A POWDER DETERGENT FOR DISHWASHING MACHINES, AND ITS MANUFACTURE

There is provided a detergent powder for dishwashing machines which consists generally of A) a component carrier, an alkalai donor, a corrosion inhibitor, an anticaking agent and pH-buffering substances; B) at least one amphoteric surfactant of a specific group; C) at least one non-ionic surfactant of a specific group; D) a combination of certain enzymes baked in granules; E) a bleaching agent which consists of percarbonate and/or persulphate; and F) a sequestering agent selected from phosphates, polyacrylates and carboxylates, the detergent having been buffered to provide a certain pH-value. There is also presented a method of manufacturing a dishwashing detergent in which the components A), D) and F) are mixed in a first stage, the components B) and C) are sprayed in a second stage and the component E) is mixed in a third stage.
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A POWDER DETERGENT FOR DISHWASHING MACHINES,
AND ITS MANUFACTURE

The present invention relates to a powder detergent for
dishwashing machines, and to a method for its manufac-
ture.

The majority of the dishwashing powders available on the
market at present are caustic (strongly alkaline) and
often contain environmentally harmful components, such
as, for instance, high concentrations of sequestering
agents, such as phosphates, nitrilotriacetic acid (NTA),
ethylendiaminetetraacetic acid (EDTA) and phosphonates
and not readily degraded surfactants, and bleaching
agents in the form of chlorine compounds. It is there-
fore an object of the present invention to provide a
detergent powder for dishwashing machines which is
equally as effective as conventional dishwashing deter-
gent powders but which is not as caustic as these latter
detergents and which will fulfill the very high demands
placed on environmental care, and in which the bleaching
agent present consists of a less environmentally harmful
compound, such as percarbonate or persulphate, instead
of the chlorine compounds used with conventional dish-
washing agents, such as chloroisocyanurate or perborate.

This object is fulfilled in accordance with the present
invention with a powder detergent for dishwashing
machines which is characterized in that it is comprised
substantially of

A) a component carrier, an alkali donor, a corrosion
inhibitor, an anticaking agent and pH-buffering sub-
stances;
B) at least one amphoteric surfactant which functions effectively in hard water, will not cause calcium carbonate to precipitate during the washing process, has a low foaming profile and is selected from the group tallow amphi-polycarboxy glycinate, N-(2-carboxy ethyl) -N-octyl-ß-alanine, mixed C8-amphocarboxylates and caprylo-amphocarboxy propionate;  
C) at least one nonionic surfactant which will function effectively in hard water, will not cause calcium carbonate to precipitate in the washing process, has a low-foaming profile and is selected from the group fatty- and oxoalcohol ethoxylates having a carbon chain of 8-14, preferably 9-11, carbon atoms in the alkanol part and a degree of ethoxylation of 3-9, preferably 4-7, mole ethylene oxide units and block polymers, preferably polyether polyols;  
D) a combination of enzymes baked in granules and selected from the group proteases, amylases and lipases occurrent in the detergent industry;  
E) a bleaching agent which consists of percarbonate and/or persulphate; and  
F) a sequestering agent selected from phosphates, polyacrylates and carboxylates, said detergent having been buffered to provide a pH-value of 8.0-11.0, preferably 8.5-10.5, for a 0.3% ready-for-use solution of the detergent in water.

The invention also relates to a method for producing the inventive powdered detergent, said method being characterized by

I) mixing the aforedefined components A), D) and F) in a first stage;  
II) mixing the solid particles obtained from stage I) with liquid substances in droplet form with the aid of an agglomeration technique, such as to coat said
particles with a surface layer of said liquid substant-
es, said substances consisting of the components B) and 
C) defined above; and

5 III) mixing the component E) in a third stage so as to 
form a dishwashing powder, said pH-buffering substances 
having been selected and supplied in a quantity such 
that a 0.3% ready-for-use solution of the detergent in 
water will have a pH-value in the range of 8.0-11.0, 
preferably 8.5-10.5, wherein when said component E) 
10 consists of or includes percarbonate, a sequestering 
agent capable of binding heavy metal ions, particularly 
Fe\(^{2+}\) and Fe\(^{3+}\), and an antioxidation agent have been 
dissolved in at least one of the liquid substances mixed 
in stage II).

15 The component A), i.e. component carrier, alkali donor, 
corrosion inhibitor, anticaking agent and pH-buffering 
substances, may be included in the inventive dishwashing 
detergent in the form of substances selected from sodium 
carbonate (soda), sodium hydrogen carbonate, sodium 
20 sesquicarbonate, sodium sulphate, silicates and disili-
cates.

In this connection, sodium carbonate is preferably used 
in combination with sodium hydrogen carbonate so as to 
25 obtain the desired pH-value. The sodium carbonate is 
normally present in a concentration in the range of 25- 
50, preferably 30-40 percent by weight. (All percentag-
es mentioned here and in the following are related to 
30 the total weight of the detergent.) The concentration 
of sodium hydrogen carbonate present is normally in the 
range of 5-30, preferably 5-15 percent by weight.

35 Sodium sesquicarbonate can be used as an alternative to 
the combination of sodium carbonate/sodium hydrogen 
carbonate, the sodium sesquicarbonate normally being
included in a concentration of 30-50, preferably 35-45 percent by weight.

Sodium sulphate is generally present in the inventive detergent in a concentration of 0-20, preferably 0-10 percent by weight.

The silicate used is preferably sodium silicate, which is normally used in a concentration of 0-20, preferably 0-5 percent by weight.

The concentration of disilicate used is generally in the range of 0-40, preferably 5-20 percent by weight.

The amphoteric surfactant (component B) is normally present in the inventive detergent in a concentration of 1.0-10, preferably 2-6 percent by weight.

Examples of tallow ampho-polycarboxy glycinate used in the present context are those products retailed under the trade names Ampholak® 7TX and Beraid® 7557 by Berol Nobel, Stockholm, Sweden. An example of N-(2-carboxyethyl) -N-octyl-β-alanine is the product retailed under the trade name Ampholak® YJH-40 by Berol Nobel, Stockholm, Sweden. An example of mixed C8-ampho-carboxylates for inclusion in the inventive detergent is the product retailed under the trade name Miranol® JEM CONC by Miranol Chemical Company, Inc., South Brunswick (Dayton), NJ, U.S.A. An example of caprylo-amphocarboxy propionate is the product retailed under the trade name Miranol® J2M-SF CONC by Miranol Chemical Company, Inc., South Brunswick (Dayton), NJ, U.S.A.

Similar to the amphoteric surfactants which form component B), the nonionic surfactants used as component C) in the inventive dishwashing detergent are liquid at
room temperature. The nonionic surfactant is normally present in the inventive detergent in a concentration of 1-10, preferably 2-6 percent by weight.

The component D) consists in a combination of enzymes baked in granules and selected from the group proteases, amylases and lipases available within the detergent industry. The presence of these components enhances the cleaning effect of the detergent.

The best effect is obtained when there is chosen an enzyme combination which includes at least one representative of each type of enzyme. The total enzyme concentration in the inventive detergent is normally in the range of 1-5 percent by weight, wherein the concentration of individual enzymes may vary in dependence on their respective properties.

An example of proteases which can be used in this connection are those products which are retailed under the trade names Esperase® and Durazym® by Novo Nordisk A/S, Denmark. Examples of α-amylases and lipases which can be used in the inventive detergent are those products which are retailed under the respective trade names Termamyl and Lipolase by Novo Nordisk A/S, Denmark.

The component E) included in the inventive detergent may be a bleaching agent consisting of percarbonate, persulphate or a mixture of these two substances. The preferred bleaching agent is percarbonate, particularly sodium percarbonate.

The concentration of percarbonate in the inventive detergent is normally 3-20, preferably 5-15 percent by weight. When persulphate is used, it will not normally have a higher concentration than 10 percent by weight,
preferably not higher than 5 percent by weight.

The component F) included in the inventive detergent is a sequestering agent chosen from the group phosphates, polyacrylates and carboxylates.

The sequestering agent used in this connection is preferably one or more substances from the group citrates, tartrates and gluconates, particularly sodium citrate and/or a mixture of sodium tartrate and potassium tartrate. Citrate is normally present in a concentration within the range of 10-30, preferably 15-20 percent by weight, while the tartrate concentration is within the range of 2-15, preferably 2-5 percent by weight. When citrate and tartrate mixtures are used, the lower part of respective preferred concentration ranges are applied in order to avoid an excessively high combined concentration. When gluconate, and then particularly sodium gluconate, is used alone, the gluconate is normally present in the inventive detergent in a concentration within the range of 5-25, preferably 5-10 percent by weight, although this percentage may advantageously be beneath 5 percent by weight when the gluconate is present in combination with another sequestering agent, such as a citrate and/or a tartrate.

As an alternative to the aforesaid carboxylates, the component F) included in the inventive detergent may consist of phosphates, preferably sodium tripolyphosphate, and which are then present in the inventive detergent in a concentration which is normally within the range of 10-35, preferably 15-20 percent by weight.

Another alternative to carboxylates as component F) in the inventive detergent are polyacrylates which are then present in a concentration which is normally within the
range of 3-20, preferably 3-7 percent by weight.

When the component E) of the inventive detergent is percarbonate, the detergent is also given a small quantity of an antioxidation agent, so as to prevent degradation of the enzymes (component D) and the surfactants. The antioxidation agent is normally present in the detergent in a concentration within the range of 0.005-0.03, preferably 0.005-0.01 percent by weight. The antioxidation agent used in this case may be a \((\text{C}_1-\text{C}_3)\)-alkyl ester of 3,4,5-trihydroxybenzoic acid, preferably ethyl 3,4,5-trihydroxybenzoate, and/or ethyl 3,4,5-trihydroxyanisole.

Further, when percarbonate is used as component E), a sequestering agent other than the sequestering agents included in component F) and/or a substance of component F) or a corresponding acid can be added in a small quantity during the manufacture of the detergent, such as a sequestering agent based on the tetradsodium salt of ethylenediaminetetraacetic acid (\(\text{Na}_4\) EDTA) having a specific bonding affinity to \(\text{Fe}^{3+}\), this further sequestering agent normally being used in a concentration within the range of 0.05-0.5, preferably 0.08-0.15 percent by weight.

When using an antioxidation agent, the agent is mixed in stage II) dissolved in the component C). Also mixed in stage II) is the aforesaid sequestering agent which is not included in component F), or a minor part of the carboxylates used as component F), said sequestering agent being dissolved in the component B).

The inventive method is particularly advantageous when percarbonate is used as component E) in the inventive detergent. Percarbonates, such as sodium percarbonate,
have a relatively unstable structure, which causes problems with regard to the durability of the product when used as a dishwashing detergent. The degradation of the percarbonate is namely accelerated in the presence of other substances, such as heavy metals, readily oxidizable substances and moisture.

The inventive method is based on the known so-called agglomeration technique for the manufacture of laundering and dishwashing detergent powders.

In this agglomeration technique, solid powder particles are mixed with liquid substances in droplet form. The agglomeration technique enables powder detergents for dishwashing machines to be manufactured at high litre weights and high concentrations of active substances.

When practicing the inventive method, the detergent components are mixed in stages, so that those substances which have an destabilizing effect on percarbonates are first encapsulated so as to obtain a surface coating which counteracts degradation, and it is only thereafter that the requisite amount of percarbonate is added to the system.

Although a number of methods of stabilizing percarbonate during its manufacture are known to the art, none of these methods has resulted in a percarbonate of desired stability for use in laundering and dishwashing detergents. However, when practicing the inventive method, there is used advantageously a percarbonate which has itself been subjected to a stabilizing procedure with the aid of earlier known techniques during the manufacture of said percarbonate.
According to one modification of the inventive method in which disilicate and/or sodium carbonate are included in component A) and sodium percarbonate is used as or included in component B), the major part of the disilicate and/or the sodium carbonate is mixed in stage I) and the remainder is mixed together with the component E) in stage III).

The invention is described below in more detail with reference to a number of working examples, although it will be understood that the invention is not restricted to these examples.

EXAMPLES

General Procedure

A number of powder detergents for dishwashing machines were produced in an agglomerator of the type Forberg Mixer from Halvor Forberg A/S, Larvik, Norway.

In a first stage (stage I in the Table below), the solid components A), D) and F) intended for this stage were mixed in the aforesaid agglomerator, said components being finely divided in air. Liquid surfactants (the components B) and C) with substances optionally dissolved therein were then sprayed onto the fluidized or airborne mixture of solid particles in the form of fine droplets in a second stage (stage II in the Table), the amphoteric surfactant being preferably sprayed first and then the nonionic surfactant. The bleaching agent (component E)) and, when applicable, a part of the substances included in component A) of the inventive detergent were then mixed with the previously mixed components in a third stage (stage III in the Table).
The various examples are set forth in the following Table.

In the Table, the number of carbon atoms in the alkyl moiety of certain surfactants has been given in the form of "C" followed by the relevant number of carbon atoms, whereas the number of ethyleneoxide units has been given in the form "EO" and the relevant number of units.

Example 7 represents the best mode contemplated at present for carrying out the invention.
TABLE

Concentrations in Percent by Weight
Example No.
1 2 3 4 5 6 7

Stage I
Component A
1. Disilicate (Sikalon™ 2 D, Woellner-Werke, Germany 17 21 17 20 15 19
2. Sodium carbonate 30 32 32 32 32 24.85
3. Sodium hydrogen carbonate 10 8 8 8 8 40 30
4. Sodium sesquicarbonate 40 30
5. Sodium sulphate 40 30
6. Sodium metasilicate 40 30

Component D
7. Termamyl (α-amylase from Novo Nordisk A/S, 2.0 2.0 2.5 2.0 2.5 2.0 1.25
   Denmark)
8. Esperase® (protease from Novo Nordisk A/S, 1.0 1.0 1.0 1.0 1.0 1.0 1.0
   Denmark)
9. Durazym® (protease from Novo Nordisk A/S, 1.0 0.5 1.0 1.0 1.0 1.0 1.0
   Denmark)
10. Lipolase (lipase from Novo Nordisk A/S, 0.5 0.5 0.5 0.5 0.5 0.5 0.5
    Denmark)

Component F
11. Sodium nitrate 15 17.5 17.5 17.5 17.5 17.5 17.5

SUBSTITUTE SHEET
<table>
<thead>
<tr>
<th>Example No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td>12. Sodium-potassium tartrate</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>2.5</td>
<td>-</td>
<td>-</td>
<td>1.0</td>
</tr>
<tr>
<td>13. Sodium gluconate</td>
<td>3.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>7</td>
</tr>
<tr>
<td>14. Sodium tripolyphosphate</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>20</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>15. Polyacrylate (Antiprex A 100, CDM, Gothenburg, Sweden)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>5</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

**Stage II**

**Component B**

<p>| 16. Tallow amphopoly-carboxy glycinate (Ampholak® 7 TX, Berol Nobel, Sweden) | 3.5¹ | - | - | - | - | - |
| 18. Mixed C8-amphocarboxylates (Miranol® JEM CONC, Miranol Chem. Co., USA) | - | - | - | - | 4.0¹ | - |
| 19. Capryloamphocarboxypropionate (Miranol® J2M-SF CONC, Miranol Chem. Co., USA) | 3.5¹ | - | - | - | 1.5 | - | - |</p>
<table>
<thead>
<tr>
<th>Example No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td>20. Oxoalcohol ethoxylate C9-11, EO 4</td>
<td>3.5 2</td>
<td>3.5 2</td>
<td>3.5 2</td>
<td>4.5 2</td>
<td>-</td>
<td>-</td>
<td>2.0 2</td>
</tr>
<tr>
<td>(Berol 91-4, Berol Nobel, Sweden)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>21. Polyetherpolyol (Dowfax 20 A 42, Dow Chemicals, USA)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>4.0 2</td>
<td>4.0 2</td>
<td>-</td>
</tr>
</tbody>
</table>

**Sequestering Agent**

| 22. Na 4 EDTA (Trilon BVT, BASF, Germany) | - | - | - | - | - | + | - |
| 23. Phosphonate | - | - | - | - | + | - | - |
| 24. Sodium Citrate or Citric Acid | + | + | + | + | - | - | - |
| 25. Sodium Potassium Tartrate or Tartaric Acid | + | + | + | + | - | - | + |

**Antioxidation Agent**

| 26. Ethyl 3,4,5-tri-hydroxibenzoate (Progallin A, Nipa Laboratories, England) | + | + | + | + | - | - | - |
| 27. Butyl hydroxyanisole | - | - | - | - | - | + | - |

**Stage III**

**Component E**

| 28. Sodium Percarbonate (Degussa, Germany) | 8.0 | 7.5 | 7.5 | 5.0 | 8.0 | 10.0 | 15 |

**TABLE (continued)**

Concentrations in Percent by Weight

**SUBSTITUTE SHEET**
<table>
<thead>
<tr>
<th>Component</th>
<th>Example No.</th>
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</thead>
<tbody>
<tr>
<td>Sodium Persulphate</td>
<td>1 2 3 4 5 6 7</td>
</tr>
<tr>
<td></td>
<td>1.0 2.5 1.5</td>
</tr>
<tr>
<td>Component A</td>
<td>4.0 5.0 5.0 3.0</td>
</tr>
<tr>
<td>Disilicate</td>
<td></td>
</tr>
</tbody>
</table>
Comments on the Examples

Example 1

5  (1) The components 24 and 25 were dissolved in the component 19 and each contributed 0.1 percent by weight to the given concentration.

(2) The component 26 was dissolved in the component 20 in an amount corresponding to 0.01 percent by weight calculated on the weight of the detergent.

Example 2

15 (1) The components 24 and 25 were dissolved in the component 16 and each contributed 0.1 percent by weight to the given concentration.

(2) The component 26 was dissolved in the component 20 in an amount corresponding to 0.01 percent by weight calculated on the weight of the detergent.

Examples 3 and 4

1) The components 24 and 25 were dissolved in the component 17 and each contributed 0.1 percent by weight to the given concentration.

25 (2) The component 26 was dissolved in the component 20 in an amount corresponding to 0.01 percent by weight of the weight of the detergent.

Example 5

30 (1) The component 23 was dissolved in the component 17 and contributed 0.15 percent by weight to the given concentration.

(2) The component 26 was dissolved in the component 21 in an amount corresponding to 0.01 percent by weight calculated on the weight of the detergent.

SUBSTITUTE SHEET
Example 6

(1) The component 22 was dissolved in the component 18 in an amount corresponding to 0.001 percent by weight calculated on the weight of the detergent.

(2) The component 27 was dissolved in the component 21 in an amount corresponding to 0.01 percent by weight calculated on the weight of the detergent.

Example 7

(1) The component 25 was dissolved in the component 17 and contributed 0.15 percent by weight to the given concentration.

(2) The component 26 was dissolved in the component 20 in an amount corresponding to 0.01 percent by weight of the weight of the detergent.

+ denotes the presence of the component concerned.

- denotes the absence of the component concerned.
CLAIMS

1. A powder detergent for dishwashing machines, characterized in that the detergent mainly consists of

A) a component carrier, an alkali donor, a corrosion inhibitor, an anticaking agent and pH-buffering substances;
B) at least one amphoteric surfactant which functions effectively in hard water, will not cause calcium carbonate to precipitate during the washing process, has a low foaming profile and is selected from the group tallow amphoteric polycarboxylate glycinate, N-(N-carboxy ethyl) -N-octyl-β-alanine, mixed C8-amphocarboxylates and caprylo-amphocarboxy propionate;
C) at least one nonionic surfactant which functions effectively in hard water, will not cause calcium carbonate to precipitate in the washing process, has a low-foaming profile and is selected from the group fatty- and oxoalcohol ethoxylates having a carbon chain of 8-14, preferably 9-11, carbon atoms in the alkanol part and a degree of ethoxylation of 3-9, preferably 4-7, mole ethylene oxide units and block polymers, preferably polyether polyols;
D) a combination of enzymes baked in granules and selected from the group proteases, amylases and lipases occurring in the detergent industry;
E) a bleaching agent which consists of percarbonate and/or persulphate; and
F) a sequestering agent selected from phosphates, polyacrylates and carboxylates, said detergent having been buffered to provide a pH-value of 8.0-11.0, preferably 8.5-10.5, for a 0.3% ready-for-use solution of the detergent in water.
2. A detergent according to Claim 1, characterized in that the sequestering agent consists of one or more substances from the group citrates, tartrates and gluconates, preferably sodium citrate and/or a mixture of sodium tartrate and potassium tartrate.

3. A detergent according to Claim 1 or 2, characterized in that the component carrier, alkali donor, corrosion inhibitor, anticaking agent and pH-buffering substances are substances chosen from the group sodium carbonate, sodium hydrogen carbonate, sodium sesquicarbonate, sodium sulphate, silicates and disilicates.

4. A detergent according to any one of Claims 1-3, in which component E) is sodium percarbonate, characterized in that the detergent also includes a minor quantity of an antioxidation agent.

5. A detergent according to Claim 4, characterized in that the antioxidation agent is a \((C_1-C_3)\)-alkylester of 3,4,5-trihydroxybenzoic acid, preferably ethyl 3,4,5-trihydroxybenzoate, and/or ethyl 3,4,5-trihydroxyanisole.

6. A method for the manufacture of a detergent for dishwashing machines in accordance with one or more of Claims 1-5, characterized by
   I) mixing the components A), D) and F) defined in Claim 1 in a first stage;
   II) mixing the solid particles obtained from stage I) with liquid substances in droplet form with the aid of an agglomeration technique, such as to coat said particles with a surface layer of said liquid substances, said substances consisting of the components B) and C)
defined in Claim 1; and

III) mixing the component E) in a third stage so as to
form a dishwashing powder, said pH-buffering substances
having been selected and supplied in a quantity such
that a 0.3% ready-for-use solution of the detergent in
water will have a pH-value in the range of 8.0-11.0,
preferably 8.5-10.5, wherein, when said component E)
consists of or includes percarbonate, a sequestering
agent capable of binding heavy metal ions, particularly
Fe$^{2+}$ and Fe$^{3+}$, and an antioxidation agent have been
dissolved in at least one of the liquid substances mixed
in stage II).

7. A modification of the method according to Claim 6,
in which component A) includes disilicate and/or sodium
carbonate and component E) is or includes sodium percar-
bonate, characterized by mixing the major
part of the disilicate and/or the sodium carbonate in
stage I) and the remainder together with the component
E) in stage III).
INTERNATIONAL SEARCH REPORT

International Application No PCT/SE 91/00894

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all)\(^6\)

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: C 11 D 1/94, 3/39

II. FIELDS SEARCHED

<table>
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<td>C 11 D</td>
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Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched\(^6\)

SE, DK, FI, NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT\(^9\)

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of Document,(^11) with indication, where appropriate, of the relevant passages(^12)</th>
<th>Relevant to Claim No.(^13)</th>
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<tbody>
<tr>
<td>A</td>
<td>Dialog Information Services, file 351, World Patent Index 81-92, Dialog accession no. 007219325, (LION-) LION HAIJIN KK, &quot;Bleaching agent comspns. cont. peroxide(s) and ionic polymers - based on maleic acid homo- and copolymers and polystyrene sulphonate, for dishwashing and laundering detergents&quot;, JP, 62141099, A, 870624, 8731</td>
<td>1-7</td>
</tr>
<tr>
<td>A</td>
<td>CH, A5, 673033 (COSMINA AG) 31 January 1990, see the whole document</td>
<td>1-7</td>
</tr>
</tbody>
</table>

* Special categories of cited documents: \(^10\)
  
  *A* document defining the general state of the art which is not considered to be of particular relevance
  
  *E* earlier document but published on or after the international filing date
  
  *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)
  
  *O* document referring to an oral disclosure, use, exhibition or other means
  
  *P* document published prior to the international filing date but later than the priority date claimed

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**X** document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

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IV. CERTIFICATION

Date of the Actual Completion of the International Search
17th September 1992

Date of Mailing of this International Search Report
23 - 09- 1992

International Searching Authority

SWEDISH PATENT OFFICE

Signature of Authorized Officer

Dagmar Järven mant
This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The Swedish Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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