



12

EUROPEAN PATENT APPLICATION

21 Application number : **91301046.8**

51 Int. Cl.⁵ : **C11D 1/12, C11D 10/04,
C11D 17/00**

22 Date of filing : **08.02.91**

30 Priority : **09.02.90 US 477682**

43 Date of publication of application :
14.08.91 Bulletin 91/33

84 Designated Contracting States :
CH DE ES FR GB IT LI NL SE

71 Applicant : **UNILEVER PLC**
Unilever House Blackfriars P.O. Box 68
London EC4P 4BQ (GB)
GB
Applicant : **UNILEVER NV**
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam (NL)
CH DE ES FR IT LI NL SE

72 Inventor : **Rys, Karla Jean**
319 Division Avenue
Hasbrouck Heights, New Jersey 07604 (US)
Inventor : **Greene, Alan Paul**
16 Grandview Circle
Flemington, New Jersey 08822 (US)
Inventor : **Osmer, Frederick Sylvio**
5 Citroen Way
Parsippany, New Jersey 07054 (US)
Inventor : **Podgorsky, Joseph James**
9 Woodward Road
Slate Hill, New York 10973 (US)

74 Representative : **Ford, Michael Frederick et al**
MEWBURN ELLIS 2 Cursitor Street
London EC4P 4BQ (GB)

54 **Detergent composition.**

57 A skin cleansing composition is provided that includes a combination of acyl isethionate ester salts with at least one sulfosuccinate. The weight ratio of acyl isethionate to sulfosuccinate will be about 10 :1 to about 2 :1.

DETERGENT COMPOSITION

The invention relates to solid skin cleansing, detergent compositions containing acyl isethionates.

Traditionally, soap has been utilized as a skin cleanser. Soap is, however, a very harsh chemical. Irritated and cracked skin result from use of soap, especially in colder climates. There are, however, certain benefits from the use of soap including low cost, ease of manufacture into bars, and good lathering properties.

There has been much commercial activity in attempting to replace soaps with milder surfactants. The introduction of mild synthetic detergent toilet bars, especially those based on sodium cocoyl isethionates, has been particularly successful. Patents relating to this technology are discussed in a published PCT application number WO 90/12860, which is incorporated by reference herein.

U.S. 4,335,025 discusses synthetic detergent bars containing critical amounts of components. For example, alkali metal salts of C₈₋₁₆ alkyl sulfosuccinate together with a selected water soluble anionic detergent which may include a C₁₀₋₁₆ acyl isethionate, a waxy extender such as a C₁₂₋₂₂ fatty acid or alcohol and the like, and other optional extenders.

U.S. 3,901,832 discusses a detergent bar containing a monoalkyl sulfosuccinate and a specific plasticizer in detergent cakes.

U.S. 3,989,642 discusses a synthetic toilet bar containing selected amounts of an alkane sulfonate, a fatty acid, and a binder modifier, which may be an alkyl sulfosuccinate.

U.S. 4,812,253 discusses various compositions containing polymeric skin mildness aids, moisturizers, soap and selected surfactants which include acyl isethionates and alkyl sulfosuccinate.

The present invention aims to provide compositions with excellent skin mildness while also possessing good lather, mush and processing properties.

According to the present invention there is provided a skin cleansing toilet bar composition comprising :

i) acyl esters of isethionic acid salts, the acyl chain length distribution of said esters being at least about 90% C₆-C₁₈ and having more than about 30% C₁₄ or lower ; and

ii) at least one sulfosuccinate wherein the weight ratio of said acyl esters to sulfosuccinate is 10 :1 to 2 :1.

Preferred skin cleansing compositions have acyl isethionates as the main active in combination with monoalkyl sulfosuccinates as the co-active. Such compositions can be substantially milder to the skin than previously known compositions.

Compositions within this invention may contain some free fatty acid. They may contain some free isethionate. They may contain some soap.

A preferred solid skin cleansing composition comprises :

(i) acyl esters of isethionic acid salts in an amount of 20 to 70% ;

(ii) at least one sulfosuccinate wherein the weight ratio of said acyl esters to sulfosuccinate is 10 :1 to 2 :1;

(iii) free fatty acid in an amount of 2-40% ;

(iv) free isethionate in an amount of 2-20% ;

(v) soap in an amount of 0-25% ;

(vi) water in an amount of 2-20% ;

(vii) miscellaneous other constituents in an amount of 0-20%.

Preferred weight ratios of acyl isethionate to sulfosuccinates are 6 :1 to 2 :1 and most preferred to maintain mildness are 2 :1 to 4.5 :1.

In this invention, the irritancy of the already mild main active, acyl isethionate, can be reduced significantly by the incorporation of sulfosuccinates.

The cleansers resulting from this mixture of actives have superior skin mildness together with good bar properties such as processability, low mush, good lather, and good tactile characteristics. In addition, they are easily processable using standard manufacturing equipment. In order for processability to be good, it is usually required for the composition to have an appropriate hardness and stickiness.

Isethionate salts in the range of about C₆-C₁₈, such as those derived from coconut fatty acids, for example, have been employed in a number of commercial cleansing products and are known to produce a voluminous, creamy lather. This active, by itself, has been shown to be very mild to the skin. These actives are solid at room temperature and serve as excellent structurants when used in bar formulations.

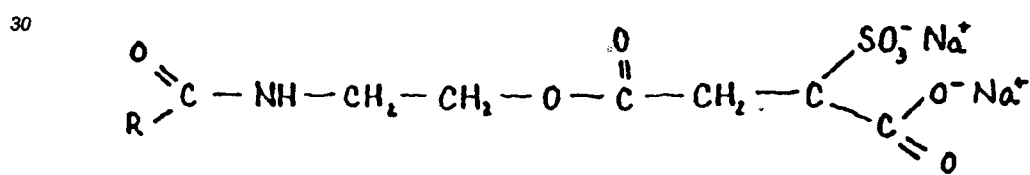
The isethionate ester salts that may be employed herein are preferably acyl ester isethionates and most preferably the cocoyl ester. These preferred esters may be prepared from the usual cocoyl fatty acids having a small percentage of fatty acid chains below C₈ with over 95% of the carbon chain distribution being between C₈ and C₁₈ and more than half being C₁₂ or less. Preferably, the acyl chain length of the esters will have at least about 90% C₆-C₁₈ and more than about 30% C₁₄ or lower. A typical cocoyl fraction will contain :

5	<u>Chain Length</u>	<u>Wt. % Fatty Acid Combined As Cocoyl Isethionate</u>
10	C ₆₋₁₀	10-25
	C ₁₂	45-55
15	C ₁₄₋₁₈	20-40
	C ₁₈ unsaturated	1-15

20 Skin testing has shown that the mixed active system of this invention significantly reduced the irritancy of the already mild acyl isethionate base. Skin testing and use property evaluation have shown that the preferred ratio of isethionate to sulfosuccinate is 2 :1 to 6 :1.

25 The sulfosuccinates useful in the invention may be any suitable sulfosuccinate ester mono-esters (half esters) or di-esters and any alkali metal, alkaline earth metal or ammonium salt, but the sulfosuccinates are preferably monoesters and preferably prepared through an alkanolamine intermediate so as to introduce an amido group into the molecule. Preferably to give processability, mildness and other use properties the sulfosuccinate monoester is prepared through a monoethanolamine intermediate.

The most preferable sulfosuccinates are, Disodium Cocamido Monoethanolamide Sulfosuccinate of the Structure 1 :



40 R in Structure 1 is derived from coconut fatty acids, but in other forms may be any convenient alkyl group. The chain length distribution of coconut fatty acids is similar to that for the coco group on the isethionate. The chain length distribution will contain at least 90% of C₈ to C₁₈ with more than half made up of C₈ to C₁₄. A typical fatty acid distribution in the cocoyl portion is as follows :

45	<u>Chain Length</u>	<u>Weight % Fatty Acid Combined As Cocoyl Amido Sulfosuccinate</u>
	C ₆₋₁₀	10-25
50	C ₁₂	45-55
	C ₁₄₋₁₈	20-40
	C ₁₈ unsaturated	1-15

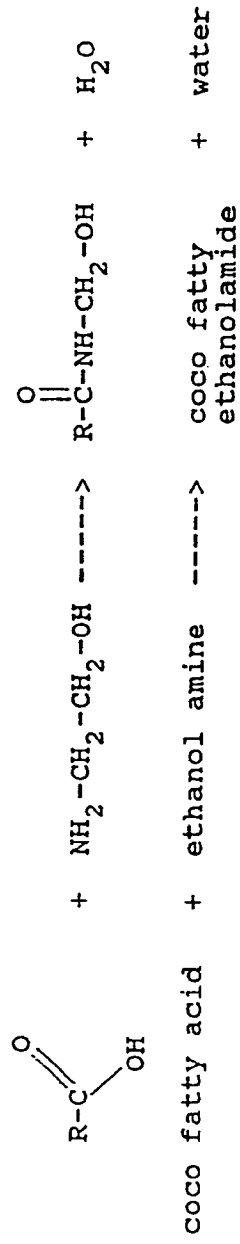
55 The sulfosuccinate of Structure 1 may conveniently be prepared by a two step reaction :

1. An ester condensation reaction of Coco alkanolamide with Maleic Anhydride.
2. Sulfonation of the double bond with Sodium Sulfite as described below.

5
10
15
20
25
30
35
40
45
50
55

Preparation of Disodium Cocamido Monoethanolamide Sulfosuccinate

a) preparation of the coco-fatty ethanolamide



5

10

15

20

25

30

35

40

45

50

55

b) ester condensation reaction of maleic anhydride
and the coco ethanolamide



coco fatty ethanolamide + maleic anhydride
(molar excess)

1. 70-100C -----> monoester + water
2. catalyst
3. 2 hours

c) sulfonation with sodium sulfite

(See Step b) + Na₂SO₃ (Structure 1)

monoester + sodium sulfite -----> disodium cocamido me a sulfosuccinate
aqueous solution

The relative amounts of isethionate esters to sulfosuccinate will preferably lie in the weight ratio of 2 :1 to 4.5 :1, or even as high as 6 :1, preferably from 3 :1 to 4 :1, optimally about 3.5 :1.

As previously noted, soap may be somewhat harsh and when present in the compositions of this invention should be at a level no higher than about 25%, preferably less than 5%, and advantageously totally absent.

Free fatty acids of about 8-22 carbon atoms are desirably incorporated within the compositions of the present invention. Some of these fatty acids are present to operate as superfatting agents and others as skin feel and creaminess enhancers. Fatty alcohols, fatty amides and the like may also be employed. Superfatting agents enhance lathering properties and may be selected from fatty acids of carbon atoms numbering 8-18, preferably 10-18, in an amount up to 40% by weight of the composition. Skin feel and creaminess enhancers, the most important of which is stearic acid, are also desirably present in these compositions.

Other performance chemicals and adjuncts may be needed or employed with these compositions. The amount of these chemicals and adjuncts may range from about 0% to about 20% by weight of the total compositions. For instance, there may be included humectants such as glycerine ; anti wear agents such as polymer JR and natural and synthetic gums and the like ; germicides, perfumes, colorants, dyes, pigments such as titanium dioxide, electrolytes, and water.

Evaluation of an active's skin mildness properties are determined through the following test procedure.

The Flex Wash Test

The products skin mildness properties were evaluated by comparison to Dove toilet bars (Dove is a registered trademark of Lever Brothers Co.), which are very mild commercial bars, through the following test procedure. The Flex Wash test procedure consists of three daily two minute washes of the antecubital fossa (flex area of elbow). This method is an "exaggerated use" method designed to differentiate very mild products. Erythema response varies only slightly with temperature and humidity fluctuations making the protocol suitable for year round testing.

Approximately 15 panelists were used as the test population. Panelist flex areas must be free of any skin condition (eczema, dryness, irritation, cuts or abrasions). Anyone taking antihistamines, anti-inflammatory drugs (more than 8 per week) or topical, oral or injectable cortisone on a regular basis was excluded from the study. The panel was divided into two sub-groups which were balanced for left handedness. Group I was assigned composition "A" for the left flex and "B" for the right flex. Group II reversed the order.

Following an evaluation, the panelist was instructed to moisten the left flex area, the sponge and test compositions formulated as toilet bars were dampened with tap water (100 ppm calcium/magnesium ions). The sponge was then stroked over the test bar 10 times by the evaluator. The "dosed" sponge was placed in the panelist's right hand. The panelist then washed the left flex area for exactly two minutes. Thereupon, the flex was rinsed and patted dry. This washing procedure was repeated on the right arm with the appropriate composition. Thus, both arms are tested simultaneously. Washing by this procedure was repeated three times daily for 5 consecutive days for a total of 15 washes. Treatment times were scheduled 1.5 hours apart. Each test site was evaluated immediately prior to washing and 4 hours after the third daily wash.

One trained assessor evaluated test sites prior to each wash and 4 hours after the third wash of the fifth day for a total of 15 evaluations. The grading scale was as follows :

0 - no erythema

0.5 - barely perceptible erythema

1 - mild spotty erythema/no edema

1.5 - mild/moderate erythema/with or without edema

2 - moderate confluent erythema/with or without edema or vesiculation

Each test site was treated in the prescribed method until a grading of "2" or greater was attained or 15 washings had been completed. When a score of "2" or greater was attained, the treatment was discontinued on that flex. The final score was then carried through for all remaining evaluations. The remaining flex was washed until either a grading of at least "2" or 15 treatments were attained, whichever was first. In the Examples of this specification, the final grading, Mean Rank Scores, is the sum total of grade scores for 15 assessments per panelist averaged over the scores from all panelists. Thus, theoretically, the average score could range from 0 to 30 ; the lower score indicating absolutely no skin irritation while the 30 score being the most severe. Mean Endpoint Erythema scores are the mean of the evaluation scores, for each panelist, at which the first arm received a grade of "2" or greater erythema score or at the completion of fifteen washes.

The following examples will more fully illustrate the embodiments of this invention. All parts, percentages and proportions referred to herein and in the appended claims are by weight of the total composition unless otherwise stated.

EXAMPLE 1

5 This example illustrates the difference in mildness between Dove and a toilet bar employing the Acyl Isethionate/Alkyl Sulfosuccinate active system, where the ratio of Isethionate to Sulfosuccinate is 2.20 :1.

Formulation 1

10	Sodium Cocoyl Isethionate	33.10
	Stearic Acid	29.80
	Sodium Isethionate	9.10
	Disodium Cocamido	
	MEA Sulfosuccinate	15.00
15	Water	10.00
	Perfume	1.00
	Sodium Chloride	0.35
	Titanium Dioxide	0.20
	Miscellaneous	1.45

20

Flex Wash

25

	<u>Mean Endpoint Erythema</u>	<u>Mean Rank Score</u>	
30	Formulation 1	0.794	10.79
	Dove	1.735	24.21

35 Statistical Analysis

Rank Scores: P = 0.0000

(Wilcoxon 2 sample)

40

EXAMPLE 2

45 The addition of small amounts of PEG-150, a polymer of ethylene oxide, does not deteriorously affect the mildness of a 2.87 :1, Isethionate to Sulfosuccinate, formulated toilet bar.

Formulation 2

50	Sodium Cocoyl Isethionate	43.6
	Stearic Acid	22.0
	Sodium Isethionate	9.3
	Disodium Cocamido	
	MEA Sulfosuccinate	15.2
	Sodium Chloride	2.6
55	PEG 150*	1.0
	Water	4.7
	Perfume	1.0
	Titanium Dioxide	0.5
	EHDP	0.02
	EDTA	0.02

* Carbowax 8000 (Union Carbide)

5

Flex Wash

10

	Mean Endpoint	Mean Rank
	<u>Erythema</u>	<u>Score</u>

15

Formulation 2	0.69	14.08
Dove	1.05	22.92

20

Statistical Analysis

Rank Scores P = 0.0096

25

(Wilcoxon 2 sample)

EXAMPLE 3

30

In the following example, mildness superior to that of Dove is again demonstrated. The Isethionate to Sulfosuccinate ratio here is 2.89 :1.

Formulation 3

35

Sodium Cocoyl Isethionate	43.9
Stearic Acid	20.1
Sodium Isethionate	11.0
Disodium Cocamido	
40 MEA Sulfosuccinate	15.2
Sodium Chloride	5.1
Water	3.2
Perfume	1.0
Titanium Dioxide	0.5
45 EHDP	0.02
EDTA	0.02

50

55

Flex Wash

5

	<u>Mean Endpoint Erythema</u>	<u>Mean Rank Score</u>
Formulation 3	0.8	12.29
10 Dove	1.5	22.71

Statistical Analysis

15

Rank Scores P = 0.0018

(Wilcoxon 2 sample)

20

EXAMPLE 4

25 In this example, the ratio of Cocoyl Isethionate to Amido Sulfosuccinate is 3.64 :1. Mildness, superior to Dove, is proven by-the Flex Wash.

Formulation 4

30 Sodium cocoyl Isethionate	43.7
Stearic Acid	23.9
Disodium Cocamido MEA Sulfosuccinate	12.0
Sodium Isethionate	13.8
Water	4.7
35 Perfume	1.0
Sodium Chloride	0.35
Titanium Dioxide	0.5
EHDP	0.02
EDTA	0.02

40

Flex Wash

45

	<u>Mean Endpoint Erythema</u>	<u>Mean Rank Score</u>
Formulation 4	0.9	12.56
50 Dove	1.6	22.44

Statistical Analysis

55

Rank Scores P = 0.0027

(Wilcoxon 2 sample)

EXAMPLE 5

5 A significant improvement in mildness over Dove, at the 90% confidence level, is achieved when the isethionate to sulfosuccinate ratio is 5.12 :1. The Mean Endpoint Erythema is substantially lower in the mixed active, Isethionate/ Sulfosuccinate bar, than in Dove.

Formulation 5

10

Sodium Cocoyl Isethionate	46.1
Stearic Acid	23.7
Disodium Cocamido MEA	
Sulfosuccinate	9.0
15 Sodium Isethionate	13.7
PEG 150	1.0
Sodium Chloride	0.35
Water	4.7
Perfume	1.0
20 Titanium Dioxide	0.5
EHDP	0.02
EDTA	0.02

*Carbowax 8000 (Union Carbide)

25

Flex Wash

	<u>Mean Endpoint Erythema</u>	<u>Mean Rank Score</u>
30 Formulation 5	0.8	15.69
Dove	1.27	21.31

35

Statistical Analysis

Rank Scores: P = 0.1028

40

EXAMPLE 6

Lather Volume measurements and bar mush evaluations were performed on the forementioned formulations. A brief description of the lather and mush tests are reported below.

45 Objective Lather Volume - This test involved rotating the toilet bar 15 half turns under running 95° F water. The bar was then set aside and the resulting lather was worked by hand for 10 seconds. A measuring funnel was then placed over the hands and both were lowered into a sink filled with water to the 0 ml mark on the measuring funnel. When the hands were fully immersed, they were removed from beneath the funnel. The funnel was then lowered to the bottom of the sink and lather volume was measured.

50 Mush - Pre-weighed bars were immersed in water for 2 hours. The increase in weight was noted. Mush was removed and the remainder of the bar was dried. After reweighing, mush was calculated as the weight of mush per 50 cm² of surface mushed. Lower values indicated better performance.

55

TABLE 1

5

Formulation #	Acyl Isethionate:	Lather Volume	Mush	
	<u>Mono-Alkyl Sulfosuccinate</u>	<u>(ml)</u>	<u>(g/50cm²)</u>	
10	1	2.20:1	125	-
	2	2.87:1	135	9.45
15	3	2.89:1	150	8.22
	4	3.64:1	160	7.15
	5	5.12:1	160	6.54
20	Commercial Dove	-	150	8.25

All of the formulations in Table 1 have excellent lather and low bar mush values.

25 EXAMPLE 7

In the following example, six mixed active bars and commercial Dove, were panel tested. The panel consisted of 15 highly discriminating female Dove users. Each panelist was asked to set a water temperature and then wet her hands. A bar was then taken from the soap dish, into both hands, lathered up, and placed back into the dish. The panelist then rinsed her hands and patted them dry with a non-woven towel. At the end of the wash the panelist was asked to complete a questionnaire. This process was repeated for each of the 6 remaining bars. The questionnaire was divided into two parts, the first part inquired about use properties and asked the panelists to judge the Ease of Lather Generation, Subjective Lather Volume, Lather Creaminess and Ease of Rinsing. The second part asked about skin after effects such as dryness, softness, irritation, smoothness, and cleanliness.

35

Results of these tests show the Isethionate/Sulfosuccinate combination has excellent consumer properties.

40

45

50

55

5 The test bars had the following compositions:

10 Hand Wash Formulations

<u>BAR</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
Isethionate: Sulfosuccinate	2.87:1	2.88:1	5.19:1	3.64:1	5.12:1	3.59:1	
15 Sodium Cocoyl Isethionate	43.6	40.9	46.7	43.7	46.1	43.1	Dove
Stearic Acid	22.0	23.7	23.9	23.9	23.7	23.7	
20 Sodium Isethionate	9.3	13.7	13.8	13.8	13.7	13.7	
Disodium Cocamido MEA Sulfosuccinate	15.2	14.2	9.0	12.0	9.0	12.0	
25 Sodium Chloride	2.6	0.35	0.35	0.35	0.35	0.35	
Carbowax 8000	1.0	1.0	0.0	0.0	1.0	1.0	
Perfume	1.0	1.0	1.0	1.0	1.0	1.0	
30 Titanium Dioxide	0.5	0.5	0.5	0.5	0.5	0.5	
EDTA	0.02	0.02	0.02	0.02	0.02	0.02	
35 EHDP	0.02	0.02	0.02	0.02	0.02	0.02	
Water	4.7	4.7	4.7	4.7	4.7	4.7	

40

45

50

55

Lather was evaluated based upon the following scale:

5

<u>GRADE</u>	<u>DEFINITION</u>	<u>GRADE</u>	<u>DEFINITION</u>
10	Outstanding	5	Fairly Good
9	Excellent	4	Moderate
8	Extremely Good	3	Fairly Poor
7	Very Good	2	Poor
6	Good	1	Terrible

10

15

20

Mean Ratings

25

<u>Bar #</u>	Subjective			
	<u>Ease of Lather Generation</u>	<u>Lather Volume</u>	<u>Lather Creaminess</u>	<u>Ease of Rinsing</u>
1	6.80	7.07	7.67	8.13
2	8.27	8.27	8.47	7.60
3	7.67	7.27	7.67	8.00
4	8.00	7.93	8.07	8.27
5	7.60	7.20	7.93	7.93
6	7.20	7.13	7.53	7.33
7	8.14	7.86	8.00	7.79

30

35

40

These results clearly show that bars 1 to 6 are quite as consumer acceptable as the commercial bar. Skin Afterfeel was evaluated based upon the following scale :

45

<u>GRADE</u>	<u>DEFINITION</u>	<u>GRADE</u>	<u>DEFINITION</u>
10	Fully Agree	5	
9		4	
8		3	
7		2	
6		1	Fully Disagree

50

55

Mean Ratings

Bar #	<u>Is Not Drying to the Skin</u>	<u>Leaves Skin Feeling Soft</u>	<u>Is Not Harsh or Irritating</u>	<u>Leaves Skin Feeling Smooth</u>	<u>Leaves Skin Feeling Clean</u>
1	7.47	8.07	8.00	8.20	7.67
2	7.40	7.67	8.07	8.33	7.93
3	7.73	7.80	8.07	8.13	8.27
4	8.00	7.93	8.20	8.33	8.00
5	7.73	7.80	8.07	8.07	7.87
6	7.07	7.47	8.00	8.07	7.87
7	7.07	7.57	7.57	7.57	7.86

These results show that the combination of the invention is not only equal to the commercial bar, but in some cases, perceivably better.

Claims

1. A skin cleansing toilet bar composition comprising :
 - i) acyl esters of isethionic acid salts, the acyl chain length distribution of said esters being at least about 90% C₆-C₁₈ and having more than about 30% C₁₄ or lower ; and
 - ii) at least one sulfosuccinate wherein the weight ratio of said acyl esters to sulfosuccinate is 10 :1 to 2 :1.
2. A composition according to claim 1 wherein the sulfosuccinate is a mono ester.
3. A composition according to claim 1 wherein the sulfosuccinate is derived from a monoethanolamide.
4. A composition according to claim 2 wherein the sulfosuccinate is an amido sulfosuccinate.
5. A composition according to claim 4 wherein the sulfosuccinate is a cocoamido sulfosuccinate.
6. A composition according to any one of the preceding claims wherein the ratio of isethionate ester to sulfosuccinate is from 6 :1 to 2 :1.
7. A composition according to any one of the preceding claims wherein the ratio of isethionate ester to sulfosuccinate is from 4.5 :1 to 3 :1.
8. A composition according to any one of the preceding claims wherein said acyl chain length has the following distribution :

	<u>Chain Length</u>	<u>Weight %</u>
5	C ₆₋₁₀	10-25
	C ₁₂	45-55
10	C ₁₄₋₁₈	20-40
	C ₁₈ unsaturated	1-15.

9. A composition according to any one of the preceding claims wherein the amount of said acyl esters of isethionic acid salts is 20 to 70% by weight of the composition.

10. A composition according to claim 9 which also comprises 2 to 40% of free fatty acid, 2 to 20% of sodium isethionate and 2 to 20% by weight of water, the amount of soap in the composition not exceeding 25% by weight and the amount of any other materials in the composition not exceeding 20% by weight.

European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 91301046.8
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	<u>US - A - 4 211 675</u> (D. MACHIN) * Claims 1,3,4; column 2, lines 14-38,58 *	1,2, 6-10	C 11 D 1/12 C 11 D 10/04 C 11 D 17/00
Y	--	3-5	
Y	<u>US - A - 3 793 215</u> (B.R. SMITH) * Claims 1,4 *	3,4	
Y	<u>DE - A1 - 3 335 705</u> (CHEMISCHE WERKE HÜLS AG) * Claim 1; page 6, line 20 *	3-5	
D,A	<u>US - A - 4 335 025</u> (G. BARKER et al.) * Claims 1,7; column 4, lines 16-20; example X *	1,2	
P,X	<u>US - A - 4 954 282</u> (K.J. RYS et al.) * Claims 1-3; tables I,IV * & WO-A1-90/12 860 -----	1-10	
D			TECHNICAL FIELDS SEARCHED (Int. Cl.5) C 11 D
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 23-05-1991	Examiner REISER
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

EPF FORM (503 03 81 (9/0401))