# (19) World Intellectual Property Organization

International Bureau





### (43) International Publication Date 27 June 2002 (27.06.2002)

### (10) International Publication Number WO 02/50225 A1

(51) International Patent Classification<sup>7</sup>: C11D 3/00, 3/20, 3/43, 3/28

(21) International Application Number: PCT/EP01/15402

(22) International Filing Date:

18 December 2001 (18.12.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

00311526.8 21 December 2000 (21.12.2000)

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(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

#### Published:

with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: ANTIMICROBIAL CLEANING COMPOSITIONS

(57) Abstract: An aqueous antimicrobial cleaning composition suitable for the hygienic cleaning of hard surfaces, which composition comprises: (i) at least one surfactant, (ii) at least one acid, and (iii) a mixture of solvents comprising (a) an N-alkylpyrrolidone derivative and (b) a co-solvent of the general formula  $R_1$ -O-(EO)<sub>m</sub>-(PO)<sub>n</sub>- $R_2$ , wherein  $R_1$  and  $R_2$  are independently  $C_{2-6}$  alkyl or hydrogen, but not both hydrogen, m and n are independently 0-5, EO represents an ethyleneoxy group and PO represents a propyleneoxy group.





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### ANTIMICROBIAL CLEANING COMPOSITIONS

### Technical Field

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The present invention relates to an aqueous antimicrobial cleaning composition which is designed in particular for the hygienic cleaning of surfaces, such as hard surfaces in the household, or in institutional or hospital environments, or the surface of the skin, or fabric surfaces. The composition contains an antimicrobially active mixture of anionic surfactant and acid, in combination with a specific mixture of solvents, and shows improved hygienic cleaning performance.

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### Background to the Invention and Prior Art

Cleaning compositions designed for application to surfaces, such as hard surfaces in the household, or in institutional or hospital environments, or the surface of the skin, or fabric surfaces, generally comprise one or more surfactants, and, optionally, one or more antimicrobial actives and/or solvents.

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Such surfaces are often contaminated with bacteria and other microorganisms, which present a risk to human health, especially when they are present near food.

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The biocidal activity of surfactants is, with a few notable exceptions, low and it is therefore commonplace to add a separate antimicrobial active to compositions.

5 The antimicrobial effectiveness of organic acids is known in the literature. Organic acids are utilised in cleaning products to provide antimicrobial activity, but generally do not achieve a sufficient level of bacterial kill, especially when present in formulations at relatively low levels.

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Some surfactants have been found to potentiate the effects of organic acids. US 4,975,217 discloses a synergistic germicidal activity for combinations of organic acid and surfactants, when used as active ingredients in germicidal products which are applied directly to the skin. Citric/malic acid in combination with anionic surfactant is particularly preferred.

W098/55096 discloses that improved germ reduction and improved residual effectiveness can be obtained in a skin cleansing antimicrobial wipe which is impregnated with a mixture of an anionic surfactant and a proton donating agent (such as organic acid), by the addition of a further antibacterial active such as TRICLOSAN.

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The present inventors have found that excellent cleaning performance (particularly with respect to the removal of fatty soil) and hygiene benefits can be obtained from a composition which comprises an acid, a surfactant, a solvent which is an N-alkylpyrrolidone derivative, and a co-solvent

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which is selected from certain alcohols, ethers and their respective alkoxylated derivatives.

Compositions of this invention are particularly suited for the surfaces such as hard surfaces in the household, or in institutional or hospital environments, or the surface of the skin, or fabric surfaces. Surprisingly the compositions provide the requisite level of bacterial kill for this type of application without requiring the addition of any further antibacterial actives such as those described in WO98/55096.

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WO95/21238 describes an acidic microemulsion or acidic all-purpose cleaner for hard surfaces which removes soap scum, lime scale and grease which includes an anionic surfactant, at least one organic acid, such as a mono-, di-, or tricarboxylic acid, and an N-alkyl-2-pyrrolidone.

US 5 470 508 discloses an aqueous oil removal/degreasing composition containing higher-alkyl pyrrolidone such as Noctyl pyrrolidone, anionic surfactant and water.

GB 2 230 791 refers to a propylene glycol based solvent, such as propylene glycol t-butyl ether, in combination with N-methyl-2-pyrrolidone, for the removal of grease and varnish from metallic surfaces such as automotive parts.

WO98/55569 and WO98/55570 relate to aqueous hard surface cleaners with improved residue removal and reduced filming/streaking. The compositions comprise a mixture of anionic surfactants, an organic solvent such as an alkylene glycol ether, and preferably include a 1-alkyl-2-pyrrolidone

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for dispersal of fragrance oils and other insoluble organic materials in the compositions. The compositions are buffered to pH greater than 6.5 with a nitrogenous buffer.

5 EP 0 598 257 describes a water-based solution for reclaiming paint from spray booths with an N-alkyl pyrrolidone, preferably N-methyl pyrrolidone, a surfactant, preferably nonionic, and an alkylene glycol monoalkyl ether such as tripropylene glycol mono methyl ether.

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EP 0 273 594 discloses a disposable semi-moist wipe for touch-up or light duty cleaning of bathroom or kitchen soil, containing a liquid cleaning composition comprising anionic or nonionic surfactant, one or more solvents for oil and dirt such as N-methyl pyrrolidone and ethylene or propylene glycol ethers, and sufficient hydroxide compound to produce a composition pH of 8 to 12.

- EP 0 690 906 describes a multi-surface cleaner for the

  removal of greasy residues comprising an amine oxide or
  quaternary ammonium salt surfactant and a polar organic
  compound of defined water solubility such as certain of the
  propylene glycol ethers and N-alkyl pyrrolidones.
- US 5 332 526 provides an environmentally safe paint stripping composition comprising N-methyl-2-pyrrolidone and lower alkyl ethers of propylene glycol and dipropylene glycol.
- 30 None of the above referenced prior art discloses or suggests the combination of ingredients which characterises

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compositions of the present invention as defined below, and the improved surface cleaning and hygienic performance obtainable thereby.

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### Summary of the Invention

The present invention provides an aqueous antimicrobial cleaning composition suitable for the hygienic cleaning of surfaces, which composition comprises:

- (i) at least one surfactant,
- (ii) at least one acid, and

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(iii) a mixture of solvents comprising (a) an N-alkylpyrrolidone derivative and (b) a co-solvent of the general formula  $R_1$ -O- $(EO)_m$ - $(PO)_n$ - $R_2$ , wherein  $R_1$  and  $R_2$  are independently  $C_{2-6}$  alkyl or hydrogen, but not both hydrogen, m and n are independently 0-5, EO represents an ethyleneoxy group and PO represents a propyleneoxy group.

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### 25 Detailed Description of the Invention

### Surfactant

Compositions of the invention contain a surfactant which is preferably anionic in nature.

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Suitable anionic surfactants for use herein include alkyl sulphates, alkyl sulphonates, alkyl aryl sulphonates, alkyl alkoxylated sulphates, or mixtures thereof.

5 Alkyl sulphates are particularly preferred.

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Suitable alkyl sulphate surfactants for use herein are represented by the formula RSO<sub>4</sub> M wherein R represents a hydrocarbon group which may suitably be a straight or branched alkyl radical containing from 6 to 20 carbon atoms, or an alkyl phenyl radical containing from 6 to 18 carbon atoms in the alkyl group. Preferably R is a straight or branched alkyl radical containing from 6 to 20 carbon atoms. M is hydrogen or a cation such as an alkali metal cation (e.g., sodium, potassium, lithium, calcium, and magnesium) or ammonium or substituted ammonium (e.g., methyl-, dimethyl-, and trimethyl ammonium cations and quaternary ammonium cations, such as tetramethyl-ammonium and dimethyl piperidinium cations and quaternary ammonium cations derived from alkylamines such as ethylamine, diethylamine, triethylamine, and mixtures thereof).

An example of a commercially available branched alkyl sulphate is DACPON 27-23 AL, ex CONDEA. This material can be described as a branched, essentially  $C_{12}/C_{13}$  sodium alkyl sulphate of formula  $(R_1)CH(R_2)-OSO_3Na$  where  $R_1$  is  $C_{1-4}$  alkyl and  $R_2$  is  $C_{8-11}$  alkyl.

An example of a commercially available linear alkyl sulphate 30 is EMPICOL LX28, ex Albright & Wilson. This material can be

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described as  $R-OSO_3Na$  where R is  $C_{10-16}$  alkyl, mainly  $C_{12-14}$  alkyl.

Suitable alkyl sulphonates for use herein include watersoluble salts or acids of the formula  $RSO_3M$  wherein R is a  $C_6$ - $C_{20}$  linear or branched, saturated or unsaturated alkyl group, preferably a  $C_8$ - $C_{18}$  alkyl group and more preferably a  $C_{10}$ - $C_{16}$  alkyl group, and M is hydrogen or a cation such as those listed above for alkyl sulphate surfactants.

An example of a commercially available  $C_{14}$ - $C_{16}$  alkyl sulphonate is HOSTAPUR SAS ex Hoechst.

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Suitable alkyl aryl sulphonates for use herein include water-soluble salts or acids of the formula  $RSO_3M$  wherein R is an aryl group, preferably a benzyl group, substituted by a  $C_6$ - $C_{20}$  linear or branched saturated or unsaturated alkyl group, preferably a  $C_8$ - $C_{18}$  alkyl group and more preferably a  $C_{10}$ - $C_{16}$  alkyl group, and M is hydrogen or a cation such as those listed above for alkyl sulphate surfactants.

Examples of commercially available alkyl aryl sulphonates are the alkyl benzene sulphonates available from Albright & Wilson under the trade name NANSA.

Suitable alkyl alkoxylated sulphate surfactants for use herein are according to the formula  $RO(A)mSO_3M$  wherein R is an unsubstituted  $C_6-C_{20}$  alkyl or hydroxyalkyl

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group having a  $C_6$ - $C_{20}$  alkyl component, preferably a  $C_8$ - $C_{18}$  alkyl or hydroxyalkyl, more preferably  $C_{10}$ - $C_{16}$  alkyl or hydroxyalkyl, A is an ethoxy or propoxy unit, m is greater than zero, typically between 0.5 and 6, more preferably between 0.5 and 3, and M is H or a cation such as those listed above for alkyl sulphate surfactants.

An example of a commercially available alkyl alkoxylated sulphate surfactant is EMPICOL ESA 70, ex Albright & Wilson. This material can be described as  $RO-(CH_2CH_2O)\,SO_3Na \ \, \text{where R is} \ \, C_{10-16} \,\, \text{alkyl} \,, \,\, \text{mainly} \,\, C_{12-14} \,\, \text{alkyl} \,.$ 

#### Acid

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Compositions of the invention contain an acid which is preferably organic in nature.

Suitable organic acids for use herein include carboxylic acids and mixtures thereof.

The carboxylic acids and mixtures thereof may suitably be selected from aliphatic, cycloaliphatic or aromatic mono-, di- , tri- or polycarboxylic acids which generally contain 2 to 10 carbon atoms, preferably 3 to 6 carbon atoms in the molecule. Hydroxycarboxylic acids may also be used.

Examples of suitable carboxylic acids include caprylic acid, propionic acid, azelaic acid, caproic acid, hydroxybenzoic acid, salicylic acid, malic acid, maleic acid, fumaric acid,

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succinic acid, glutaric acid, adipic acid, tartaric acid and mixtures thereof.

Especially preferred are mixtures of dicarboxylic acids, in particular mixtures including adipic, glutaric and succinic acid. These mixtures are preferred as they are commercially available. Typical commercially available mixtures comprise 30-35% adipic acid, 45-50% glutaric acid and 10-18% succinic acid. Such a mixture is available as SOKALAN DCS ex BASF.

10 Another suitable mixture is available as RADIMIX ex Radici.

The use of essentially pure acids is not excluded but these have limited commercial availability.

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### N-alkylpyrrolidone derivative

Compositions of this invention contain an N-alkylpyrrolidone derivative.

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Suitable N-alkyl pyrrolidone derivatives are N-(n-alkyl) pyrrolidones where the alkyl group has from 6 to 20, preferably from 8 to 14 carbon atoms.

25 Preferred N-alkyl pyrrolidone derivatives are N-(n-octyl)-2-pyrrolidone, N-(n-decyl)-2-pyrrolidone, N-(n-decyl)-2-pyrrolidone.

Particularly preferred is N-(n-octyl)-2-pyrrolidone,

30 available commercially as SURFADONE LP-100 ex International

Speciality Products, Inc.

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#### Co-solvent

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Compositions of the invention contain a co-solvent which can be characterised by the general formula  $R_1$ -O- $(EO)_m$ - $(PO)_n$ - $R_2$ , wherein  $R_1$  and  $R_2$  are independently  $C_{2-6}$  alkyl or hydrogen, but not both hydrogen, m and n are independently 0-5, EO represents an ethyleneoxy group and PO represents a propyleneoxy group.

Suitable materials of this type include the  $C_{1-4}$  alkyl ethers 10 of alkylene glycols such as ethylene glycol, propylene glycol and oligomers thereof having 2 or 3 repeating units. Examples include monoethers such as ethylene glycol mono-nbutyl ether, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol mono-n-hexyl ether, 15 propylene glycol monomethyl ether, propylene glycol monoethyl ether, propylene glycol mono-n-butyl ether, isopropylene glycol monoethyl or monopropyl or monobutyl ether, diethylene glycol monoethyl or monopropyl or 20 monobutyl ether, di- or tripropylene glycol monomethyl ether, di- or tripropylene glycol monoethyl ether, and mixtures thereof.

Preferably the co-solvent is selected from di-ethylene glycol mono n-butyl ether, mono-ethylene glycol mono n-butyl ether, propylene glycol n-butyl ether, isopropanol, ethanol, butanol and mixtures thereof.

Particularly preferred is propylene glycol n-butyl ether, 30 sold by Dow Chemical Company as Dowanol PnB. Other suitable

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materials include Dowanol PM and Dowanol DPnB, both also commercially available from Dow Chemical Company.

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Compositions of the invention will generally have a pH between 3.0 and 6.0, preferably 3.2-4.5.

It is believed that the relatively low pH of the composition is important in achieving both the cleaning and the antimicrobial synergies which are exhibited by the composition according to the invention. However below pH 3.0 surface damage may occur. The most preferred pH is around 3.8.

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A water soluble base such as an alkali metal hydroxide, (typically sodium hydroxide) is generally used to regulate the pH to the required level.

#### 20 Product Form and Ingredient Levels

Compositions of the invention are aqueous compositions which preferably contain relatively low levels of actives.

Typically the principal ingredient is water, which is

normally present at a level of at least 50%, preferably at least 80%, more preferably at least 90%, by weight based on total weight. The use of distilled or demineralised water is preferred, but not essential to the invention.

30 Compositions of the invention are designed in particular for the hygienic cleaning of hard surfaces. By "hard surfaces"

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is meant those surfaces which are typically found in the household, or in institutional or hospital environments, and which are prone to microbial contamination. Examples include lavatory fixtures, lavatory appliances (toilets, bidets, shower stalls, bathtubs and bathing appliances), wall and flooring surfaces and those surfaces associated with kitchen environments and other environments associated with food preparation.

10. Compositions of the invention may also be useful in laundry or personal care applications where an antimicrobial effect is desirable, such as in hygienic stain removal products for fabrics, or hygienic products for the skin such as antimicrobial hand cleansers.

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Compositions of the invention may suitably be supplied as a ready to use product packaged in a conventional plastics container. Examples of suitable packaging forms include spray dispensers, foam dispensers, toilet cleaner

dispensers, and all packaging forms which are adapted to dose the product for neat use. By "neat use" is meant that the product is not diluted by the consumer prior to use.

Typical ingredient percentages (by weight based on total weight) for such a product will be as follows:

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Surfactant: from 1 to 10%, preferably about 4%

Acid: from 2 to 6%, preferably about 4%

N-alkylpyrrolidone derivative: from 0.2 to 4%, preferably from 0.5 to 2%.

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Co-solvent: from 0.2 to 4%, preferably from 0.5 to 2%.

Water: at least 70%, preferably at least 85%.

5 The above composition may be used by the consumer in neat form or diluted form. By "diluted form" is meant that the composition is diluted by the user, typically with water. If the composition is diluted by the consumer prior to use, then a suitable dilution level of the composition is from 1 to 50, preferably from 4 to 30, most preferably from 15 to 25 times its weight of water. Neat use is preferred.

The composition may be formulated as a liquid having two or more phases which can be temporarily converted to an emulsion by shaking. Electrolytes such as citrate and polyacrylate are typically used to produce such multiphase compositions.

Most preferably, compositions of the invention may be

supplied in the form of wipes. By "wipe" it is meant a

disposable substrate such as a porous or absorbent sheet or

cloth which has been pre-treated with a composition of the

invention so as to incorporate the composition of the

invention into or onto the substrate prior to its use by the

consumer.

This product form is particularly preferred since it allows for safe and convenient one-step hygienic cleaning disinfection of surfaces by the user.

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Typically the wipe is formed by absorbing a composition of the present invention onto the substrate to form a moist wipe. A batch of wipes can then be placed in a container which can be opened when needed and when closed, sufficiently seals to prevent evaporation of any components from the composition. The composition of the invention is impregnated at the desired weight onto one or both sides of a substrate which may be formed from any woven or nonwoven fibre, fibre mixture or foam of sufficient wet strength and absorbency to hold an effective amount of the composition.

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Preferable materials used to form the substrate include in general all nonwoven materials with sufficient liquid absorption capacity to contain the aqueous composition of the invention and to deliver it in use.

Illustrative examples of suitable nonwoven materials include fibres from natural sources such as viscose, cellulose, or from synthetic origin such as polypropylene or polyester. Especially suitable are mixtures of these materials such as viscose/polyester, viscose/polypropylene, and cellulose/polyester.

Optionally one or more latex binders can be added.

These latex binders can typically include one or more monomers selected from styrene-2-ethyl hexyl acrylate, butyl acrylate, methyl methacrylate, ethyl acrylate, methyl acrylate, acrylonitrile and vinyl acetate.

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The materials can be formed into webs using technologies generally known in the art such as carding, drylaid, wetlaid, airlaid and extrusion.

Webs can be bonded using technologies known in the art such as needlepunch, stitchbond, hydroentangling, chemical bonding, thermal bonding, spunbinding and meltblowing.

A commercially available example of a suitable material used to form the substrate is Akena VPE ex Orlandi, (a viscose/polyester material available in different specific weights varying from 40 grams/sq. m to 150 grams/sq. m).

Other commercially available examples include Tecnojet C500, a spunlace material containing 70% viscose and 30% polyester of a specific weight of 50 grams/ sq. m, available from Tecnofibra, Tecnojet A 1000, a material containing 100% viscose with a specific weight of 100 grams/ sq. m, available from Tecnofibra, and Hydraspun 8579, a nonwoven fabric manufactured from a blend of cellulosic and synthetic fibres with a specific weight of 48 grams/ sq. m.

Compositions of the invention designed to be used in the form of a wipe will typically have ingredient percentages (by weight based on total weight) as follows:

Surfactant: from 0.1 to 2%, preferably about 0.3%

Acid: from 0.1 to 2.0%, preferably from 0.4 to 0.8%.

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N-alkylpyrrolidone derivative: from 0.05 to 2%, preferably from 0.2 to 0.6%.

Co-solvent: from 0.05 to 5%, preferably from 0.1 to 3%, more preferably from 0.2 to 5%.

Water: at least 80%, preferably at least 90%.

In a wipe according to the invention, the weight ratio of composition of the invention to substrate suitably ranges from 1:1 to 6:1, and is preferably from 1:1 to 4:1, more preferably from 2:1 to 3:1.

# 15 Optional Ingredients

The composition according to the invention can contain other optional ingredients which aid in their cleaning performance and maintain the physical and chemical stability of the product.

Examples include: perfumes, colours and dyes, further hygiene agents, foam-control agents, viscosity modifying agents and mixtures thereof.

The invention will now be illustrated by the following nonlimiting Examples, in which all percentages are by weight based on total weight, unless otherwise indicated.

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### **EXAMPLES**

# Examples 1 to 4

5 Examples 1 to 4 having ingredients as shown in the following Table illustrate formulations according to the invention:

INGREDIENT	wt%					
	Example	Example	Example	Example		
	1	2	3	4		
Glutaric acid	0.40	0.00	0.00	0.00		
Succinic acid	0.00	0.40	0.00	0.00		
Adipic acid	0.00	0.00	0.40	0.00		
Mix of acids (1)	0.00	0.00	0.00	0.4		
C <sub>12-14</sub> sodium alkyl	0.30	0.15	0.3	0.30		
sulphate 28% active <sup>(2)</sup>						
Isopropyl alcohol	1.80	1.80	1.80	1.80		
Propylene glycol	0.30	0.30	0.30	0.30		
mono-n-butyl ether (3)	*					
N-(n-octyl)-2-	0.40	0.40	0.40	0.40		
pyrrolidone (4)						
Sodium Hydroxide	0.05	0.05	0.05	0.05		
Water	to 100%	to 100%	to 100%	to 100%		

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- (1) Sokalan DCS, ex BASF
- (2) Dacpon 27-23 AL, ex Condea
- 5 (3) Dowanol PnB, ex Dow
  - (4) Surfadone LP100, ex ISP

The formulations of Examples 1 to 4 all had a pH of 3.8

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The disinfecting properties of the formulations of Examples 1 to 4 were measured by evaluating their bactericidal activity according to the methodology described in European standard EN 1276. This specifies a test method and requirements for the minimum bactericidal activity of a disinfecting composition. The formulations of Examples 1 to 4 pass this test at high soil with 5 minutes contact time, giving at least log 5 kill against the test bacterial strains P.aeruginosa, E.coli, S.aureus and Ent.hirae.

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### Examples 5 & 6

Examples 5 and 6 (according to the invention) and Comparative Examples A and B (not according to the invention) were prepared.

Each composition was used to make an antimicrobial wipe by impregnating a substrate with that particular composition. The substrate was spunlace comprising viscose/polyester at a ratio of 70:30 with a specific weight of 50 grams/sq. m. In

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each case, the composition to substrate weight ratio was 2.6:1.

The compositions were evaluated for their cleaning

performance on fatty soil. The following Table shows the ingredients of the compositions used and the results of the evaluation of cleaning performance:

	Comparative Example A	Example 5	Comparative Example B	Example 6
Ingredient	wt%	wt%	wt%	wt%
Isopropyl Alcohol	2.0	2.0	2.0	2.0
Monopropylene glycol n-butyl ether	1.0	0.5	-	_
Dipropylene glycol n-butyl ether	_	-	-	0.5
N-octyl pyrrolidone	_	0.5	1.0	0.5
Mix of acids (1)	0.4	0.4	0.4	0.4
Primary alkyl sulphate	0.3	0.3	0.3	0.3
Perfume	0.15	0.15	0.15	0.15
Cleaning performance on scale 1-10 1= poor 10= excellent	7	9	7.5	9

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It can be seen that Examples 5 & 6 are both superior to Comparative Example A and Comparative Example B in terms of their cleaning performance on fatty soil.

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### CLAIMS

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- 1. An aqueous antimicrobial cleaning composition suitable for the hygienic cleaning of hard surfaces, which composition comprises:
  - (i) at least one surfactant,
  - (ii) at least one acid, and
- (iii) a mixture of solvents comprising (a) an N
  alkylpyrrolidone derivative and (b) a cosolvent of the general formula R<sub>1</sub>-O-(EO)<sub>m</sub>
  (PO)<sub>n</sub>-R<sub>2</sub>, wherein R<sub>1</sub> and R<sub>2</sub> are independently

  C<sub>2-6</sub> alkyl or hydrogen, but not both hydrogen,
  m and n are independently 0-5, EO represents

  an ethyleneoxy group and PO represents a
  propyleneoxy group.
- A composition according to claim 1, in which the surfactant is an anionic surfactant selected from alkyl sulphates, alkyl sulphonates, alkyl aryl sulphonates, alkyl alkoxylated sulphates, or mixtures thereof.
- 3. A composition according to claim 1 or 2, in which the acid is a carboxylic acid selected from caprylic acid, propionic acid, azelaic acid, caproic acid, hydroxybenzoic acid, salicylic acid, malic acid, maleic acid, fumaric acid, succinic acid, glutaric acid, adipic acid, tartaric acid and mixtures thereof.

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- 4. A composition according to any of claims 1 to 3, in which the N-alkyl pyrrolidone derivative is selectd from N-(n-octyl)-2-pyrrolidone, N-(n-decyl)-2-pyrrolidone, N-(n-decyl)-2-pyrrolidone and N-(n-tetradecyl)-2-pyrrolidone.
- 5. A composition according to any of claims 1 to 4, in which the co-solvent is selected from di-ethylene glycol mono n-butyl ether, mono-ethylene glycol mono n-butyl ether, propylene glycol n-butyl ether, isopropanol, ethanol, butanol and mixtures thereof.
- 6. A composition according to any of claims 1 to 5, which has a pH between 3.0 and 6.0.

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- 7. A composition according to any of claims 1 to 6, which is in the form of a wipe.
- 8. A composition according to any of claims 1 to 6, which is packaged in a spray dispenser.
  - 9. A composition according to any of claims 1 to 6, which is packaged in a foam dispenser.
- 25 10. A composition according to any of claims 1 to 6, which is packaged in a toilet cleaning dispenser.

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PCT/EP 01/15402 A. CLASSIFICATION OF SUBJECT MATTER IPC 7 C11D3/00 C11D C11D3/20 C11D3/43 C11D3/28 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 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) EPO-Internal, WPI Data, PAJ C. DOCUMENTS CONSIDERED TO BE RELEVANT Category ° Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. X US 6 090 765 A (SHANNON J CHRISTOPHER ET 1 - 10AL) 18 July 2000 (2000-07-18)  $\operatorname{column}$  2, line 36 - $\operatorname{column}$  3, line 10 column 3, line 56 -column 4, line 15 column 4, line 32,33 column 4. line 60-65 claims 4-8; tables 4,5 X WO 88 00184 A (GAF CORP) 1 - 1014 January 1988 (1988-01-14) page 61, line 24 -page 62, line 15 page 73, line 9-24 Further documents are listed in the continuation of box C. Patent family members are listed in annex. Х Special categories of cited documents: "T" later document published after the international filing date or priority date and not in conflict with the application but 'A' document defining the general state of the art which is not considered to be of particular relevance cited to understand the principle or theory underlying the "E" earlier document but published on or after the international "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the "O" document referring to an oral disclosure, use, exhibition or other means document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. \*P\* document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 13/03/2002 5 March 2002 Authorized officer Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016 Pentek, E

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