate containing 6 moles of condensed ethylene oxide.

Each of the above detergent compositions exhibited excellent deterative properties and was non-gelling upon standing when tested by the open-beaker gel test.

WHAT WE CLAIM IS:—

1. A liquid detergent composition which is suitable for heavy duty laundering and is clear, non-gelling and substantially devoid of phosphate and nitrogenous builders comprising:—

(a) a detergent mixture of nonionic detergent and anionic detergent in a weight ratio in the range from 15:1 to 1:1, the nonionic detergent comprising from 100 to 50% by weight based on the weight of the nonionic detergent of C_{10} — C_{15} fatty alcohol ethoxylate containing from 6 to 11 moles of condensed ethylene oxide per mole of fatty alcohol, with the proviso that the number of moles of condensed ethylene oxide is from 2/5 to 8/11 of the average number of carbon atoms in the fatty alcohol, and from 0 to 50% by weight of a C_{14} — C_{20} fatty alcohol ethoxylate containing from 10 to 14 moles of condensed ethylene oxide per mole of condensed fatty alcohol, with the proviso that the number of moles of condensed ethylene oxide is from 7/10 to 1 times the number of carbon atoms in the fatty alcohol, and the anionic detergent comprising a sulphated ethoxylated C_{10} — C_{20} fatty alcohol containing 2 to 6 moles of condensed ethylene oxide per mole of condensed fatty alcohol;

(b) sufficient lower alkanol (as hereinbefore defined) to solubilize the detergent mixture; and

(c) water;

the liquid detergent composition being substantially neutral in water.

2. A detergent composition as claimed in Claim 1 wherein the nonionic detergent comprises a nonionic C_{10} — C_{15} fatty alcohol ethoxylate containing from about 6 to 9 moles of condensed ethylene oxide per mole of condensed fatty alcohol, with the proviso that the number of moles of ethylene oxide is from 2/5 to 3/5 of the average number of carbon atoms in the fatty alcohol.

3. A composition as claimed in Claim 1 wherein the nonionic detergent comprises a mixed C_{12} — C_{13} fatty alcohol ethoxylate containing about 7 moles of condensed ethylene oxide per mole of condensed fatty alcohol.

4. A detergent composition as claimed in any of the preceding Claims wherein nonionic detergent constitutes from 25 to 75% by weight of the composition; anionic detergent constitutes from 5 to 25% by weight of the composition; lower alkanol constitutes from 5 to 35% by weight of the composition and water constitutes from 5 to 35% by weight of the composition.

5. A detergent composition as claimed in Claim 4 wherein nonionic detergent constitutes approximately 40% by weight of the composition and anionic detergent constitutes approximately 10% by weight of the composition.

6. A detergent composition as claimed in any of the preceding Claims which also includes a brightener system.

7. A detergent composition as claimed in Claim 6 including from 0.5 to 5% by weight of the brightener system.

8. The detergent composition as claimed in any of the preceding Claims which also includes a metal sequestrant.

9. A detergent composition as claimed in Claim 8 which includes from 0.2 to 3% by weight of a metal sequestrant.

10. A detergent composition as claimed in any of the preceding Claims the pH of a 25% aqueous solution of which is in the range from 6.8 to 8.8.

11. A detergent composition as claimed in Claim 1 and substantially as described in any of the Examples.

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