

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 0 837 124 A2

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:
22.04.1998 Bulletin 1998/17

(51) Int Cl.⁶: **C11D 3/42, C11D 1/72,
C11D 1/722, C11D 3/20**

(21) Application number: **97810741.5**

(22) Date of filing: **06.10.1997**

(84) Designated Contracting States:
**AT BE CH DE DK ES FI FR GB GR IE IT LI LU MC
NL PT SE**

(72) Inventors:
• **Kuratli, Rolf**
4058 Basel (CH)
• **Schmidlin, Anita**
4334 Sisseln (CH)

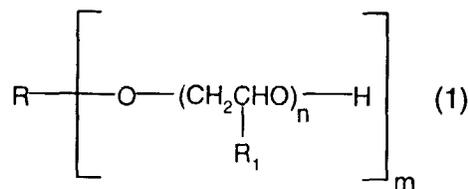
(30) Priority: **15.10.1996 GB 9621421**

(71) Applicant: **Ciba Specialty Chemicals Holding Inc.**
4057 Basel (CH)

(54) Fluorescent whitening agent formulation

(57) The present invention provides a liquid fluorescent whitening agent formulation which is a solution having a viscosity of 50 to 5000 mPas, preferably 100 to 3500 mPas, especially 100 to 1000 mPas and which comprises:

- 10-25%, preferably 15-20% by weight of an anionic fluorescent whitening agent, based on the total weight of the formulation;
- 15-35%, preferably 20-30% by weight of a polar solvent, based on the total weight of the formulation; and
- 45-75%, preferably 50-70% by weight, based on the total weight of the formulation, of a nonionic surfactant having the formula:



in which m is 1, 2, 3 or 4 and, when m is 1, R is a C_8 - C_{18} -alkyl residue or a C_8 - C_{18} -alkylcarboxy residue; when m is 2, R is a C_2 - C_4 alkylene glycol residue from which the two hydroxy groups have been removed; when m is 3, R is the residue of trimethylolpropane from which the three hydroxy groups have been removed; and when m is 4, R is the residue of pentaerythritol residue from which the four hydroxy groups have been removed; R_1 is hydrogen, methyl or ethyl; and n is a number ranging from 1 to 40.

EP 0 837 124 A2

Description

The present invention relates to a fluorescent whitening agent formulation and, in particular, to a liquid fluorescent whitening agent formulation comprising a solution of a fluorescent whitening agent in a specific type of nonionic surfactant.

Traditionally, detergent manufacturers have produced solid detergent products by spray drying an aqueous slurry containing the various components of the detergent composition, including a fluorescent whitening agent. For this purpose, the detergent manufacturers prefer to use the fluorescent whitening agent component in the form of a liquid formulation, in order to facilitate the incorporation of the fluorescent whitening agent into the slurry to be spray-dried. As a consequence, fluorescent whitening agents are often supplied to the detergent manufacturers in the form of an aqueous slurry. Such aqueous slurries of fluorescent whitening agent, however, usually contain large amounts of water, which must be removed during subsequent spray drying, and are often beset by problems, such as the formation of clumps during spray-drying, as well as instability to sedimentation on storage.

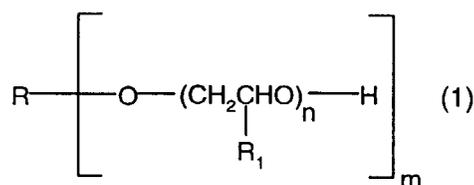
It has recently been found that detergent manufacturers can considerably simplify the detergent production process by applying a liquid formulation of a fluorescent whitening agent, at the end of the detergent production process, to a previously spray-dried granular-form mixture of the remaining detergent components. For this application, there is a need to provide the fluorescent whitening agent in a liquid form in which the water content is reduced. Moreover, the detergent manufacturers have expressed a wish that the fluorescent whitening agent be provided in the form of a homogeneous solution, namely in a form which avoids the problems associated with traditional aqueous slurries. A still further requirement is that the viscosity of said homogeneous solution be sufficiently low to permit conventional pumping of the fluorescent whitening agent formulation.

Also known are liquid formulations of fluorescent whitening agents which use organic solvents and are substantially anhydrous. These liquid formulations, however, are usually highly viscous and need to be incorporated into the detergent powder by means of special mixing techniques.

Surprisingly, it has now been found that anionic fluorescent whitening agents are soluble in a mixture comprising a predominant amount of a certain type of nonionic surfactant and a lesser amount of water, to provide stable, clear solutions of low viscosity.

Accordingly, the present invention provides a liquid fluorescent whitening agent formulation which is a solution having a viscosity of 50 to 5000 mPas, preferably 100 to 3500 mPas, especially 100 to 1000 mPas and which comprises:

- a) 10-25%, preferably 15-20% by weight of an anionic fluorescent whitening agent, based on the total weight of the formulation;
- b) 15-35%, preferably 20-30% by weight of a polar solvent, based on the total weight of the formulation; and
- c) 45-75%, preferably 50-70% by weight, based on the total weight of the formulation, of a nonionic surfactant having the formula:



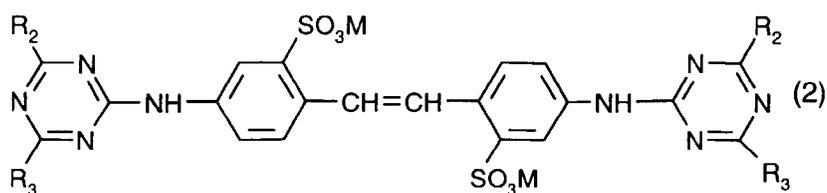
in which m is 1, 2, 3 or 4 and, when m is 1, R is a C₈-C₁₈-alkyl residue or a C₈-C₁₈-alkylcarboxy residue; when m is 2, R is a C₂-C₄alkylene glycol residue from which the two hydroxy groups have been removed; when m is 3, R is the residue of trimethylolpropane from which the three hydroxy groups have been removed; and when m is 4, R is the residue of pentaerythritol residue from which the four hydroxy groups have been removed; R₁ is hydrogen, methyl or ethyl; and n is a number ranging from 1 to 40.

Preferably, m is 1, 3 or 4 and, when m is 1, R is a C₈-C₁₈-alkyl residue or a C₈-C₁₈-alkylcarboxy residue; when m is 3, R is the residue of trimethylolpropane from which the three hydroxy groups have been removed; and when m is 4, R is the residue of pentaerythritol residue from which the four hydroxy groups have been removed; R₁ is hydrogen, methyl or ethyl; and n is a number ranging from 1 to 40.

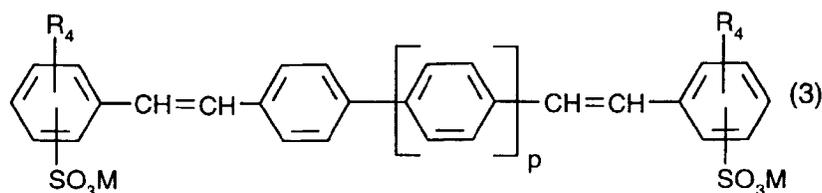
The viscosity of the liquid fluorescent whitening agent formulation according to the present invention is determined at a shear force of 100/s and at a temperature of 25°C. ± 1°C.

Preferred anionic fluorescent whitening agents for use in the present invention are those having either of the

formulae:



or

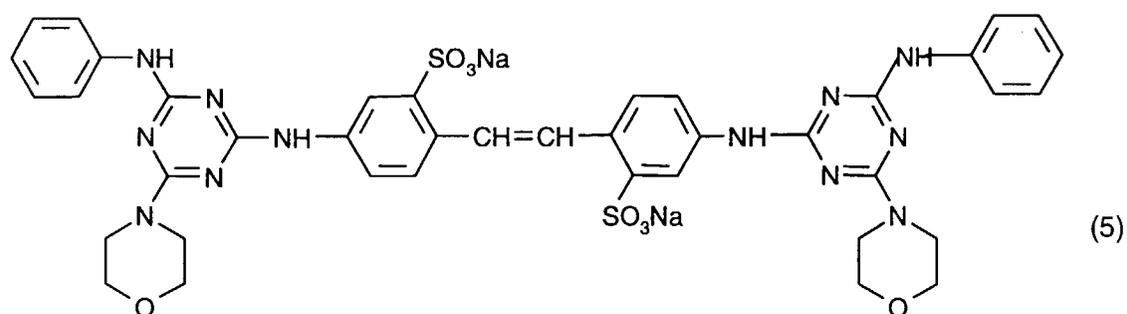
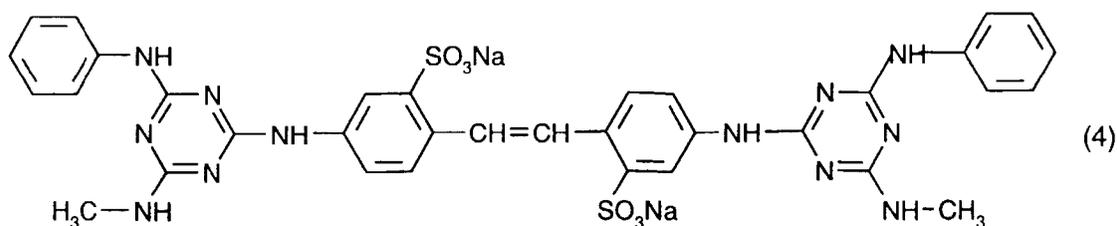


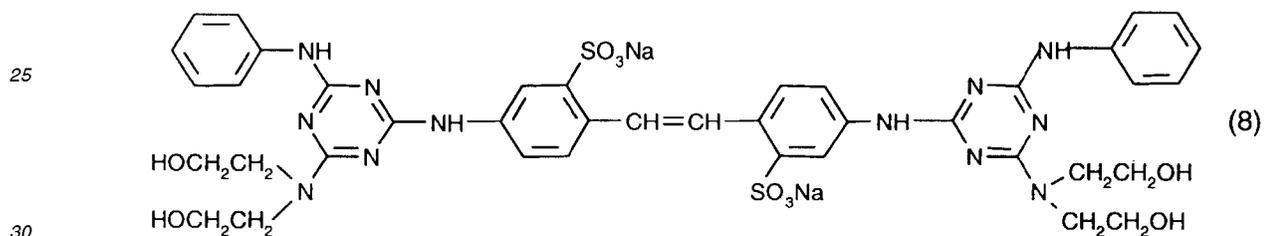
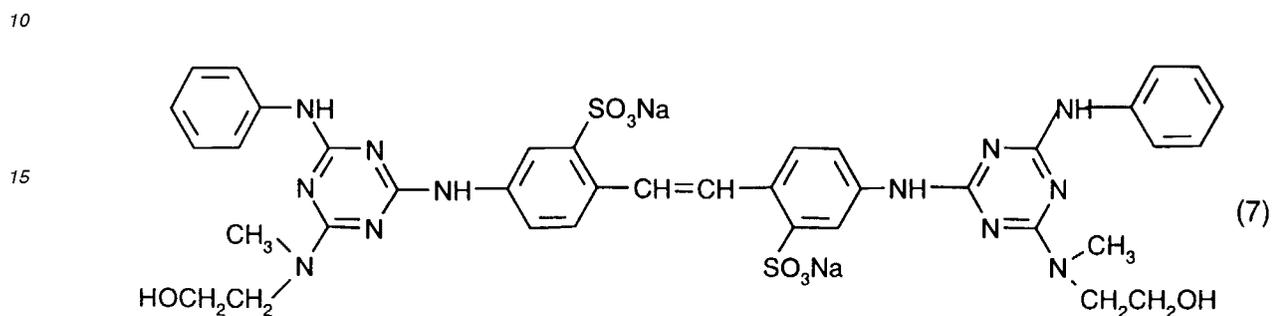
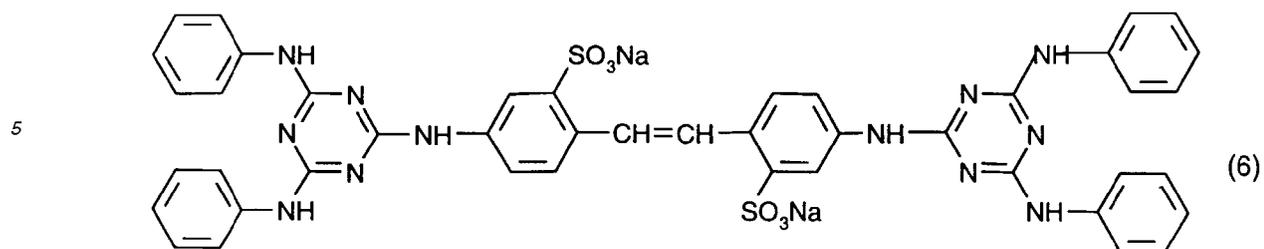
25 in which R_2 and R_3 , independently, are OH, NH_2 , O- C_1 - C_4 -alkyl, O-aryl, NH- C_1 - C_4 -alkyl, $\text{N}(\text{C}_1$ - C_4 -alkyl) $_2$, $\text{N}(\text{C}_1$ - C_4 -alkyl)(C_1 - C_4 -hydroxyalkyl), $\text{N}(\text{C}_1$ - C_4 -hydroxyalkyl) $_2$, NH-aryl, morpholino, S- C_1 - C_4 -alkyl(aryl), Cl or OH; R_4 is H, SO_3M , O- C_1 - C_4 -alkyl, CN, Cl, $\text{COO-}\text{C}_1$ - C_4 -alkyl, or $\text{CON}(\text{C}_1$ - C_4 -alkyl) $_2$; M is H, Li, Na, K, Ca, Mg, ammonium, mono-, di-, tri- or tetra- C_1 - C_4 -alkylammonium, mono-, di- or tri- C_1 - C_4 -hydroxyalkylammonium or ammonium that is di- or tri-substituted with a mixture of C_1 - C_4 -alkyl and C_1 - C_4 -hydroxyalkyl groups; and p is 0 or 1.

30 In the compounds of formulae (2) or (3), C_1 - C_4 -alkyl groups are, e.g., methyl, ethyl, n-propyl, isopropyl and n-butyl, especially methyl. Aryl groups are, e.g., naphthyl or, especially, phenyl.

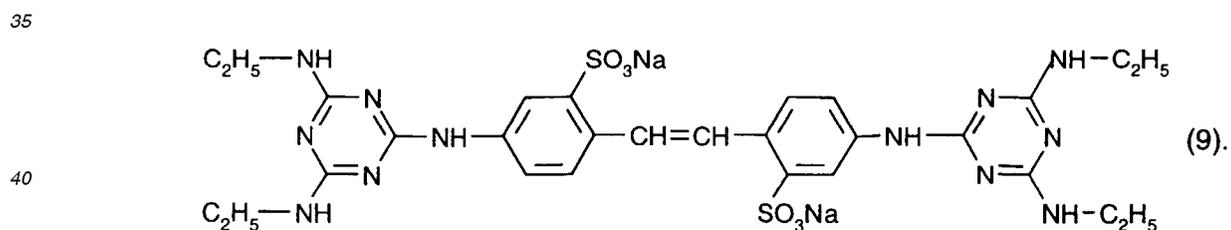
35 Preferred compounds of formula (2) are those in which R_2 and R_3 , independently, are methoxy, phenoxy, NH_2 , NH-methyl, $\text{N}(\text{methyl})_2$, $\text{N}(\text{methyl})(\text{hydroxyethyl})$, NH-ethyl, $\text{N}(\text{hydroxyethyl})_2$, NH-phenyl, morpholino, S-methyl(phenyl), Cl or OH.

Specific examples of preferred compounds of formula (2) are those having one of the formulae:



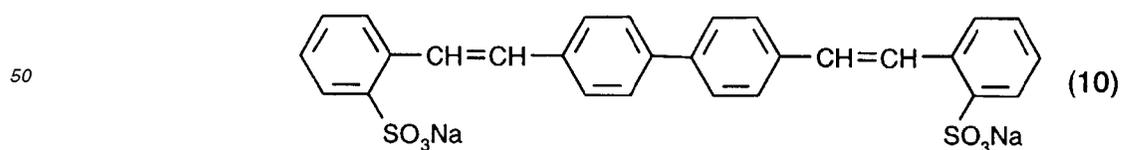


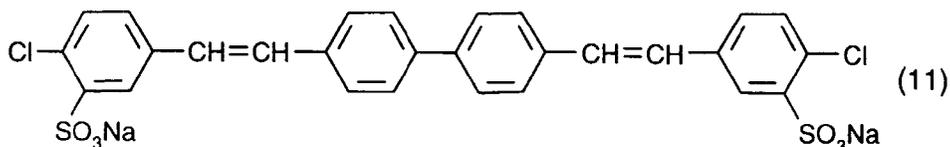
and



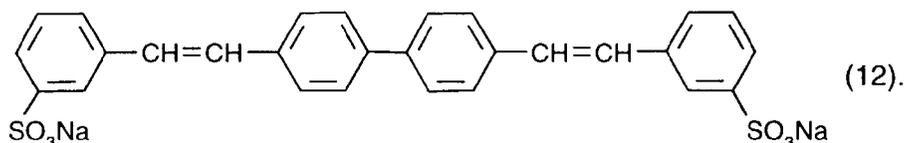
45 Preferred compounds of formula (3) are those in which R_4 is H or Cl and p is 1.

Specific preferred examples of compounds of formula (3) are those having one of the formulae:





10 and



20 Especially preferred is the compound of formula (10) or a crystal modification thereof, as described in EP-A-0 577 557.

If desired, a mixture of two or more anionic fluorescent whitening agents may be used as component a) of the formulation according to the present invention.

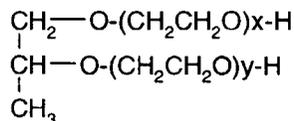
25 The polar solvent, component b) of the formulation according to the present invention, is preferably water. If desired, however, water may be used together with one or more polar co-solvents such as an alkylene glycol, e.g. ethylene glycol or 1,2-propylene glycol, or a polyalkylene glycol, especially polyethylene glycol or polypropylene glycol. The amount of any co-solvent preferably ranges from 0-15% by weight, based on the total weight of the formulation.

30 The radical R in the compounds of formula (1), when m is 1, is derived from a C₈-C₁₈-alkyl monoalcohol. Examples of such monoalcohols include naturally-occurring monoalcohols such as lauryl alcohol, myristyl alcohol, cetyl alcohol and stearyl alcohol, as well as synthetic alcohols such as 2-ethylhexanol, 1,1,3,3-tetramethylbutanol, octan-2-ol, isononyl alcohol, trimethylhexanol, trimethylnonyl alcohol, n-decanol, C₉-C₁₃-oxoalcohols, tridecyl alcohol, isodecyl alcohol or C₈-C₁₈-linear primary alcohols. Such C₈-C₁₈-linear primary alcohols are commercially available under the trade name Alfols, typical examples being Alfol (8-10), Alfol (9-11), Alfol (10-14), Alfol (12-13) and Alfol (16-18). The name "Alfol" is a registered trade mark.

35 Preferred examples of compounds of formula (1) in which m is 1 include, e.g., polyadducts of 3-20 moles of ethylene oxide with 1 mole of a C₈-C₁₈-alkyl monoalcohol. Especially interesting are polyadducts of 3-20 moles of ethylene oxide with 1 mole of a C₁₁-C₁₃-oxoalcohol and polyadducts of 8-20 moles of ethylene oxide with 1 mole of a C₁₂-C₁₄-fatty alcohol.

40 The radical R in the compounds of formula (1), when m is 2, is derived from a C₂-C₄alkylene glycol residue from which the two hydroxy groups have been removed. Examples of such C₂-C₄alkylene glycols include ethylene glycol, n-butylene glycol and, especially propylene glycol.

Preferred examples of compounds of formula (1) in which m is 2 include, e.g., polyadducts of 2-20 moles of ethylene oxide with 1 mole of a C₂-C₄alkylene glycols, preferably polyethylene glycol ethers of propylene glycol, in particular those having the formula:



in which x and y are each an integer within the range of from 1 to 10, preferably within the range of from 1 to 5, the sum of x and y preferably being about 10.

The alkyl radical R in the compounds of formula (1), when m is 3, is derived from trimethylolpropane.

55 Preferred examples of compounds of formula (1) in which m is 3 include polyadducts of 3-20 moles of ethylene oxide with 1 mole of trimethylolpropane. Especially interesting are polyadducts of 7-20 moles of ethylene oxide with 1 mole of trimethylolpropane.

The alkyl radical R in the compounds of formula (1), when m is 4, is derived from pentaerythritol.

Preferred examples of compounds of formula (1) in which m is 4 include polyadducts of 4-20 moles of ethylene oxide with 1 mole of pentaerythritol. Especially interesting are polyadducts of 5-20 moles of ethylene oxide with 1 mole of pentaerythritol.

Mixtures of two or more of the compounds of formula (1) may be used as component c) of the formulation according to the present invention.

The compounds of formula (2) to (12) are known and may be obtained by known methods.

Optional auxiliaries which may be present in the formulation of the present invention include stabilisers which are effective in adjusting the flow properties of the formulation, anti-foam agents, alkaline agents, fabric softeners, anti-redeposition agents, antioxidants, auxiliary builders such as polyacrylic acid and fragrances.

Examples of such stabilisers include, e.g., kaolin, an Mg/Al silicate, especially bentonite, montmorillonite, a zeolite or a highly dispersed silicic acid.

The formulation of the present invention may be produced by mixing fluorescent whitening agent, solvent and the compound of formula (1) together with any optional auxiliaries, and homogenising the mixture so obtained, preferably at an elevated temperature, e.g. at 40-100°C. Mixing is conveniently effected by a suitable stirring device.

The resulting formulation is normally a clear and stable solution. On occasion, however, it may be necessary to filter the formulation in order to remove minor amounts of insoluble components.

The formulation of the present invention is particularly suitable for incorporation into a dry detergent composition, conveniently by adding the required amount of the formulation of the present invention to a dry detergent composition and then homogenising the mixture so obtained. The formulation of the present invention may also be used, however, for the production of liquid detergents by adding the required amount of the formulation of the present invention to a liquid detergent composition and then homogenising the mixture so obtained.

The following Examples further illustrate the present invention. Parts and percentages shown therein are by weight unless otherwise stated. The viscosity data quoted in the Examples are expressed in mPas and are determined at a shear force of 100/s and at a temperature of 25°C. ± 1°C.

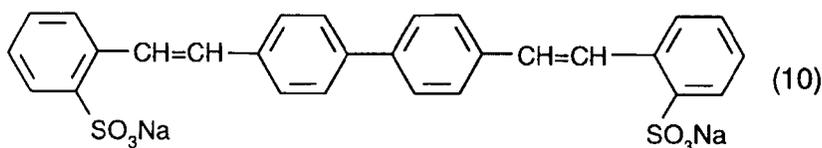
Example 1

The following materials are charged into a 1 litre flask:

100g. of deionised water;

600g. of an adduct of 1 part of a C₁₃-oxoalcohol with 9 parts of ethylene oxide; and

300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula:



The suspension so obtained is heated, with stirring, to an internal temperature of 70-80°C. After 4-5 hours, an almost clear solution is obtained which contains less than 1% by weight of undissolved components. The solution is cooled to 40-50°C. and filtered. A clear yellow solution is obtained having an active substance content of 15% by weight, a water content of 25% by weight and a viscosity of 462 mPas.

Examples 2 to 7

Using the procedure described in Example 1, further solution formulations of the compound of formula (10) are prepared by varying the amounts of the respective components and/or by varying the number of parts of ethylene oxide in the condensation product. The results are set out in the following Table.

EP 0 837 124 A2

Table

Example	cpd. (10) presscake % by wt.	water % by wt.	parts of EO in adduct	adduct % by weight	Appearance of product at 25°C	Viscosity mPas
2	30	10	8	60	slightly opalescent	397
3	30	15	9	55	slightly opalescent	505
4	30	0	10	70	clear	1060
5	30	10	10	60	clear	575
6	40	0	15	60	clear	1880
7	40	0	20	60	clear	3263

Example 8

The following materials are charged into a 1 litre flask:

600g. of an adduct of 1 part of a C₁₃-oxoalcohol with 10 parts of ethylene oxide;
 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10); and:
 100g. of 1,2-propyleneglycol.

Using the procedure described in Example 1, a liquid formulation is obtained which is a clear solution at 25°C. and has a viscosity of 702 mPas.

Example 9

The following materials are charged into a 1 litre flask:

100g. of deionised water;
 600g. of an adduct of 1 part of a C₁₁-oxoalcohol with 11 parts of ethylene oxide; and
 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10).

Using the procedure described in Example 1, a liquid formulation is obtained which is a clear solution at 25°C. and has a viscosity of 397 mPas.

Example 10

The following materials are charged into a 1 litre flask:

100g. of deionised water;
 420g. of an adduct of 1 part of a C₁₃-oxoalcohol with 8 parts of ethylene oxide;
 180g. of an adduct of 1 part of a C₁₃-oxoalcohol with 15 parts of ethylene oxide; and
 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10).

Using the procedure described in Example 1, a liquid formulation is obtained which is a clear solution at 25°C. and has a viscosity of 434 mPas.

Example 11

The following materials are charged into a 1 litre flask:

150g. of deionised water;

EP 0 837 124 A2

385g. of an adduct of 1 part of a C₁₃-oxoalcohol with 8 parts of ethylene oxide;
165g. of an adduct of 1 part of a C₁₃-oxoalcohol with 15 parts of ethylene oxide; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

5 Using the procedure described in Example 1, a liquid formulation is obtained which is a clear solution at 25°C. and has a viscosity of 550 mPas.

Example 12

10 The following materials are charged into a 1 litre flask:

50g. of deionised water;
455g. of an adduct of 1 part of a C₁₃-oxoalcohol with 8 parts of ethylene oxide;
15 195g. of an adduct of 1 part of a C₁₃-oxoalcohol with 15 parts of ethylene oxide; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

20 Using the procedure described in Example 1, a liquid formulation is obtained which is a clear solution at 25°C. and has a viscosity of 540 mPas.

Examples 13 to 16

25 The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 15 parts of ethylene oxide; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

30 Using the procedure described in Example 1, a liquid formulation (Example 13) is obtained which is a clear solution at 25°C. and has a viscosity of 1600 mPas.

Similar good results are obtained when the adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 15 parts of ethylene oxide is replaced by an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 20 parts of ethylene oxide (Example 14; viscosity 3261 mPas); or by an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 8 parts of ethylene oxide (Example 15; viscosity 829 mPas); or when no deionised water is used and the amount of the moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10) is increased to 400g (Example 16; viscosity 2417 mPas).

Examples 17 and 18

The following materials are charged into a 1 litre flask:

100g. of deionised water;
45 420g. of an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 8 parts of ethylene oxide;
180g. of an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 15 parts of ethylene oxide; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

50 Using the procedure described in Example 1, a liquid formulation (Example 17) is obtained which, after filtration, is a clear solution at 25°C. and has a viscosity of 732 mPas.

Similar good results are obtained when the amount of the adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 8 parts of ethylene oxide is reduced to 300g. and the amount of the adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 15 parts of ethylene oxide is increased to 300g (Example 18; viscosity 893 mPas).

Examples 19 and 20

The following materials are charged into a 1 litre flask:

EP 0 837 124 A2

150g. of deionised water;
385g. of an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 8 parts of ethylene oxide;
165g. of an adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 15 parts of ethylene oxide; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
5 having the formula (10).

Using the procedure described in Example 1, a liquid formulation (Example 19; viscosity 807 mPas) is obtained which, after filtration, is a clear solution at 25°C.

10 Similar good results are obtained when the amount of the adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 8 parts of ethylene oxide is reduced to 275g. and the amount of the adduct of 1 part of a C₁₂-C₁₄-fatty alcohol with 15 parts of ethylene oxide is increased to 275g (Example 20; viscosity 913 mPas).

Examples 21 to 23

15 The following materials are charged into a 1 litre flask:

600g. of an adduct of 1 part of pentaerythritol with 5 parts of ethylene oxide; and
400g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
20 having the formula (10).

Using the procedure described in Example 1, a liquid formulation (Example 21; viscosity less than 500 mPas) is obtained which is a clear solution at 25°C.

25 Similar good results are obtained when the adduct of 1 part of pentaerythritol with 5 parts of ethylene oxide is replaced with the adduct of 1 part of trimethylolpropane with 7 parts of ethylene oxide (Example 22; viscosity 446 mPas); or with the adduct of 1 part of trimethylolpropane with 20 parts of ethylene oxide (Example 23; viscosity 558 mPas).

Examples 24 to 26

30 The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of pentaerythritol with 5 parts of ethylene oxide; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
35 having the formula (10).

Using the procedure described in Example 1, a liquid formulation (Example 24; viscosity less than 500 mPas) is obtained which is a clear solution at 25°C.

40 Similar good results are obtained when the adduct of 1 part of pentaerythritol with 5 parts of ethylene oxide is replaced with the adduct of 1 part of trimethylolpropane with 7 parts of ethylene oxide (Example 25; viscosity 101 mPas); or with the adduct of 1 part of trimethylolpropane with 20 parts of ethylene oxide (Example 26; viscosity 206 mPas).

Examples 27 and 28

45 The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of castor oil with 40 parts of ethylene oxide; and
50 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10).

55 The suspension so obtained is heated, with stirring, to an internal temperature of 70-80°C. After 4-5 hours, an almost clear solution is obtained which contains less than 1% by weight of undissolved components. The solution is cooled to 50-60°C. and filtered. A clear yellow solution is obtained having an active substance content of 15% by weight, a water content of 25% by weight and a viscosity of 3000 mPas. The solution remains clear after standing for 2 days.

Similar results are obtained when the adduct of 1 part of castor oil with 40 parts of ethylene oxide is replaced by

EP 0 837 124 A2

an adduct of 1 part of castor oil with 11 parts of ethylene oxide.

Example 29

5 The following materials are charged into a 1 litre flask:

1132g. of an alkylpolyglycoside (53%) based on a C₁₀-C₁₂alkanol; and
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
10 having the formula (10).

The suspension so obtained is heated, with stirring, to an internal temperature of 70-80°C. The evaporated water is separated. After 4-5 hours, an almost clear solution is obtained which contains less than 1% by weight of undissolved components. The solution is cooled to 50-60°C. and filtered. A clear dark brown solution is obtained having an active substance content of 12% by weight, a water content of 42% by weight and a viscosity of 3500 mPas.

15

Examples 30 and 31

The following materials are charged into a 1 litre flask:

20 600g. of polyethylene glycol of mean molecular weight 300; and
400g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

25 The suspension so obtained is heated, with stirring, to an internal temperature of 70-80°C. After 4-5 hours, an almost clear solution is obtained which contains less than 1% by weight of undissolved components. The solution is cooled to 50-60°C. and filtered. A clear yellow solution is obtained having an active substance content of 20% by weight and a water content of 20% by weight. The solution remains clear after standing for 2 hours.

30 Similar results are obtained when the combination of 200g. of deionised water with 200g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10) is replaced
by a combination of 100g. of deionised water and 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10).

Example 32

35 The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of sorbitol monooleate with 20 parts of ethylene oxide; and
40 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

45 The suspension so obtained is heated, with stirring, to an internal temperature of 70-80°C. After 4-5 hours, an almost clear solution is obtained which contains less than 1% by weight of undissolved components. The solution is cooled to 50-60°C. and filtered. A clear yellow solution is obtained having an active substance content of 15% by weight, a water content of 25% by weight and a viscosity of 3300 mPas. The solution remains clear after standing for 2 days.

Example 33

50 The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of an ethoxylated lauric acid polyglycerol ester; and
55 300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent
having the formula (10).

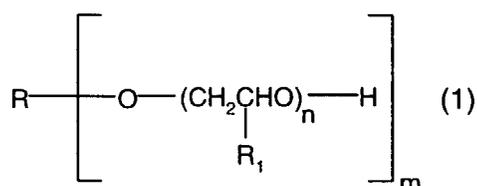
The suspension so obtained is heated, with stirring, to an internal temperature of 70-80°C. After 4-5 hours, a clear solution is obtained which contains less than 1% by weight of undissolved components. The solution is cooled to

50-60°C. and filtered. A clear yellow solution is obtained having an active substance content of 15% by weight, a water content of 25% by weight and a viscosity of 517 mPas. The solution remains clear after standing for 6 days.

5 Claims

1. A liquid fluorescent whitening agent formulation which is a solution having a viscosity of 50 to 5000 mPas and which comprises:

- 10 a) 10-25% by weight of an anionic fluorescent whitening agent, based on the total weight of the formulation;
 b) 15-35% by weight of a polar solvent, based on the total weight of the formulation; and
 c) 45-75% by weight, based on the total weight of the formulation, of a nonionic surfactant having the formula:



25 in which m is 1, 2, 3 or 4 and, when m is 1, R is a C₈-C₁₈-alkyl residue or a C₈-C₁₈-alkylcarboxy residue; when m is 2, R is a C₂-C₄alkylene glycol residue from which the two hydroxy groups have been removed; when m is 3, R is the residue of trimethylolpropane from which the three hydroxy groups have been removed; and when m is 4, R is the residue of pentaerythritol residue from which the four hydroxy groups have been removed; R₁ is hydrogen, methyl or ethyl; and n is a number ranging from 1 to 40.

30 2. A formulation according to claim 1 in which m is 1, 3 or 4 and, when m is 1, R is a C₈-C₁₈-alkyl residue or a C₈-C₁₈-alkylcarboxy residue, when m is 3, R is the residue of trimethylolpropane from which the three hydroxy groups have been removed and when m is 4, R is the residue of pentaerythritol residue from which the four hydroxy groups have been removed; R₁ is hydrogen, methyl or ethyl; and n is a number ranging from 1 to 40.

35 3. A formulation according to claim 1 or 2 which has a viscosity of 100 to 3500 mPas.

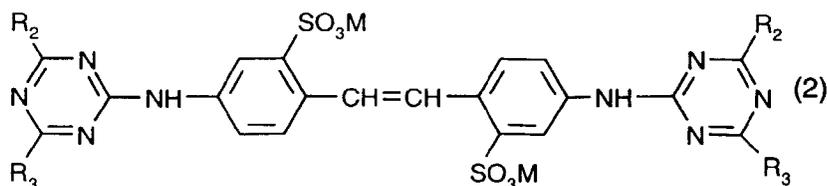
40 4. A formulation according to claim 3 which has a viscosity of 100 to 1000 mPas.

5. A formulation according to any of the preceding claims which contains 15-20% by weight of an anionic fluorescent whitening agent, based on the total weight of the formulation.

6. A formulation according to any of the preceding claims which contains 20-30% by weight of a polar solvent, based on the total weight of the formulation.

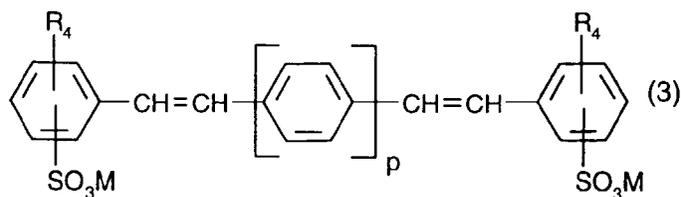
7. A formulation according to any of the preceding claims which contains 50-70% by weight, based on the total weight of the formulation, of a nonionic surfactant having the formula (1).

8. A formulation according to any of the preceding claims in which the anionic fluorescent whitening agent has either of the formulae:



or

5



10

in which R_2 and R_3 , independently, are OH, NH_2 , O-C₁-C₄-alkyl, O-aryl, NH-C₁-C₄-alkyl, N(C₁-C₄-alkyl)₂, N(C₁-C₄-alkyl)(C₁-C₄-hydroxyalkyl), N(C₁-C₄-hydroxyalkyl)₂, NH-aryl, morpholino, S-C₁-C₄-alkyl(aryl), Cl or OH; R_4 is H, SO_3M , O-C₁-C₄-alkyl, CN, Cl, $\text{COO-C}_1\text{-C}_4\text{-alkyl}$, or $\text{CON(C}_1\text{-C}_4\text{-alkyl)}_2$; M is H, Li, Na, K, Ca, Mg, ammonium, mono-, di-, tri- or tetra-C₁-C₄-alkylammonium, mono-, di- or tri-C₁-C₄-hydroxyalkylammonium or ammonium that is di- or tri-substituted with a mixture of C₁-C₄-alkyl and C₁-C₄-hydroxyalkyl groups; and p is 0 or 1.

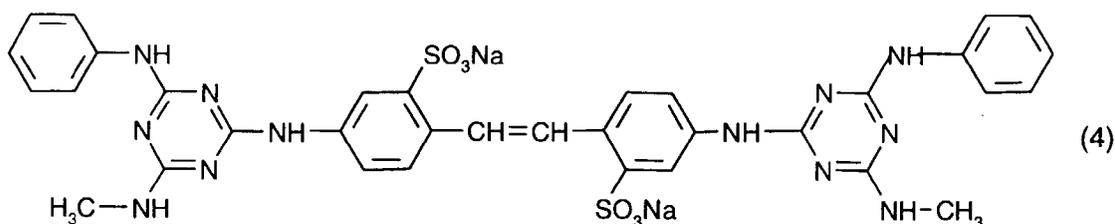
15

20

9. A formulation according to claim 8 in which R_2 and R_3 , independently, are methoxy, phenoxy, NH_2 , NH-methyl, N(methyl)₂, N(methyl)(hydroxyethyl), NH-ethyl, N(hydroxyethyl)₂, NH-phenyl, morpholino, S-methyl(phenyl), Cl or OH.

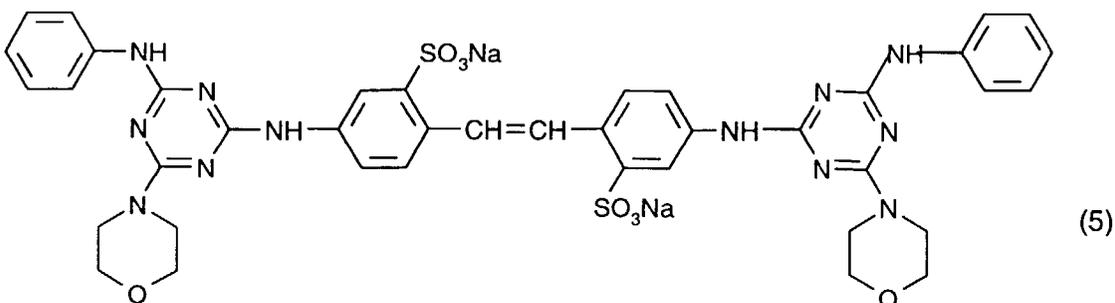
10. A formulation according to claim 8 or 9 in which the compound of formula (2) has one of the formulae:

25



30

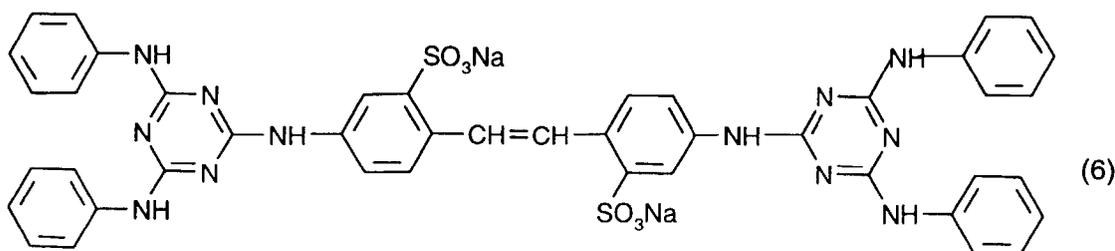
35



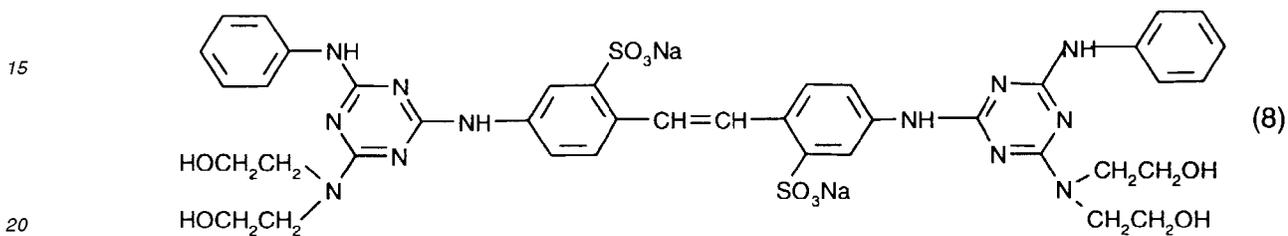
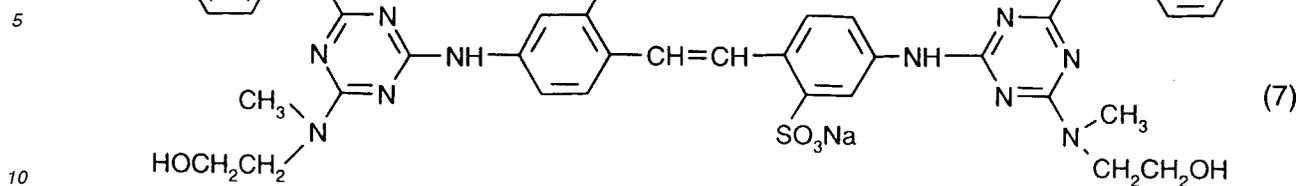
40

45

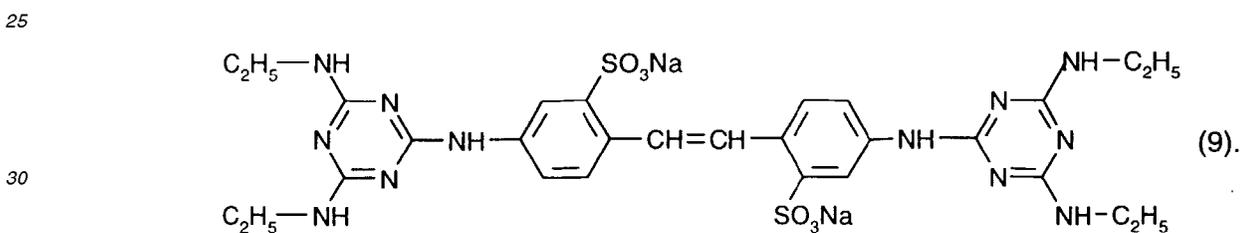
50



55

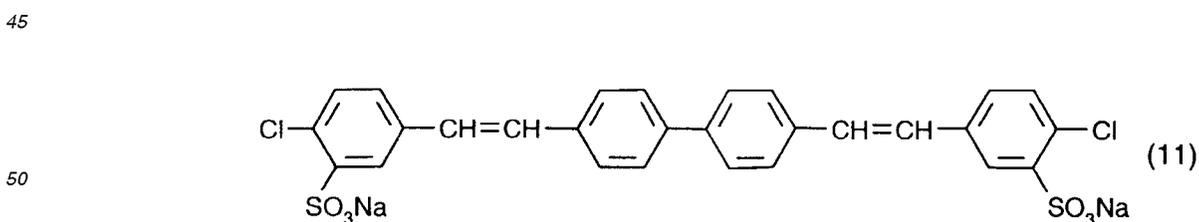
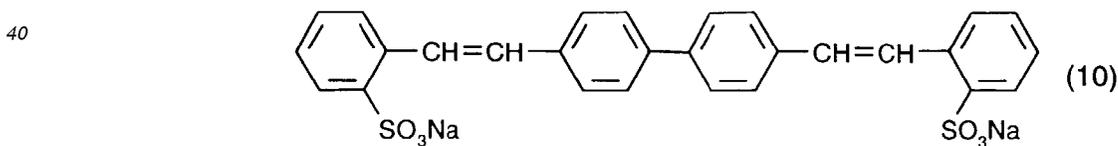


or



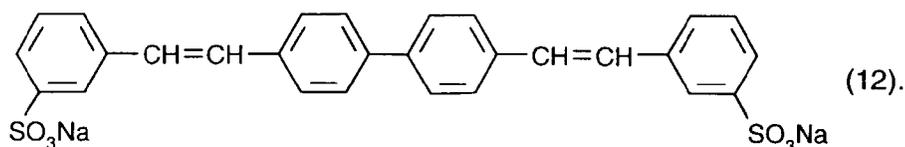
35 11. A formulation according to claim 8 in which, in the compounds of formula (3), R₄ is H or Cl and p is 1.

12. A formulation according to claim 8 or 11 in which the compound of formula (3) has one of the formulae:

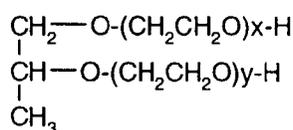


or

55



- 10 13. A formulation according to claim 12 in which the compound of formula (3) is the compound of formula (10) or a crystal modification thereof.
- 15 14. A formulation according to any of the preceding claims in which a mixture of two or more anionic fluorescent whitening agents is used as component a) of the formulation.
- 15 15. A formulation according to any of the preceding claims in which component b) is water.
16. A formulation according to claim 15 in which water is used together with one or more polar co-solvents.
- 20 17. A formulation according to claim 16 in which the polar co-solvent is an alkylene glycol or a polyalkylene glycol.
18. A formulation according to claim 17 in which the alkylene glycol is ethylene glycol or 1,2-propylene glycol.
19. A formulation according to claim 17 in which the polyalkylene glycol is polyethylene glycol or polypropylene glycol
- 25 20. A formulation according to any of claims 16 to 19 in which the amount of co-solvent ranges from 0-15% by weight, based on the total weight of the formulation.
- 30 21. A formulation according to any of the preceding claims in which, in the compound of formula (1), m is 1 and the compound is a polyadduct of 3-20 moles of ethylene oxide with 1 mole of a C₈-C₁₈-alkyl monoalcohol.
22. A formulation according to claim 21 in which the polyadduct is a polyadduct of 3-20 moles of ethylene oxide with 1 mole of a C₁₁-C₁₃-oxoalcohol or a polyadduct of 8-20 moles of ethylene oxide with 1 mole of a C₁₂-C₁₄-fatty alcohol.
- 35 23. A formulation according to any of claims 1 to 20 in which, in the compound of formula (1), m is 2 and the compound is a polyadduct of 2-20 moles of ethylene oxide with 1 mole of a C₂-C₄alkylene glycol.
24. A formulation according to claim 23 in which the C₂-C₄alkylene glycol is ethylene glycol or n-butylene glycol.
- 40 25. A formulation according to claim 23 in which the C₂-C₄alkylene glycol is propylene glycol.
26. A formulation according to claim 25 in which, in the compound of formula (1) has the formula:



in which x and y are each an integer within the range of from 1 to 10.

27. A formulation according to claim 26 in which x and y are each an integer within the range of from 1 to 5.
- 55 28. A formulation according to claim 27 in which the sum of x and y is about 10.
29. A formulation according to any of claims 1 to 20 in which, in the compound of formula (1), m is 3 and the compound is a polyadduct of 3-20 moles of ethylene oxide with 1 mole of trimethylolpropane.

EP 0 837 124 A2

30. A formulation according to claim 29 in which the compound is a polyadduct of 7-20 moles of ethylene oxide with 1 mole of trimethylolpropane.

5 31. A formulation according to any of claims 1 to 20 in which, in the compound of formula (1), m is 4 and the compound is a polyadduct of 4-20 moles of ethylene oxide with 1 mole of pentaerythritol.

32. A formulation according to claim 31 in which the compound is a polyadduct of 5-20 moles of ethylene oxide with 1 mole of pentaerythritol.

10 33. A formulation according to any of the preceding claims in which a mixture of two or more of the compounds of formula (1) is used as component c) of the formulation.

15 34. A formulation according to any of the preceding claims in which there is also present one or more auxiliaries selected from stabilisers which are effective in adjusting the flow properties of the formulation, anti-foam agents, alkaline agents, fabric softeners, anti-redeposition agents, antioxidants, auxiliary builders and fragrances.

20 35. A process for the production of a formulation according to claim 1 comprising mixing the anionic fluorescent whitening agent, solvent and the compound of formula (1), together with any auxiliaries, and homogenising the the mixture so obtained.

36. A process according to claim 35 in which the process is conducted at a temperature in the range of from 40-100°C.

25 37. A method for the production of a detergent comprising adding the required amount of the formulation of claim 1 to a detergent composition and then homogenising the mixture so obtained.

30

35

40

45

50

55