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⑤④ **Liquid detergent compositions.**

⑤⑦ Aqueous clear enzyme-free dishwashing liquid compositions are provided incorporating a mixture of alkane sulphonate, alkyl ether sulphate and optional alkyl benzene sulphonate surfactants together with one or more suds boosters and a hydrotrope system, in which from 0.35% to 0.7% by weight of magnesium ion is included in order to provide enhanced sudsing performance. In order to provide a single phase system the level chloride ions should not exceed 1.75% by weight and the level of sulphate ions should not exceed 0.35% by weight provided that where both are present the level of sulphate ions should be no more than 0.25% by weight.

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LIQUID DETERGENT COMPOSITIONS

Field of the Invention

This invention relates to aqueous liquid dishwashing detergent compositions and especially to substantially unbuilt hand dishwashing detergent compositions incorporating
5 alkane sulphonate surfactant together with a source of magnesium ions.

Background of the Invention

The use of magnesium salts and magnesium surfactants such as alkyl sulphates, alkyl ether sulphates and alkyl
10 benzene sulphonates in dishwashing detergent formulations is known, and British Patent Specification Nos. 1,524,441, 1,551,074 and 2,010,893A are representative disclosures of the state of the art. The art teaches that these formulations have enhanced performance, particularly when
15 used in water of low mineral hardness. Liquid detergent compositions containing alkane sulphonate surfactants are also known in the art. Compositions primarily intended for fabric washing are described in B.P. 1,054,217, 1,329,508 and 1,291,163, the last named of which discloses
20 a mixture of an alkane sulphonate, an ethoxylated alkyl sulphate, and a proteolytic enzyme together with an enzyme-stabilising agent in the form of a calcium or magnesium salt, specifically the acetate or the chloride.

Enzymes are not however customary components of hand
25 dishwashing liquid detergent formulations, particularly in formulations where skin mildness is an important in-use requirement.

Dishwashing liquid detergent compositions containing alkane sulphonates are also known, examples of such
30 disclosures including those in B.P. 1,339,069, 1,382,295, 1,451,228 and 1,567,421. However products made in

accordance with these teachings have all been found to be non-optimal in one area of performance or another eg. raw material cost, phase stability on storage, sudsing in water of high or low mineral hardness, effect on hands, suds profile, physical stability under cyclic temperature conditions etc.

The present invention is concerned with an aqueous hand dishwashing detergent composition that is enzyme-free. More particularly, it concerns an enzyme-free dishwashing detergent composition containing alkane sulphonate surfactant, the sudsing performance of which has been enhanced by the incorporation of specific levels of magnesium ion.

According to the present invention there is provided a clear single-phase enzyme-free dishwashing liquid composition comprising 20-40% of a C_{14} - C_{17} alkane sulphonate, 5-15% of an anionic surfactant selected from water soluble C_{10} - C_{16} alkyl ethoxy sulphates containing from 1-6 ethoxy groups per mole of alkyl sulphate, and mixtures thereof with water soluble C_{10} - C_{16} alkyl benzene sulphonates, 0-5% of a suds booster selected from C_{10} - C_{16} alkyl amides, C_{10} - C_{16} alkyl C_2 - C_3 alkanolamides, C_{10} - C_{16} alkyl di C_1 - C_3 alkyl or di C_2 - C_3 hydroxyalkyl amine oxides and from 0.35%-0.7% magnesium ion, the composition containing no more than 1.75% by weight of chloride ions or more than 0.35% by weight of inorganic sulphate ions provided that where chloride ions are also present the level of sulphate ions should not exceed 0.25% by weight, the counter ions other than magnesium necessary to provide a pH of 6.0-7.5 being selected from sodium, potassium, ammonium and alkanolammonium.

Preferably the composition comprises 22-36% C_{16-17} alkanesulphonate, 6-12% C_{12-14} alkyl ethoxy sulphate and 0.5% magnesium ion with a maximum of 0.5% inorganic salt anions.

Secondary alkane sulphonates useful in the present invention preferably have from 13 to 18 carbon atoms per

molecule, and most desirably 13 to 15, and are characterised by a high solubility in water compared to alkyl aryl sulphonates and other sulphuric acid reaction products used for dishwashing detergent compositions. These

5 sulphonates are preferably prepared by subjecting a cut of paraffin, corresponding to the chain lengths specified above, to the action of sulphur dioxide and oxygen in accordance with the well-known sulfoxidation process. The product of this reaction is a secondary sulphonic

10 acid which is then neutralized with a suitable base to provide a water-soluble secondary alkyl sulphonate. Similar secondary alkyl sulphonates may be obtained by other methods, e.g. by the sulphochlorination method in which chlorine and sulphur dioxide are reacted with

15 paraffins in the presence of actinic light, the resulting sulphonyl chlorides being hydrolyzed and neutralized to form the secondary alkyl sulphonates. Whatever technique is employed, it is normally desirable to produce the sulphonate as the monosulphonate, having no unreacted

20 starting hydrocarbon or having only a limited proportion thereof present and with little or no inorganic salt by-product. Similarly, the proportions of disulphonate or higher sulphonated material will be minimized but some may be present. The monosulphonate may be terminally

25 sulphonated or the sulphonate group may be joined on the 2-carbon or other carbon of the linear chain. Similarly, any accompanying disulphonate, usually produced when an excess of sulphonating agent is present, may have the sulphonate groups distributed over different carbon atoms

30 of the paraffin base, and mixtures of the monosulphonates and disulphonates may be present.

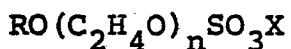
Mixtures of monoalkane sulphonates wherein the alkanes are of 14 and 15 carbon atoms are particularly preferred wherein the sulphonates are present in the weight ratio

35 of C₁₄ to C₁₅ paraffins in the range from 1:3 to 3:1,

preferably 1:2 to 2:1. Surprisingly, this particular mixture produces detergents which clean dishes better and which suds longer, especially in hard water, than other mixtures of paraffin sulphonates, e.g. those of
5 13 to 17 carbon atoms. This is also true, to a lesser extent, of the individual components of the C₁₄ and C₁₅ mixture.

The alkane sulphonates are used at a level of from 20% to 40%, more generally at a level of from 22% to 36%,
10 preferably from 24% to 34% and most preferably from 26% to 32% by weight of the composition.

The alkyl ethoxy sulphates used in the compositions of the present invention can be represented by the formula



15 where R is a C₁₀-C₁₆ preferably 12-15 linear or branched chain, primary alkyl group, n is a value from 1 to 6 preferably 2-5 and X is a water soluble cation. C₁₀-C₁₆ alcohols, derived from natural fats or Ziegler olefin build-up or OXO synthesis form suitable sources for the
20 alkyl group. Examples of synthetically derived materials include Dobanol 23 (RTM) sold by Shell Chemicals (UK) Ltd., Ethyl 24 sold by the Ethyl Corporation, a blend of C₁₃-C₁₅ alcohols in the ratio 67% C₁₃, 33% C₁₅ sold under the trade name Lutensol by BASF GmbH and Synperonic
25 (RTM) by ICI Ltd, and Lial 125 sold by Liquichimica Italiana. Examples of naturally occurring materials from which the alcohols can be derived are coconut oil and palm kernel oil and the corresponding fatty acids.

The alkyl ethoxy sulphates are used in an amount of
30 from 5% to 15% by weight of the composition, and, where they constitute the only anionic surfactant species other than the alkane sulphonate, are normally used at from 10% to 15% by weight.

The alkyl ethoxy sulphates can also be used in
35 admixture with water soluble linear or branched C₁₀-C₁₆

alkyl benzene sulphonates. In such alkyl benzene sulphonates, the alkyl group is preferably linear and contains 11-13 carbon atoms, a material with an average carbon chain length of 11.8 being most preferred. The phenyl isomer distribution, ie. the point of attachment of the alkyl chain to the benzene nucleus, is not critical but alkyl benzenes having a high 2-phenyl isomer content are preferred.

Where mixtures of the alkyl ethoxy sulphates and alkyl benzene sulphonates are used the levels of alkyl benzene sulphonate should be such that the ratio of the combined weight of alkane sulphonate and alkyl benzene sulphonate to the weight of alkyl ethoxy sulphate should not exceed 10:1 and preferably should not exceed 5:1. In practice the level of alkyl benzene sulphonate will not usually exceed 12% by weight of the composition and will normally be in the range 4%-10% by weight. Correspondingly, the overall alkyl ethoxy sulphate level will be reduced where such mixtures are employed.

An essential component of the formulation is a source of magnesium ions at a level to provide 0.35% to 0.70% by weight of magnesium ion in the product, preferably 0.40% to 0.60% by weight. Subject to the requirement that the final product is a clear single-phase liquid, the magnesium ion can be provided either by addition of a water soluble magnesium salt such as the chloride or acetate to the formulation, or by the use of magnesium oxide or hydroxide slurry as a component of the neutralising medium for one or other of the anionic surfactants during manufacture.

The requirement that the final product is a clear, single phase liquid creates a constraint on the level of dissolved inorganic salts that can be tolerated by the formulation without phase separation or crystallisation occurring. By 'tolerated' is meant the ability of a

formulation to withstand storage without the formation of other solid or liquid phases. A minimum requirement is that storage at ambient temperature (viz 20°C) should not result in such phase separation but preferably the

5 temperature at which phase separation or crystal deposition occurs should necessitate cooling of the composition to a sub-ambient temperature. This sub-ambient temperature is conventionally known as the chill point temperature of a liquid detergent composition and is defined as that

10 temperature below which solid inorganic crystals separate from the composition on storage. It has been found that, in order to achieve acceptable chill point temperatures, the composition of the present invention should not contain more than 1.75% by weight of chloride ions or

15 more than 0.35% of sulphate ions, provided that where chloride ions are also present the level of sulphate ions should not exceed 0.25% by weight and preferably should not exceed 0.15% by weight. Formulations having low chill point temperatures, i.e. < 5°C, have sulphate levels at

20 the lower end of this range.

The tolerance of the formulations to inorganic salt level has been found to be dependent on the inorganic salt type and also on the counter ion types present in the formulations. Thus formulations in which the counter

25 ions (other than the magnesium) are mainly or exclusively ammonium or potassium are more tolerant to inorganic salts than those where the counter ions are mainly or exclusively sodium ions. Furthermore chloride ions can be tolerated at much higher levels than sulphate ions.

30 It has been found, for example, that in a formulation comprising

30% MC₁₅-C₁₇ paraffin sulphonate
 15% MC₁₂(EO)₃ sulphate
 10% ethyl alcohol solvent
 35 with 0.475% Magnesium ion added as magnesium

hydroxide forming part of the counter ion M, when M is ammonium, the level of sulphate ion that the formulation can tolerate without precipitation is 0.35% by weight of the formulation whereas when M is sodium, 5 the level of sulphate ion that can be tolerated is no more than 0.20% by weight. By contrast in the same system, 1.75% chloride ion can be tolerated, irrespective of the counter ion employed, although at this level of chloride ion the presence of sulphate ions in excess of 10 about 0.15% by weight will lead to deposition of a crystalline precipitate.

The commercial production of paraffin sulphonates, alkyl benzene sulphonates and alkyl ether sulphates uses techniques that are well known in the art and involves 15 the use of sulph(on)ating agents such as SO_3 , oleum, chlorosulphonic acid or SO_2 , all of which give rise to residues of sulphates and/or chlorides in the active materials. The levels of sulphates and/or chlorides can be minimised by careful control of the sulph(on)ation 20 conditions, and by air, or inert gas, sparging of the acid sulph(on)ate products but in general at least 0.1% by weight of inorganic sulphate (on a finished product basis) arise from this source, even under ideal conditions. Thus, little or no sulphate ion can be added to these 25 systems if precipitation is to be avoided, and the addition of the magnesium ion as magnesium sulphate, is not feasible from a product stability standpoint.

Indeed, the low tolerance of the compositions of the present invention to inorganic sulphates, produced 30 as a by-product of the sulph(on)ation process frequently results in a 'desalting' step being necessary to provide acceptable product stability on storage. Where appreciable levels of inorganic sulphate are present, such as the 4-6% by weight of sulphate found in commercially 35 available paraffin sulphonates, this 'desalting' step is

essential in order to avoid precipitation and deposition of inorganic sulphate from compositions in accordance with the invention. This desalting step is normally carried out on the surfactant concerned prior to mixing
5 with other formulation components and can be performed by adding a lower aliphatic alcohol such as ethanol or isopropanol to the neutralised surfactant in order to precipitate the sulphate as a crystalline solid. The precipitated sulphate is then removed by filtration or
10 centrifugation.

As noted above, the formulations of the present invention are more tolerant of chloride ions than sulphate ions and thus where chloride ions are produced by a sulphonation by-product, such as in the chlorosulphation
15 of alkyl ethers, subsequent treatment of the surfactant is normally unnecessary. The level of chloride ion arising from this source is normally of the order of 0.5% on a total product weight basis and preferred formulations, made using SO_3 sulphonation techniques,
20 are essentially free of chloride ions.

The higher tolerance of the compositions of the invention to chloride ions leads to the situation that provided the system is substantially free of inorganic salts prior to the incorporation of the magnesium ion,
25 the latter can be incorporated as the chloride salts. In this context 'substantially free' means no more than 0.25% by weight of the composition of inorganic anions. Preferably the compositions contain less than 0.15% by weight of other inorganic anions when levels of chloride
30 ion in excess of about 1% by weight are present.

Nevertheless, the incorporation of a water soluble magnesium salt into the formulation, whilst being permissible, is not a preferred technique as it increases the risk of phase separation or crystalline salt deposition,
35 and, in the case of magnesium chloride, may give rise to

corrosion problems in the processing equipment. As noted above, the addition of magnesium sulphate in an amount sufficient to provide the benefits of the present invention causes crystalline salt precipitation. Even
5 where a salt such as magnesium acetate is employed as the source of magnesium ion, its addition to the formulation may cause sulphate and chloride salts to precipitate, even though these may only be present at levels which would otherwise be acceptably low. Accordingly the use
10 of magnesium oxide or hydroxide neutralisation of one or more of the anionic surfactant species is a preferred means of introducing the magnesium ion into the system.

The other cations used in the neutralisation of the anionic surfactants may be sodium, potassium, ammonium
15 or alkanolammonium, but ammonium is a preferred cation because of its depressive effect on the chill point temperature of the compositions. Preferred compositions have chill points $< 0^{\circ}\text{C}$ and in order to achieve this at least 50% of the surfactant cations should be ammonium
..20 ions.

A desirable component of the invention is a suds boosting agent at a level of up to 5%, preferably 3-4% by weight.

The suds-promoting agent can be any of $\text{C}_{12}\text{-C}_{14}$ mono-
25 and di $\text{C}_2\text{-C}_3$ alkanolamide, $\text{C}_{12}\text{-C}_{14}$ alkyl amides condensed with up to 15 moles of ethylene oxide per mole of amide and tertiary amine oxides containing a $\text{C}_8\text{-C}_{18}$ alkyl group.

Examples of the alkanolamides are coconut alkyl monoethanolamide, coconut alkyl diethanolamide and
30 coconut alkyl mono and di isopropanolamides.

Examples of the ethoxylated amides include coconut
alkyl amide condensed with six moles of ethylene oxide,
lauryl amide condensed with eight moles of ethylene
oxide, myristyl amide condensed with ten moles of
35 ethylene oxide and coconut amide condensed with eight

moles of ethylene oxide. Amine oxides useful in the present invention have one alkyl or hydroxyalkyl moiety of 8 to 18 carbon atoms, preferably 8 to 16 carbon atoms and two moieties selected from alkyl groups and hydroxy-
5 alkyl groups containing 1 to 3 carbon atoms. Examples of such amine oxides include dimethyloctylamine oxide, diethyldecylamine oxide, bis - (2 - hydroxyethyl) dodecylamine oxide, dimethyldodecylamine oxide, dipropyltetradecylamine oxide, methylethylhexadecylamine oxide, and
10 dimethyl - 2 - hydroxyoctadecylamine oxide.

A highly preferred example of the tertiary amine oxide is a C_{12} - C_{14} alkyl dimethyl amine oxide in which the C_{12} - C_{14} alkyl group is derived from coconut oil.

The balance of the formula comprises a hydrotrope-
15 water system in which the hydrotrope may be urea, a C_1 - C_3 alkanol, or a lower alkyl benzene sulphonate salt such as toluene, cumene or xylene sulphonate. The preferred hydrotrope is ethanol which is employed at from 6% to 10% by weight of the composition preferably at from 7%
20 to 9%.

Optional ingredients of the liquid detergent compositions of the invention include thickeners such as guar gum, antibacterial agents such as glutaraldehyde and Bronopol (RTM), antitarnish agents such as benzoxotriazole,
25 heavy metal chelating agents such as EDTA or EDTMP, perfumes and dyes. The pH of the compositions may be anywhere within the range 6-7.5 but as manufactured the compositions normally have a pH in the range 6.6-7.3 preferably 6.6-6.9 in order to maintain colour stability.

30 The technique of incorporation of the magnesium ions is not thought to be critical and the compositions can be made in a number of ways.

The individual anionic surfactants can be made as aqueous solutions of alkali metal or ammonium salts which are
35 then mixed together with the hydrotrope, and the suds

booster, if this is included, following which the magnesium ion can be introduced as a water soluble salt such as the chloride or acetate. Optional minor ingredients are then added after which the pH and viscosity is
5 adjusted. This method has the advantage of utilising conventional techniques and equipment but results in the introduction of additional chloride or acetate ions which can increase the chill point temperature (the temperature at which inorganic salts precipitate as
10 crystals in the liquid).

An alternative method is to neutralise the alkyl ether sulphuric acid or the alkane sulphonic acid with a magnesium oxide or hydroxide slurry which avoids the introduction of additional inorganic anions. Although
15 not essential, it is convenient to carry out the neutralisation of the alkyl ether sulphate in a 'heel' formed by a dispersion of the magnesium oxide or hydroxide slurry in a mixture of the hydrotrope and the other surfactant. Any alkyl benzene sulphonate present in
20 the formulation can be neutralised separately or in the same 'heel', and the neutralised sulphonate and sulphate surfactants together with the hydrotrope are then added to the final mixing tank and the suds booster and any optional ingredients added before the pH is adjusted
25 as above.

The invention is illustrated in the following examples in which the percentage of the components are by weight on the finished composition.

Example 1

The following composition was made up

	C ₁₄ -C ₁₅ s-alkane sulphonate	35)	incorporating 0.44
	C ₁₂ -C ₁₃ alkyl (EO) ₃ sulphate	15)	parts Mg ⁺⁺ ion, and
		9	2.21 parts NH ₄ ⁺ ion
5	Ethanol		
	Urea	3	
	Perfume Colour & Miscellaneous	1	
	Water	<u>37</u>	
		100	

- 10 The C₁₄-C₁₅ s-alkane sulphonate, available as a paste of the ammonium salt containing 60% active and 4% ammonium sulphate was desalted by treating 58.33 parts paste with 9 parts ethanol and filtering off the precipitated ammonium sulphate to leave 65.23 parts of desalted paste
- 15 containing 0.23 parts ammonium sulphate. This desalted paste was then used as a heel for the neutralisation of the alkyl ether sulphuric acid. The latter was prepared by sulphation of an ethoxylated linear C₁₂-C₁₃ alcohol using an SO₃-air mixture and contained 0.46% H₂SO₄. 2.12
- 20 parts of a 50% slurry of magnesium hydroxide in water was added to the paste heel and then the alkyl ether sulphuric acid was added with agitation. After the neutralisation of the alkyl ether sulphuric acid the remaining com-
- 25 ponents were added and the pH was trimmed to 6.8 using ammonium hydroxide or citric acid. The resulting liquid detergent was a stable clear liquid with a sulphate content, expressed as (NH₄)₂SO₄, of 0.32% by weight of the composition.

Example 2

30	C ₁₄ -15 s-alkane sulphonate	30)	incorporating 0.44
	C ₁₂ -C ₁₃ alkyl (EO) ₃ sulphate	10)	parts Mg ⁺⁺ ion, and
	Linear C ₁₁₋₈ alkyl benzene	5)	1.93 parts NH ₄ ⁺ ion
	sulphonate		
	Ethanol	9	
	Urea	4	
35	Perfume Colour & Miscellaneous	1	
	Water	<u>41</u>	
		100	

A similar procedure is followed as for Example 1. The ammonium alkane sulphonate surfactant is desalted to leave 57.2 parts of paste containing ammonium sulphate and 0.20 parts ammonium sulphate and the alkyl benzene sulphonic acid (produced by SO_3 sulphonation and containing 0.11 parts H_2SO_4) is added to this desalted paste. The magnesium hydroxide slurry is then mixed in followed by the alkyl ether sulphuric acid. When neutralisation of the latter is complete the pH is brought up to pH 6.8 with ammonium hydroxide and the remainder of the ingredients added to complete the formulation. The final product is a clear stable liquid with a sulphate content, expressed as $(\text{NH}_4)_2 \text{SO}_4$ of 0.43% by weight of the composition.

15 Example 3

$\text{C}_{14}\text{-C}_{15}$ s-alkane sulphonate	25)	incorporating 0.44
$\text{C}_{12}\text{-C}_{13}$ alkyl $(\text{EO})_3$ sulphate	10)	parts Mg^{++} ion, and
Linear $\text{C}_{11.8}$ alkyl benzene sulphonate	5)	1.62 parts NH_4^+ ion
Coconut monoethanolamide	3	
20 Ethanol	10	
Perfumes Colour & Misc.	1	
Water	<u>46</u>	
	100	

The product is made up using the procedure of Example 25 2 to give a clear liquid having a sulphate content expressed as $(\text{NH}_4)_2 \text{SO}_4$ of 0.40% by weight of the composition.

CLAIMS

1. An enzyme-free dishwashing liquid composition comprising a mixture of C_{14} - C_{17} s-alkane sulphonates, C_{10} - C_{16} alkyl ethoxy sulphates containing an average of 1-6 ethoxy groups per mole of alkyl sulphate, optionally together with an alkyl alkanolamide suds booster in a hydrotrope-water vehicle characterised in that the composition is a clear single phase liquid comprising
 - 20-40% alkane sulphonate
 - 5-15% alkyl ethoxy sulphate or a mixture of alkyl ethoxy sulphate and C_{10} - C_{16} alkyl benzene sulphonate
 - 0.35-0.7% magnesium ion
 the composition containing no more than 1.75% by weight of chloride ion or more than 0.35% by weight of inorganic sulphate ion provided that where chloride ions are also present the level of sulphate ions should not exceed 0.25% by weight, the counter ions other than magnesium necessary to provide a pH of 6.0-7.5 being selected from sodium, potassium, ammonium and alkanol ammonium.
2. A dishwashing liquid composition according to Claim 1 characterised in that the composition comprises
 - 24%-34% alkane sulphonate
 - 5%-11% alkyl ethoxy sulphate
 - 4%-10% C_{11} - C_{13} alkyl benzene sulphonate
 - 0.4%-0.6% magnesium ion.
3. A dishwashing liquid composition according to either one of Claims 1 and 2 characterised in that the composition is substantially free of chloride ions.
4. A dishwashing composition according to any one of Claims 1-3 wherein the counter ions other than magnesium are ammonium ions.

5. A dishwashing composition according to Claim 3 wherein the counter ions other than magnesium are sodium ions, and the level of sulphate ion is less than 0.20% by weight.



European Patent
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EUROPEAN SEARCH REPORT

0062371

Application number

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DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
P,X	<u>EP - A1 - 0 039 110</u> (PROCTER & GAMBLE LTD. et al.) * claims 1, 2, 6; page 7, lines 7 to 12, page 8, lines 13 to 17 * --	1-4	C 11 D 1/12 C 11 D 1/29 C 11 D 17/00
D,A	<u>GB - A - 2 010 893</u> (UNILEVER LTD.) --		
D,A	<u>GB - A - 1 054 217</u> (ESSO RESEARCH AND ENGINEERING CO.) --		TECHNICAL FIELDS SEARCHED (Int.Cl. ³)
A	<u>DE - A1 - 2 636 967</u> (PROCTER & GAMBLE CO) * claims 1, 10 * & GB - A - 1 551 074 --		C 11 D 1/00 C 11 D 3/00 C 11 D 17/00
A	<u>DE - A1 - 2 703 998</u> (HENKEL KGaA) * claim 1 * & US - A - 4 153 570 --		
A	<u>DE - A1 - 2 413 944</u> (UNILEVER N.V.) * claim 7 * -----		CATEGORY OF CITED DOCUMENTS
<input checked="" type="checkbox"/> The present search report has been drawn up for all claims			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
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