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(54) Title: HIGH SUDS FABRIC WASHING LIQUID COMPOSITION

(57) Abstract: The present invention relates to a high suds fabric washing liquid composition comprising a detergent active compound and a detergency builder mixture of citric acid and phosphonic acid, wherein the detergent active species is a mixture consisting of a linear or branched alkyl benzene sulphonate and a fatty alcohol ether sulfate.



„High suds fabric washing liquid composition“

The present invention relates to a high suds fabric washing liquid composition comprising an anionic detergent active compound and a detergency builder mixture. The present invention also describes a method of using such a fabric washing liquid composition.

There are now a number of constraints on the formulation of washing compositions. These include the intended use, which influences product form, and legal restrictions, which may influence phosphorus content and also the processing route taken. Further considerations relate to the main use of the washing composition, i.e. if the composition will be used in hand-washing, in twin-tub or in automatic (front loading) washing machines.

Many consumers that do hand-washing equate a high level of long lasting foam with a strong cleaning power. Accordingly, laundry detergents for hand-washing are high foaming compositions.

At present, most high suds fabric washing compositions are powders. A drawback of many washing powders is that they often leave residues on the treated fabrics and that they are irritating to the skin.

Liquid washing compositions do not have the drawback regarding residues but usually do not provide sufficient and long-lasting foam. For that reason, suds-enhancing and/or foam-enhancing compounds are added. Suds-enhancing and/or foam-enhancing compounds are well known. Polymeric materials are one example of such suds-enhancing components and/or foam-enhancing components.

WO 2000/71240 A1 discloses zwitterionic polymeric suds enhancers to increase the suds and/or foam volume and suds and/or foam retention in suds-forming and/or foam-forming compositions.

A drawback of polymeric suds enhancers is that they do not add to the washing and cleaning performance of a washing composition and, thus, increase the costs of a washing composition.

It is an object of the present invention to provide a high suds fabric washing liquid composition that does not require a suds-enhancing and/or foam-enhancing compound.

This object is achieved by a high suds fabric washing liquid composition comprising an anionic detergent active compound and a detergency builder mixture of citric acid and phosphonic acid,

wherein the detergent active compound is a mixture consisting of a linear alkyl benzene sulphonate and a fatty alcohol ether sulfate.

It has surprisingly been found that the use of a specified anionic detergent active compound mixture and a specified detergency builder mixture leads to a fabric washing liquid composition that has a high foaming power and a long lasting foam.

It is preferred that the anionic detergent active compound is present in an amount of from 13 to 18 % by weight.

It is also preferred that the linear alkyl benzene sulphonate and the fatty alcohol ether sulfate are present in a ratio of 1 : 1.

It has been shown that the best results with regard to foaming power and foaming lasting are achieved when the anionic detergent active compound contains the linear alkyl benzene sulphonate and the fatty alcohol ether sulfate in equal amounts.

Furthermore, it is preferred that the citric acid is present in the acid form and/or neutralized form. In addition, it is preferred that the phosphonic acid is present in the acid form and/or neutralized form. Depending on the pH of the composition the two detergency builders can be present in the neutralized and/or acid form. Their detergency builder performance is not affected by the form.

It is preferred that the composition additionally comprises a perfume.

A perfume gives a pleasant smell to the fabric washing liquid composition and the fabrics treated therewith.

In a further embodiment it may also be preferred that the composition additionally comprises a nonionic detergent active compound. The presence of a nonionic detergent active compound improves the cleaning power of the fabric washing liquid composition and helps to stably and homogeneously disperse the perfume in the fabric washing liquid composition.

In yet a further embodiment it may also be preferred that the composition additionally comprises an amphoteric detergent active compound, preferably a betaine. The presence of an amphoteric detergent active compound, especially of a betaine improves the cleaning power of the fabric washing liquid composition and enhances the foam level of the fabric washing liquid composition. In addition, compositions comprising an amphoteric detergent active compound, preferably a betaine exhibit enhanced skin mildness.

The present application also relates to a method to prolongate the perfume impression on treated fabrics by contacting the fabrics with an aqueous washing liquid of a high suds fabric washing liquid composition comprising a perfume, an anionic detergent active compound and a detergency builder mixture of citric acid and phosphonic acid, wherein the anionic detergent active compound is a mixture consisting of a linear alkyl benzene sulphonate and a fatty alcohol ether sulfate.

The invention is described in greater detail below on the basis of examples, among other things.

In general, the present invention relates to a high suds fabric washing liquid composition comprising a detergent active compound and a detergency builder mixture of citric acid and phosphonic acid, wherein the detergent active compound is a mixture consisting of a linear or branched alkyl benzene sulphonate and a fatty alcohol ether sulfate.

The detergent active compound consists of a linear or branched alkyl benzene sulphonate and a fatty alcohol ether sulfate.

Fatty alcohol ether sulfates are water-soluble salts of the formula $RO(A)_mSO_3M$, in which R is an unsubstituted C_{10} - C_{24} -alkyl or -hydroxyalkyl radical, preferably a C_{12} - C_{20} -alkyl or -hydroxyalkyl radical, more preferably C_{12} - C_{18} -alkyl or -hydroxyalkyl radical. A is an ethylene oxide or propylene oxide unit, m is an integer greater than 0, preferably between approximately 0.5 and approximately 6, more preferably between approximately 0.5 and approximately 4, and M is a cation, for example sodium, potassium, lithium, calcium, magnesium, ammonium or a substituted ammonium cation. Preferred examples include C_{12} - C_{18} fatty alcohol ether sulfates where A is an ethylene oxide unit and the content of ethylene oxide units is 1, 2, 2.5, 3 or 4 mol per mole of the fatty alcohol ether sulfate, and in which M is sodium or potassium. Fatty alcohols with 1 to 4 ethylene oxide units, in particular 1 to 2 ethylene oxide units. A particularly preferred fatty alcohol ether sulfate is sodium lauryl ether sulfate with 2 ethylene oxide units.

The amount of fatty alcohol ether sulfate is preferably from 5 to 10 % by weight of said washing liquid composition and more preferably from 6 to 9 % by weight of said composition.

The linear alkylbenzene sulphonate is preferably a linear alkylbenzene sulphonate having an alkyl chain length of C_8 - C_{15} . In particular, the linear alkylbenzene sulphonate is a C_9 - C_{13} alkyl benzene sulphonate, a C_{10} - C_{13} alkyl benzene sulphonate or a C_{10} - C_{15} alkyl benzene sulphonate.

The amount of linear alkylbenzene sulphonate is preferably from 5 to 10 % by weight of said washing liquid composition, more preferably from 6 to 9 % by weight of said composition.

The linear alkylbenzene sulphonate and the fatty alcohol ether sulfate can be present in the form of their sodium, potassium, or ammonium salts and as soluble salts of organic bases such as mono-, di-, or triethanolamine. The linear alkylbenzene sulphonate and the fatty alcohol ether sulfate are preferably present in the form of their sodium or potassium salts; in particular in the form of the sodium salts.

The fabric washing liquid composition only contains fatty alcohol ether sulfates and linear alkylbenzene sulphonates as anionic detergent active species. In a preferred embodiment the linear alkyl benzene sulphonate and the fatty alcohol ether sulfate are present in a ratio of 1 : 1.

The anionic detergent active compound is preferably present in the fabric washing liquid composition in an amount of from 13 to 18 % by weight and more preferred in an amount of 15 % by weight.

The fabric washing liquid composition also contains a detergency builder mixture of citric acid and phosphonic acid. The use of citric acid or salts thereof is advantageous since they are water-soluble organic detergency builders. The citric acid or its salt is preferably present in the fabric washing liquid composition in an amount of from 0.5 to 2 % by weight.

Phosphonic acids and their phosphonate salts are strong chelating agents. Suitable phosphonates to be used herein may include alkali metal ethane 1-hydroxy diphosphonates (HEDP), alkylene poly (alkylene phosphonate), as well as amino phosphonate compounds, including amino aminotri(methylene phosphonic acid) (ATMP), nitrilo trimethylene phosphonates (NTP), ethylene diamine tetra methylene phosphonates, and diethylene triamine penta methylene phosphonates (DTPMP). The phosphonate compounds may be present either in their acid form or as salts of different cations on some or all of their acid functionalities. Preferred phosphonates to be used herein are diethylene triamine penta methylene phosphonate (DTPMP) and ethane 1-hydroxy diphosphonate (HEDP). Such phosphonate chelating agents are commercially available from Monsanto under the trade name DEQUEST®. The phosphonic acid or its salt is preferably present in the fabric washing liquid composition in an amount of from 0.3 to 0.8 % by weight.

The fabric washing liquid composition may additionally comprise a nonionic detergent active compound at a level up to 5 % by weight of the fabric washing liquid composition.

The nonionic active detergent species that can be used are by preference alkoxyated, advantageously ethoxylated, in particular primary alcohols having by preference 8 to 18 carbon atoms and an average of 1 to 12 mol ethylene oxide (EO) per mol of alcohol, in which the alcohol residue can be linear or preferably methyl-branched in the 2-position, or can contain mixed linear

and methyl-branched residues, such as those that are usually present in oxo alcohol residues. Particularly preferred, however, are alcohol ethoxylates having linear residues made up of alcohols of natural origin having 12 to 18 carbon atoms, e.g. from coconut, palm, tallow, or oleyl alcohol, and an average of 2 to 8 EO per mol of alcohol. The preferred ethoxylated alcohols include, for example, C₁₂₋₁₄ alcohols with 3 EO, 4 EO, 5 EO, or 7 EO, C₉₋₁₁ alcohols with 7 EO, C₁₃₋₁₅ alcohols with 3 EO, 5 EO, 7 EO, or 8 EO, C₁₂₋₁₈ alcohols with 3 EO, 5 EO, or 7 EO, and mixtures thereof, such as mixtures of C₁₂₋₁₄ alcohol with 3 EO and C₁₂₋₁₈ alcohol with 7 EO. The degrees of ethoxylation indicated represent statistical averages, which can correspond to an integral or a fractional number for a specific product. Preferred alcohol ethoxylates exhibit a restricted distribution of homologs (narrow range ethoxylates, NRE). In addition to these nonionic active detergent species, fatty alcohols with more than 12 EO can also be used. Examples of these are tallow fatty alcohol with 14 EO, 25 EO, 30 EO, or 40 EO. Nonionic active detergent species that contain EO and PO groups together in the molecule are also usable according to the present invention. Block copolymers having EO-PO block units or PO-EO block units, but also EO-PO-EO copolymers or PO-EO-PO copolymers, can be used in this context. Also usable, of course, are mixed alkoxyated nonionic active detergent species in which EO and PO units are distributed statistically rather than in block fashion. Such products are obtainable by the simultaneous action of ethylene oxide and propylene oxide on fatty alcohols. These nonionic active detergent species are obtainable, for example, under the commercial name Dehydol® (from Cognis).

Also suitable nonionic active detergent species are mixtures of a (more) branched ethoxylated fatty alcohol and a non-branched ethoxylated fatty alcohol, such as mixtures of C₁₆₋₁₈ fatty alcohol having 7 EO and 2-propylheptanol having 7 EO.

Further classes of nonionic active detergent species used in preferred fashion are alkoxyated fatty acid alkyl esters, active detergent species of the amine oxide type, polyhydroxy fatty acid amides or alkylpolyglucosides.

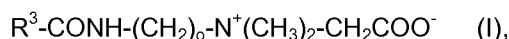
Most preferred the fabric washing liquid composition comprises a C₁₂₋₁₈ fatty alcohol having 7 EO or a C₁₃₋₁₅ oxo alcohol having 7 EO as nonionic surfactant.

Most preferred the fabric washing liquid composition comprises a mixture of nonionic and anionic active detergent compounds.

The fabric washing liquid composition may also additionally comprise an amphoteric active detergent compound at a level up to 2 % by weight of the fabric washing liquid composition. The amphoteric active detergent compound is preferably present in the fabric washing liquid composition in an amount of from 0.5 to 1.5 % by weight.

In a preferred embodiment, the amphoteric active detergent compound is a betaine. A betaine is any neutral chemical compound with a positively charged cationic functional group such as a quaternary ammonium or phosphonium cation (generally: onium ions) which bears no hydrogen atom and with a negatively charged functional group such as a carboxylate group which may not be adjacent to the cationic site.

Suitable betaines comprise compounds represented by the following formula



wherein

R^3 is a linear or branched, substituted or unsubstituted alkyl-, aryl- or alkylarylgroup, and
 o is an integer of from 1 to 4, preferably 3.

Preferred surfactants represented by formula (I) are those wherein

R^3 is a linear or branched, substituted or unsubstituted C_8 - C_{18} -alkylgroup, preferably an alkylgroup which is derived from coconut oil, and
 o is 3.

A preferred betaine for use in the fabric washing liquid composition is cocamidopropyl betaine (CAPB), an organic compound derived from coconut oil and dimethylaminopropylamine.

In a preferred embodiment, the fabric washing liquid composition additionally comprises a perfume. Non-limiting examples of suitable perfumes include blooming perfumes, perfume oils, and perfume raw materials comprising alcohols, ketones, aldehydes, esters, ethers, nitriles alkenes, and mixtures thereof.

In another preferred embodiment, the fabric washing liquid composition comprises an encapsulated perfume and a free perfume.

Preferably, the fabric washing liquid composition is an aqueous fabric washing liquid composition.

In addition to the anionic detergent active compound and the builder mixture, a fabric washing liquid composition may contain further ingredients that further improve the applications-engineering or aesthetic properties of the fabric washing liquid composition. In the context of the present invention, the fabric washing liquid composition by preference additionally contains one or more substances from the group of bleaching agents, bleach activators, bleach catalysts, enzymes, non-aqueous solvents, pH adjusting agents, thickeners, fluorescing agents, dyes, hydrotopes, silicone oils, anti-redeposition agents, anti-gray agents, shrinkage preventers, wrinkle protection agents, dye transfer

inhibitors, antimicrobial active substances, germicides, fungicides, antioxidants, preservatives, corrosion inhibitors, antistatic agents, bittering agents, ironing adjuvants, proofing and impregnation agents, swelling and anti-slip agents, softening compounds and UV absorbers.

From the above mentioned further ingredients enzymes, non-aqueous solvents, pH adjusting agents, fluorescing agents, dyes, silicone oils, soil-release polymers, anti-gray agents, dye transfer inhibitors, thickeners and preservatives are most preferred included into a fabric washing liquid composition.

The fabric washing liquid composition according to the present invention can be used to wash and/or clean textile fabrics.

The fabric washing liquid composition is manufactured using usual and known methods and processes. For example, the constituents of the fabric washing liquid composition can be simply mixed in agitator vessels, the water, non-aqueous solvent, and active detergent compounds usefully being prepared first. After cooling under stirring the further constituents are then added in portions.

Table 1 below shows the composition of two fabric washing liquid compositions E1 and E2 according to the present invention. Quantities are indicated in wt% of active matter.

Table 1:

	E1	E2
C ₁₁₋₁₃ -alkylbenzenesulfonic acid, Na salt	7	8
Sodium lauryl ether sulfate with 2 EO	7	8
C ₁₂₋₁₈ fatty alcohol with 7 EO	1	1
Citric acid, Na salt	1.5	1.5
Ethane 1-hydroxy diphosphonate, Na salt	0.4	0.4
Fluorescent dye	0.05	0.05
Perfume	0,6	0,6
Enzymes, dyes, preservatives	+	+
Water	To make 100	

The fabric washing liquid compositions E1 and E2 gave rise to stable fabric washing liquid compositions.

The fabric washing liquid compositions E1 and E2 showed a good cleaning performance on textile fabrics. In addition, textile fabrics treated with one of the fabric washing liquid compositions E1 and E2 exhibit an appealing and long-lasting smell.

The performance of fabric washing liquid composition E1 was compared with a commercially available high suds fabric washing solid composition.

The high suds fabric washing solid composition S1 comprised 15.7 % by weight of the sodium salt of an alkylbenzenesulfonic acid, 2 % by weight of a fatty alcohol sulfate, 0.3 % by weight of soap, 27.9 % by weight of a builder mixture comprising sodium silicate, magnesium silicate, soda ash, Zeolith A, sodium salt of citric acid and bicarbonate, 5.2 % by weight of bleach and bleach activators, enzymes, optical brightener, perfume, water and filler (sodium sulfate).

In contrast to fabric washing liquid composition E1, the comparative solid fabric washing composition did not contain a fatty alcohol ether sulfate and a phosphonic acid (salt).

When used under identical washing conditions (dosages: E1 = 150 g/wash load and S1 = 80 g/wash load) the cleaning and whitening power of both detergents was considered equal by consumers (number of consumers: 200). Regarding ease of rinsing and ease of dissolving consumers preferred fabric washing liquid composition E1 over comparative composition S1 (53 % and 74 % versus 25 % and 11 %). 54 % of the consumers also preferred the lather of fabric washing liquid composition E1 whereas only 26 % preferred the lather of comparative detergent S1. Regarding the assessment criterion "hand care" 44% of the consumers preferred fabric washing liquid composition E1 and only 15 % of the consumers preferred comparative detergent S1. The rest of the consumers considered both compositions equally good.

Regarding the perfume impression consumers clearly preferred fabric washing liquid composition E1 over comparative composition S1.

Table 2 below shows the consumer preferences (in %) regarding the perfume during the different stages of a washing process.

Table 2: Perfume evaluation

	E1	S1
Overall perfume impression	68	23
Open pack	68	23
During wash	68	23
Wet Laundry	68	23
Dry garments	69	22
Long lasting	66	22

The fact that 76 % preferred fabric washing liquid composition E1 regarding the softness of fabrics shows that the builder mixture of E1 seems to be much more effective in binding calcium and other metal ions and thus, effectively hinder the formation of undesirable incrustations on the textile fabrics.

In another test the foaming power and foaming lasting of liquid compositions E1 and E2 and solid composition S1 were evaluated.

A bucket was loaded with 38 liter of water and one of the detergents to be tested (dose: E1 = 150g, E2 = 150g and S1 = 80g) at 30 °C. The resulting aqueous mixture was stirred for 2 minutes and the foam level was determined. Afterwards 4 SBL 2004 towels with standardized soiling (32 g soil ballast) were added to the aqueous mixtures and after 10 minutes of stirring the foam level was again determined. The procedure was repeated twice. The foam levels are shown in table 3 below.

Table 3: Foam levels in cm

Time and soil load	E1	E2	S1
2 min without soil load	26	24.5	16
10 min and 4 SBL 2004 towels	19	22	2
20 min and 8 SBL 2004 towels	14	16.5	--
30 min and 12 SBL 2004 towels	9	13.5	--

The data show that liquid compositions E1 and E2 exhibit much better foaming power and foaming lasting than solid composition S1. Even with further increased soil load, liquid compositions E1 and E2 still exhibit high foam levels.

Table 4 below shows the composition of two further fabric washing liquid compositions E3 and E4 according to the present invention. Quantities are indicated in wt% of active matter.

Table 4:

	E3	E4
C ₁₁₋₁₃ -alkylbenzenesulfonic acid, Na salt	8	8
Sodium lauryl ether sulfate with 2 EO	8	8
C ₁₂₋₁₈ fatty alcohol with 7 EO	1	1
Citric acid, Na salt	1.4	1.5
Ethane 1-hydroxy diphosphonate, Na salt	0.4	0.4
Fluorescent dye	0.1	0.05

Photobleach	0.01	
Perfume	0,6	0,6
Opacifier	--	0,1
Enzymes, dyes, preservatives	+	+
Water	To make 100	

Also, the fabric washing liquid compositions E3 and E4 showed a good cleaning performance on textile fabrics as well as excellent foaming power and foaming lasting. In addition, textile fabrics treated with one of the fabric washing liquid compositions E3 and E4 exhibit an appealing and long-lasting smell.

Table 5 below shows the composition of another fabric washing liquid composition E5 according to the present invention. Quantities are indicated in wt% of active matter.

Table 5:

	E5
C ₁₁₋₁₃ -alkylbenzenesulfonic acid, Na salt	6.5
Sodium lauryl ether sulfate with 2 EO	6.5
C ₁₂₋₁₈ fatty alcohol with 7 EO	0.7
Cocamidopropyl betaine	1
Citric acid, Na salt	1.5
Ethane 1-hydroxy diphosphonate, Na salt	0.4
Fluorescent dye	0.04
Perfume	0.55
Enzymes, dyes, preservatives	+
Water	To make 100

The fabric washing liquid composition E5 gave rise to a stable fabric washing liquid composition.

The fabric washing liquid composition E5 showed a good cleaning performance on textile fabrics. Compared to fabric washing liquid composition E1 showed improved cleaning performance on the following stains: grass, mustard, tomato puree, cocoa, grape juice, baby food, strawberry jam, hamburger grease and red wine. In addition when compared to commercially available powder compositions, fabric washing liquid composition E5 showed improved cleaning performance on the following stains grape juice, red wine, strawberry jam, tomato puree and cocoa.

In another test the foaming power and foaming lasting of liquid composition E5 and solid composition S1 were evaluated.

A bucket was loaded with 38 liter of water and one of the detergents to be tested (dose: E5 = 150g and S1 = 80g) at 30 °C. The resulting aqueous mixture was stirred and the foam level was determined. Afterwards 4 SBL 2004 towels with standardized soiling (32 g soil ballast) were added to the aqueous mixtures and after 10 minutes of stirring the foam level was again determined. The procedure was repeated once. The foam levels are shown in table 6 below.

Table 6: Foam levels in cm

Time and soil load	E5	S1
0 min (without soil load)	24.4	17.8
10 min and 4 SBL 2004 towels	16.8	9
20 min and 8 SBL 2004 towels	2	0.5

The data show that liquid composition E5 exhibit much better foaming power and foaming lasting than solid composition S1. Even with further increased soil load, liquid composition E1 still exhibit high foam levels.

Claims

1. A high suds fabric washing liquid composition comprising an anionic detergent active compound and a detergency builder mixture of citric acid and phosphonic acid, wherein the anionic detergent active compound is a mixture consisting of a linear alkyl benzene sulphonate and a fatty alcohol ether sulfate.
2. The high suds fabric washing liquid composition according to claim 1, wherein the anionic detergent active compound is present in an amount of from 13 to 18 % by weight.
3. The high suds fabric washing liquid composition according to claim 1, wherein the linear alkyl benzene sulphonate and the fatty alcohol ether sulfate are present in a ratio of 1 : 1.
4. The high suds fabric washing liquid composition according to any of the claims 1 to 3, wherein the citric acid is present in the acid form and/or neutralized form.
5. The high suds fabric washing liquid composition according to any of the claims 1 to 4, wherein the phosphonic acid is present in the acid form and/or neutralized form.
6. The high suds fabric washing liquid composition according to any of the claims 1 to 5, wherein the composition additionally comprises a perfume.
7. The high suds fabric washing liquid composition according to any of the claims 1 to 6, wherein the composition additionally comprises a non-ionic detergent active compound.
8. The high suds fabric washing liquid composition according to any of the claims 1 to 7, wherein the composition additionally comprises an amphoteric detergent active compound, preferably a betaine.
9. A method to prolongate the perfume impression on treated fabrics by contacting the fabrics with an aqueous washing liquid of a high suds fabric washing liquid composition comprising a perfume, an anionic detergent active compound and a detergency builder mixture of citric acid and phosphonic acid, wherein the anionic detergent active compound is a mixture consisting of a linear alkyl benzene sulphonate and a fatty alcohol ether sulfate.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/061913

A. CLASSIFICATION OF SUBJECT MATTER INV. C11D1/37 C11D1/83 C11D3/00 C11D3/20 C11D3/36 C11D1/22 C11D1/29 ADD. 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) 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 practicable, search terms used) EPO-Internal, WPI Data														
C. DOCUMENTS CONSIDERED TO BE RELEVANT <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 10%; padding: 5px;">Category*</th> <th style="width: 70%; padding: 5px;">Citation of document, with indication, where appropriate, of the relevant passages</th> <th style="width: 20%; padding: 5px;">Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">X</td> <td style="padding: 5px;">US 6 090 762 A (CLAPPERTON RICHARD MALCOLM ET AL) 18 July 2000 (2000-07-18) example 24 -----</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1,4-7</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">X</td> <td style="padding: 5px;">US 2007/225198 A1 (] PANANDIKER RAJAN KESHAV ET AL) 27 September 2007 (2007-09-27) examples 24-27 -----</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1,4-8</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">X</td> <td style="padding: 5px;">WO 92/03529 A1 (NOVO NORDISK AS) 5 March 1992 (1992-03-05) example 1 -----</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1,4-7</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	X	US 6 090 762 A (CLAPPERTON RICHARD MALCOLM ET AL) 18 July 2000 (2000-07-18) example 24 -----	1,4-7	X	US 2007/225198 A1 (] PANANDIKER RAJAN KESHAV ET AL) 27 September 2007 (2007-09-27) examples 24-27 -----	1,4-8	X	WO 92/03529 A1 (NOVO NORDISK AS) 5 March 1992 (1992-03-05) example 1 -----	1,4-7
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☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.														
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Date of the actual completion of the international search 21 August 2013	Date of mailing of the international search report 10/09/2013													
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