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(54) Title: STABLE, HIGH GLYCEROL LIQUIDS COMPRISING SULFOSUCCINIC ACID MONOESTERS

(57) Abstract: The invention discloses high glycerol content (greater than or equal to 10% by wt.) liquid compositions comprising sulfosuccinic acid monoesters wherein the compositions are stable. Stability is provided by a stabilization system comprising a minimum level of a defined salt and a defined thickener. A method of enhancing stability of high content glycerin compositions is also disclosed.



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STABLE, HIGH GLYCEROL LIQUIDS COMPRISING SULFOSUCCINIC ACID
MONOESTERS

Field of the Invention

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The present invention relates to high glycerin content liquid compositions comprising one or more defined sulfosuccinic acid monoesters. More specifically, while high glycerin content has been found to destabilize such compositions, applicants have found that a combination of a minimum level of fatty acid salt (e.g., alkali metal salt or alkanolamine salt), and a thickener selected from acrylate copolymers, hydroxy alkyl cellulose, cationic guar gums (e.g., Jaguar C13S) stabilize the compositions. The compositions of the invention may be used in skin cleansing, shower gel, and hair compositions.

Compositions containing sulfosuccinic acid monoesters are disclosed in U.S. Patent No. 4,749,515 to Miyamoto et al..

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However, because of its moisturizing properties, it would also be greatly beneficial to add glycerin to such compositions to relieve skin dryness. In such compositions, however, it has been found that large amounts of glycerin (e.g., 10% by wt. and above) lead to instability and phase separation. While not wishing to be bound by theory, it is thought that this may be due to the high specific gravity of glycerin.

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Unexpectedly, applicants have now found that this instability may be overcome by using a stabilization system comprising:

- 5 (a) at least 4% by wt. of an alkali metal soap of C₁₂-C₂₄ fatty acid and/or alkanolamine soap; and
- (b) 0.01 to 5% by wt. of a thickening copolymer selected from acrylate copolymers, hydroxy alkyl cellulose (e.g., hydroxy ethyl or propyl cellulose), cationic
- 10 guar gum, and isostearic acid. Acrylate copolymers are particularly preferred.

Brief Summary of the Invention

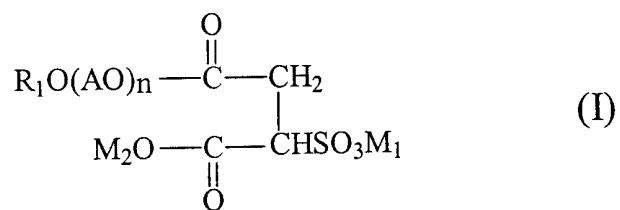
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The present invention relates to stable, high glycerin liquid detergent compositions. More particularly, the compositions comprise:

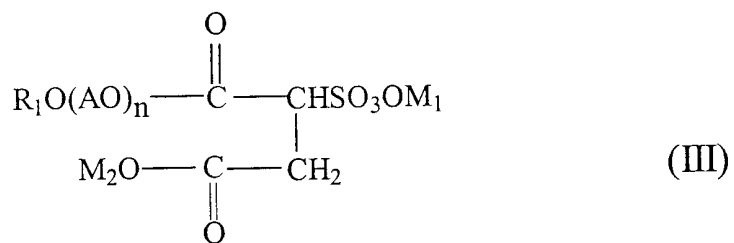
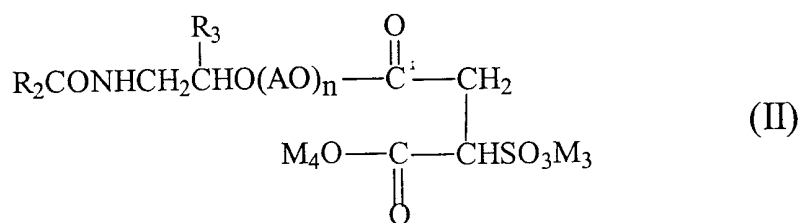
- 20 (1) 1-25%, preferably 5 to 20% of one or more sulfosuccinic acid monoesters represented by Formula (I), (II), (III) or (IV);

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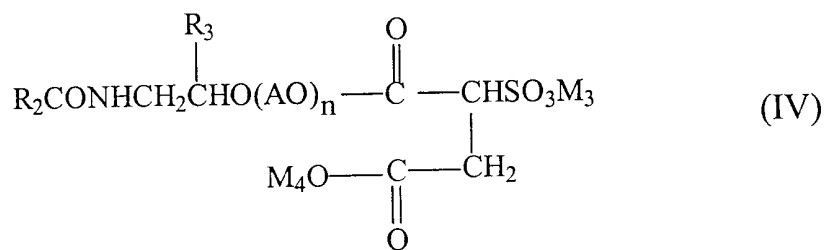
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10



or



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wherein each of M_1 to M_4 is H, NH_4 , an alkali metal or a hydroxyalkyl substituted ammonium. R_1 and R_2 are each an alkyl or hydroxyalkyl group having about 8 to 20 carbon atoms, R_3 is H or CH_3 , AO is an oxyalkylene group having 2 or 3 carbon atoms, and n represents an integer from 0 to 20;

- (2) 10% and above, preferably 10 to 50% by wt., preferably 15 to 40% by wt. of glycerin;
- 10 (3) at least 4% by wt., preferably at least 5% to 15% by wt. of an alkali metal soap of C_{12} - C_{24} fatty acid and/or alkanolamine soap;
- 15 (4) 0.01 to 5% by wt., preferably 0.1 to 3% by wt. (levels may vary depending on which thickener is used) of a thickener selected from acrylate copolymer (e.g., alkyl acrylate copolymer), hydroxy alkyl cellulose, cationic guar gum and isostearic acid; and
- 20 (5) balance water.

In a second embodiment, the invention relates to a method of enhancing the stability of high glycerin compositions comprising defined sulfosuccinic acid monoesters by adding a stabilization system comprising components (3) and (4) above.

The pH of the compositions is about 6 to 8.5, preferably 6.5 to 8.5 and viscosity is about 100 to 5000 centistokes

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preferably 100 to 200 measured using a Brookfield Viscometer, Spindle No.2 or 3, 12 rpm, for about 30 seconds at 25°C.

- 5 The stability of the composition is defined by the absence of separation after 4 weeks storage at room temperature.

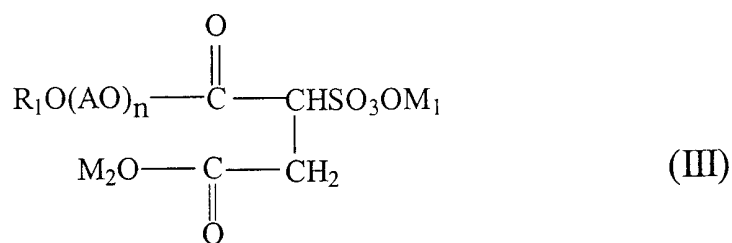
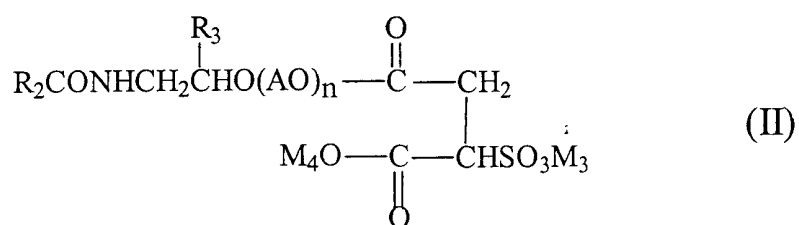
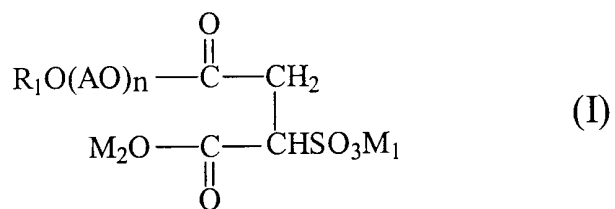
Detailed Description of the Invention

- 10 The present invention relates to liquid compositions comprising one or more defined sulfosuccinic acid monoesters and relatively high amounts of glycerin while retaining stability. More specifically, by using a combination of fatty acid salts and specific thickeners (preferably
15 acrylate copolymers and isostearic acid), it has been found that stable, high content glycerin (greater than or equal to 10% by wt. to about 50% by wt. glycerin), sulfosuccinic monoester compositions may be formed.

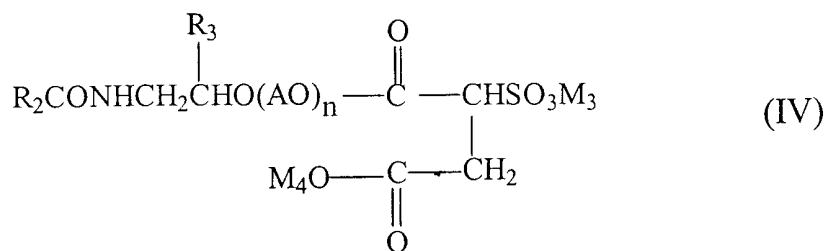
- 20 Compositions of the present invention are set forth in greater detail below.

- The compositions of the present invention comprise 1-25%, preferably 5-20% by wt. of one or more sulfosuccinic acid
25 monoesters represented by Formula I, II, III or IV and mixtures thereof as follows:

- 6 -



or



- 7 -

In these formulae, each of M_1 to M_4 may be selected from H, NH_4 , an alkali metal and hydroxyalkyl-substituted ammonium groups. M_1 and M_2 , and M_3 and M_4 may be the same or
5 different. Alkali metals may include lithium, sodium, potassium and the like. The hydroxyalkyl substituted ammonium groups, which may preferably have 1 to 3 carbon atoms in the hydroxyalkyl group, may include monoethanol ammonium, diethanol ammonium, triethanol ammonium, methyl
10 diethanol ammonium and the like. Preferably, M_1 to M_4 are hydrogen, sodium or triethanol ammonium.

R_1 and R_2 are each straight or branched alkyl or hydroxyalkyl groups having about 8 to 20 carbon atoms, for
15 example, hexyl, decyl, hydroxydecyl, dodecyl, hydroxytetradecyl, tetradecyl, nonadecyl, or the like. Any alkyl group having less than 8 or more than 20 carbon atoms may optionally be contained in the molecule, provided that the total number of carbon atoms in the sulfosuccinic acid
20 monoester contained in the detergent composition of the present invention is in the range from 8 to 20. This number of carbon atoms provides good foaming properties, whereas when the number of carbon atoms is either less than 8 or more than 20 less foaming is observed.

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In the formulae (II) and (IV), R_3 is either H or CH_3 .

In the formulae, AO represents an oxyalkylene group having 2 or 3 carbon atoms, that is, oxyethylene or oxypropylene

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group. Both oxyethylene and oxypropylene groups may be present in the molecule.

The letter n represents an integer from 0 to 20, preferably 1 to 10. If n is more than 20, the foaming properties of the resulting liquid detergent compositions may be poor.

The compounds represented by the formulae (I), (II), (III) and (IV) may be prepared by any known methods.

10

For example, the sulfosuccinic acid monoesters represented by the general formulae (I) or (II) may be prepared by reacting an alkylene oxide adduct of a higher fatty acid with maleic anhydride to produce an ester of maleic anhydride and further reacting the ester of maleic anhydride with a sulfite.

15

Alternatively, the sulfosuccinic acid monoesters represented by the general formulae (II) or (IV) may be prepared by reacting a lower alcohol ester of a higher fatty acid with an alkanolamine, adding an alkylene oxide to the reaction product, further reacting the resulting addition product with maleic anhydride to produce an ester of maleic anhydride, and reacting the ester of maleic anhydride with a sulfite.

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As part of the required stability system of the invention, compositions of the invention also require at least 4%, preferably at least 5% to about 15% by wt. of an alkali metal soap of a C₁₂-C₁₄ fatty and/or alkanolamine salt.

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Suitable fatty acid soaps include, but are not limited to, sodium or potassium salts of lauric or myristic acid. Suitable alkanolamine salts include but are not limited to ethanolamine, triethanolamine etc.

5

A second component of the stability system is a thickener. The thickener is selected from acrylate copolymers, hydroxy alkyl celluloses, cationic guar gums, and isostearic acid.

- 10 Among the acrylates which may be used are included alkyl acrylate copolymer e.g. Leoarl MS200 ex Lion and Acuryn 33 ex Rohm and Haas; Acryl copolymer e.g. Auryn 22 ex Rohm and Haas. Hydroxy alkyl celluloses include hydroxy ethyl, hydroxy propyl or hydroxy methyl cellulose. Cationic guar
15 gums include Jaguar gums.

- The thickener may be used in amounts between 0.01 to 5% by wt. although the amounts may depend on which thickener is used. For example, acrylate copolymer may comprise 0.1-3%
20 by wt. while isostearic acid may comprise from 1-5% by wt of the composition.

The balance of the composition (generally about 20-70%, preferably 25-65% by wt.) is water.

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Optional Components

Compositions of the invention may contain 0.1 to 10% by wt. of a surfactant selected from anionic, nonionic, amphoteric and cationic surfactants and mixtures thereof.

Suitable anionic detergents include, but are not limited, to alkyl and alkylene carboxylates having 10 to 20 carbon atoms, alkyl ether carboxylates, fatty alcohol sulfates, fatty alcohol ether sulfates, alkylol amido-sulfates and sulfonates, fatty acid alkylol amido-polyglycol ether sulfates, alkane sulfonates and hydroxyalkane sulfonates, olefin sulfonates, acyl esters of isethionates, α -sulfo fatty acid esters, alkyl-benzene sulfonates, alkylphenol glycol ether sulfonates, sulfo-succinates, sulfosuccinic acid half and di-esters, fatty alcohol ether phosphates, protein fatty acid condensation products, alkyl monoglyceride sulfates and sulfonates, alkyl glyceride ether sulfonates, fatty acid methyl taurides, fatty acid sarcosinates, sulfo-ricinoleates. These compounds and mixtures thereof are used in the form of water-soluble or water-dispersible salts, for example sodium, potassium, magnesium, ammonium, mono-, di- and triethanol-ammonium salts and analogous alkylol ammonium salts.

25

A particularly preferred anionic surfactant is polyoxyalkylene alkylether sulfate which may be used in an amount of, for example of 0.01 to 5% by wt.

30 Suitable nonionic surface-active agents include, for example fatty alcohol ethoxylates (alkyl-polyethylene glycols),

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alkylphenol-polyethylene glycols, alkylation-polyethylene glycols, fatty amine ethoxylates (alkylamino-polyethylene glycols), fatty acid ethoxylates (acyl polyethylene glycols), polypropylene glycol ethoxylates (Pluronic
5 (Registered Trademark)), fatty acid alkyl amides (fatty acid amino polyethylene glycols), saccharose esters, sorbitol esters, and polyglycol ether.

Suitable amphoteric surface-active agents to be added to the
10 shampoos include N-alkyl- β -amino dipropionates having 12 to 18 alkyl carbon atoms as alkali metal salts and mono-, di- and trialkylol-ammonium salts, N⁺-acylamido-alkyl-N, N-dimethylacetobetaine, preferably N-acyl-amidopropyl-N,N-dimethylacetobetaine having 8 to 18 acyl carbon atoms,
15 alkyl-dimethylsulfopropyl-betaine having 12 to 18 alkyl carbon atoms, amphoteric surfactants of the imidazoline type (Trademarks: Miranol Steinapon), preferably the sodium salt of 1-(β -carboxy-methyloxethyl)-1-(carboxymethyl)-2-lauryl-imidazolinium; amine oxides, for example alkyl-dimethyl-
20 amine oxide having 12 to 18 alkyl carbon atoms, fatty acid amidoalkyl-dimethylamine oxide.

A particularly preferred amphoteric surfactant is cocoamide
propyl betaine.

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Among suitable cationics are, for example, quaternary ammonium salts, such as dialkyl-dimethyl-ammonium, chloride or bromide having 10 to 24, preferably 12 to 18 carbon atoms, in the alkyl portion, alkyl-dimethyl-ethylammonium
30 chloride or bromide having 10 to 24 alkyl carbon atoms, alkyl-trimethyl-ammonium chloride or bromide having 10 to 24

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alkyl carbon atoms, preferably cetyl-trimethyl ammonium chloride or bromide, alkyl-trimethyl ammonium chloride or bromide having 10 to 22 alkyl carbon atoms, or alkyl-dimethyl-benzyl ammonium chloride or bromide having 10 to 24, preferably 12 to 18, carbon atoms in the alkyl portion.

Other optional components are as follows:

The liquid personal cleansing compositions of the present invention may optionally also include water-dispersible, gel-forming polymers. This polymer is preferably an anionic, nonionic, cationic or hydrophobically modified polymer, selected from cationic polysaccharides of the cationic guar gum class with molecular weights of 1,000 to 3,000,000, anionic, cationic and nonionic homopolymers derived from acrylic and/or methacrylic acid, anionic, cationic and nonionic cellulose resins; cationic copolymers of dimethyldialkyl ammonium chloride and acrylic acid; cationic homopolymers of dimethyldialkyl ammonium chloride; cationic polyalkylene and ethoxypolyalkylene imines polyethylene glycol having molecular weights from 10,000 to 4,000,000; and mixtures thereof. Preferably, the polymer is selected from Sodium Polyacrylate, Hydroxy Ethyl Cellulose, Cetyl Hydroxy Ethyl Cellulose, and Polyquaternium 10.

25

The polymer is preferably included in the compositions of the present invention at a level of from about 0.1 parts to 1 part, more preferably 0.1 parts to 0.5 parts of the composition. The polymers improve the sensory feel of the lipid on skin in addition to providing product stabilization. The improved sensory feel results from

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reduced tackiness and greasiness and improved smoothness. In an especially preferred embodiment a mixture of polymers is used, for example those polymers preferred for product stabilization, and/or those preferred for improved sensory
5 feel. Preferred polymers for improved sensory feel are selected from polyethylene glycol, hydroxypropyl guar, guar hydroxypropyl trimonium chloride, polyquaternary 3, 5, 6, 7, 10, 11 and 24 and mixtures thereof.

10 A variety of additional ingredients may be incorporated into the compositions of the present invention. These materials include, but not limited to, liquid appearance aids, salts and their hydrates and other "filler materials" as listed in U.S. Patent No. 5,340,492 to Kacher et al., and U.S. Patent
15 No, 4,919,934 to Deckner et al. ,both of which are herein incorporated by reference.

Other non-limiting examples of these additional ingredients include vitamins and derivatives thereof (e.g., ascorbic
20 acid, vitamin E, tocopherol acetate, and the like); sunscreens; thickening agents (e.g., polyol alkoxy ester, available as Crothix from Croda at levels up to 2% and xanthan gum at levels up to 2%); preservatives for maintaining the anti-microbial integrity of the
25 compositions; anti-acne medicaments (resorcinol, salicylic acid, and the like); antioxidants; skin soothing and healing agents such as aloe vera extract, allantoin and the like; chelators (e.g., EDTA and hydroxy ethan diphosphoric acid) and sequestrants; and agents suitable for aesthetic
30 purposes such as fragrances, essential oils, skin sensates, pigments, pearlescent agents (e.g., mica and titanium

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dioxide), additives to impart a draggy rinse feel (e.g., fumed silica), additives to enhance deposition (e.g., maleated soybean oil at levels up to 3%), lakes, colorings, and the like (e.g., clove oil, menthol, camphor, eucalyptus oil and eugenol).

The compositions of the present invention have a pH of about 6 to 8.5, preferably 6.5 to 8.5.

10 Compositions of the present invention have a viscosity of 100-5000 centistokes measured using a Brookfield Viscometer, Spindle 2 and 3 at 12 rpm for 30 seconds at 25°C.

15 The compositions of the present invention exhibit no precipitation or phase separation after 4 weeks of storage at room temperature (i.e., about 25°C).

EXAMPLES

20 Examples 1, 2 and 3

The following examples were prepared as follows:

- 15 -

	Example1	Example2	Example3
Disodium Laurylether sulfsuccinate(DSLES)	4.25%	5%	7%
Disodium Lauryl sulfsuccinate(DSLS)	4.25%	5%	7%
Potassium Laurate		3%	3%
Potassium Myristate		2%	2%
Lauric acid	4.25%		
Isostearic acid			4%
Potassium hydroxyde	0.6%		
Cocamido propyl betaine(CAPB)	2.6%	3%	0.6%
Sodium Laurylether sulfate(SLES)	1.7%	2%	0.4%
Glycerine	20%	20%	20%
Dipropylene glycol		3%	
Amphoteric polymer	0.3%		0.3%
Cationic polymer		0.6%	
Alkyl acrylate polymer	0.5%	0.5%	0.5%
Ethylene glycol distearate(EGDS)	2%	2%	2%
Dibutylhydroxy toluene(BHT)	0.05%	0.05%	0.05%
EDTA	0.05%	0.05%	0.05%
Hydroxy ethane diphosphoric acid		0.2%	0.2%
Preservative	0.3%	0.3%	0.3%
Perfume	0.7%	0.7%	0.7%
Water	To 100%	To 100%	To 100%

Processing: Example 1

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Example 1 above was processed as follows:

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- 1) A mixture of glycerin, water, potassium hydroxide and SLES was heated to 75-80°C until becoming quite fluid(mixture-1);
- 2) Lauric acid, preservative and BHT were added to mixture-1 with agitation(mixture-2);

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- 3) DSLES, DSLS and CAPB were added to mixture-2 with agitation(mixture-3);
- 4) A mixture of EDTA and water was mixed at 75-80°C (mixture-4);
- 5 5) A mixture of amphoteric polymer and water was added into mixture-4 with agitation (mixture-5);
- 6) EGDS and alkyl acrylate polymer were separately added to mixture-5 with agitation(mixture-6);
- 7) Mixture-6 was cooled to 45°C;
- 10 8) Perfume was added to mixture-9 and cooled to 35°C.

Examples 5,6 & Comparative Example A

	Example 5	Comparative A	Example 6	Comparative A
Disodium Laurylether sulfsuccinate(DSLES)	7%	7%	4.25%	4.25%
Disodium Lauryl sulfsuccinate(DSLS)	7%	7%	4.25%	4.25%
Potassium Laurate	3%	3%		
Potassium Myristate	2%	2%		
Lauric acid			4.25%	4.25%
Isostearic acid	4%			
Potassium hydroxyde			0.6%	0.6%
Cocamido propyl betaine(CAPB)	0.6%	0.6%	2.6%	2.6%
Sodium Laurylether sulfate(SLES)	0.4%	0.4%	1.7%	1.7%
Glycerine	20%	20%	20%	20%
Dipropylene glycol				
Amphoteric polymer	0.3%	0.3%	0.3%	0.3%
Cationic polymer				
Alkyl acrylate polymer	0.5%	0.5%	0.5%	
Ethylene glycol distearate(EGDS)	2%	2%	2%	2%
Dibutylhydroxy toluene(BHT)	0.05%	0.05%	0.05%	0.05%
EDTA	0.05%	0.05%	0.05%	0.05%
Hydroxy ethane diphosphoric acid	0.2%	0.2%		
Preservative	0.3%	0.3%	0.3%	0.3%
Perfume	0.7%	0.7%	0.7%	0.7%
Water	To 100%	To 100%	To 100%	To 100%
RT stability:	good	no good	good	no good
Separation				

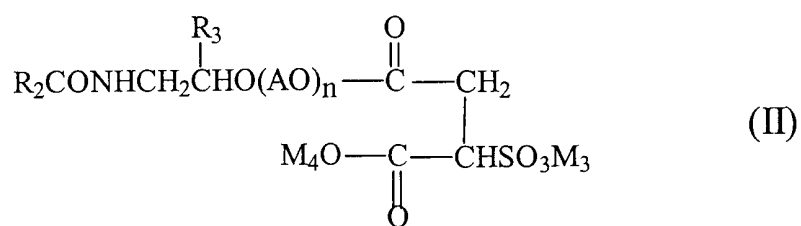
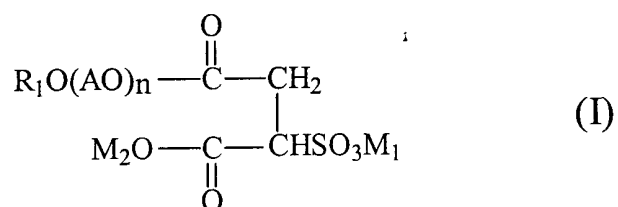
5 From the data obtained for Example 5 and Comparative A, it can be seen that 4% isostearate provided good stability (no phase separation) while an absence of isostearic acid (Comparative A) resulted in instability. Similarly, the use of alkyl acrylate (Example 6) resulted in stability while

10 the absence (Comparative B) resulted in instability.

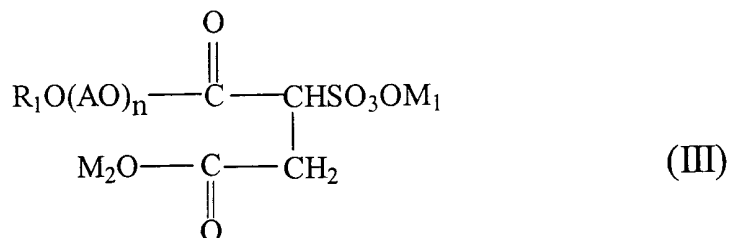
CLAIMS

5 1. A stable, high glycerol liquid detergent composition comprising:

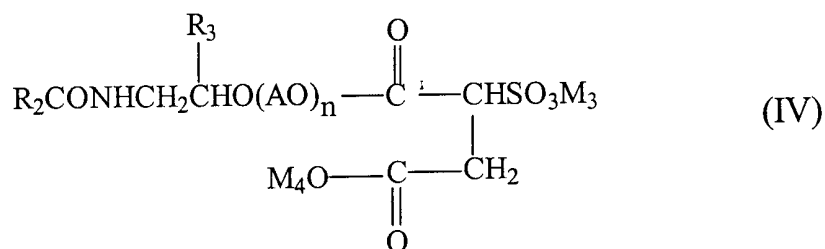
(a) 1-25%, by wt. of one or more sulfosuccinic acid monoesters represented by Formulae (I), (II), (III) or (IV).



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or



wherein each of M_1 to M_4 is H, NH_4 , an alkali metal or a hydroxyalkyl substituted ammonium; R_1 and R_2 are each an alkyl or hydroxyalkyl group having about 8 to 20 carbon atoms; R_3 is H or CH_3 ; AO is an oxyalkylene group having 2 or 3 carbon atoms, and n represents an integer of 0 to 20;

(b) 10% to 50% by wt. glycerin;

(c) at least 4% by wt. of an alkali metal soap of C_{12} - C_{24} fatty acid and/or alkanol soap; and

(d) 0.01-5%, by wt. of a thickener selected from acrylate copolymer, hydroxy alkyl cellulose, cationic guar gums and isostearic acid; and

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(e) balance water.

2. A composition according to claim 1, wherein the
5 composition additionally comprises 0.01-10% of a
surfactant selected from anionic, nonionic and
amphoteric and cationic surfactants and mixtures
thereof.
- 10 3. A composition according to 2, wherein the amphoteric
surfactant is a betaine.
4. A composition according to any one of claims 1 to 3,
wherein the pH of the composition is about 6-8.5.
- 15 5. A composition according to any of the preceding claims,
wherin the composition has a viscosity of 100 to 5000
centistokes.
- 20 6. A composition according to any of the preceding claims,
wherein the composition has no precipitation or phase
separation after 4 weeks at room temperature.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/11359

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/48 A61K7/50 C11D1/32

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 130 056 A (JAKOBSON GERALD ET AL) 14 July 1992 (1992-07-14) claims 1,6,9,13,23 ---	1-5
X	US 5 132 037 A (GREENE ALAN P ET AL) 21 July 1992 (1992-07-21) column 2, line 63; claims ---	1-5
P,X	EP 0 995 433 A (JOHNSON & JOHNSON CONSUMER) 26 April 2000 (2000-04-26) claims 1,4; example 7 ---	1,2
P,X	WO 00 06677 A (BANKS DAVID M ; SMITH KEVIN L (US); BOSSE MICHAEL J (US); MEYER MAR) 10 February 2000 (2000-02-10) claims 1,4,11,13,28 --- -/--	1



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

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Date of the actual completion of the international search

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PCT/EP 00/11359

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 99 29409 A (BETZDEARBORN INC) 17 June 1999 (1999-06-17) claims 1,9,10,15 ----	1
X	US 4 284 534 A (EHRlich JOSEPH R) 18 August 1981 (1981-08-18) claims 1,2,11 ----	1
A	US 5 939 378 A (ABBAS SYED HASAIN ET AL) 17 August 1999 (1999-08-17) claim 5 ----	1
X	DATABASE WPI Section Ch, Week 199109 Derwent Publications Ltd., London, GB; Class A21, AN 1991-060270 XP002163989 & HU 54 191 A (OFFSET ES JATEKKART), 28 January 1991 (1991-01-28) abstract ----	1
X	JP 10 237494 A (KANEBO LTD) 8 September 1998 (1998-09-08) abstract -----	1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/11359

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 5130056	A	14-07-1992	DE 3902374 A EP 0379658 A JP 2233800 A	02-08-1990 01-08-1990 17-09-1990
US 5132037	A	21-07-1992	AU 634777 B AU 5561790 A BR 9007347 A CA 2015868 A,C DE 69010907 D DE 69010907 T EP 0479795 A ES 2057564 T WO 9013283 A JP 2514112 B JP 7501785 T US 5290471 A US 5234619 A ZA 9003408 A	04-03-1993 29-11-1990 28-04-1992 05-11-1990 25-08-1994 24-11-1994 15-04-1992 16-10-1994 15-11-1990 10-07-1996 23-02-1995 01-03-1994 10-08-1993 29-01-1992
EP 0995433	A	26-04-2000	AU 5262899 A BR 9904718 A CN 1252990 A JP 2000143500 A	20-04-2000 28-11-2000 17-05-2000 23-05-2000
WO 0006677	A	10-02-2000	AU 5235399 A US 6140285 A	21-02-2000 31-10-2000
WO 9929409	A	17-06-1999	US 5942219 A AU 8595198 A	24-08-1999 28-06-1999
US 4284534	A	18-08-1981	GB 2086407 A,B	12-05-1982
US 5939378	A	17-08-1999	US 5858955 A	12-01-1999
HU 54191	A	28-01-1991	NONE	
JP 10237494	A	08-09-1998	NONE	