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(54) **Fluorescent whitening agent formulation**

Fluoreszierende Weisstöner-Formulierung

Formulation d'agent de blanchiment fluorescent

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Description

[0001] 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.

[0002] 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.

[0003] 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.

[0004] Also known are liquid formulations of fluorescent whitening agents which use organic solvents and are substantially anhydrous (e.g. FR-A-2654123, EP-A-0703293). These liquid formulations, however, are usually highly viscous and need to be incorporated into the detergent powder by means of special mixing techniques.

[0005] US-A-3,953,380 discloses liquid detergent compositions comprising fluorescent whitening agent at 0,5-5%, 40-75% non-ionic detergent and 5-35% water.

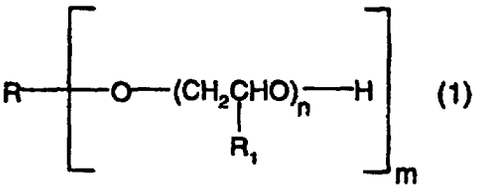
[0006] Surprisingly, it has now been found that anionic fluorescent whitening agents are soluble in a mixture comprising a predominant amount of a certain type of nonionic surfactant and a lesser amount of water, to provide stable, clear solutions of low viscosity.

[0007] 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 **water,** 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:



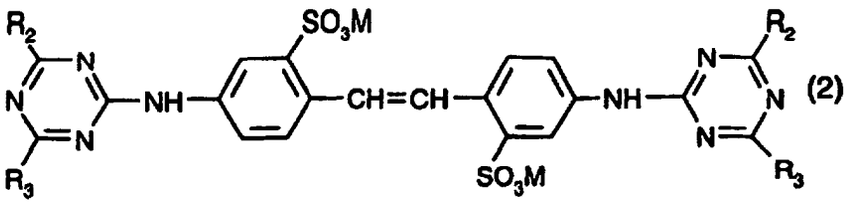
(1)

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_4alkylene 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.

[0008] Preferably, m is 1, 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 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.

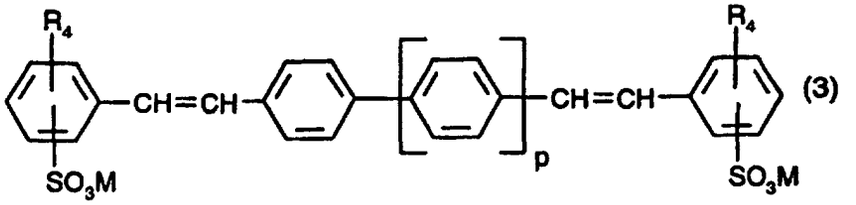
[0009] The viscosity of the liquid fluorescent whitening agent formulation according to the present invention is determined at a shear force of 100/s and at a temperature of 25°C. ± 1°C.

[0010] Preferred anionic fluorescent whitening agents for use in the present invention are those having either of the formulae:



(2)

or



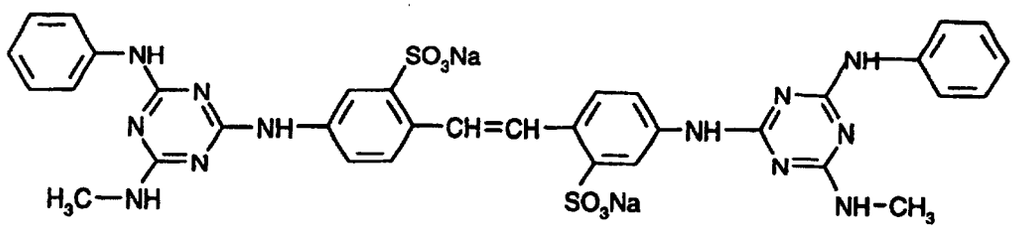
(3)

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, N(C_1-C_4-alkyl)$_2$, N(C_1-C_4-alkyl)(C_1-C_4-hydroxyalkyl), N(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, COO-C_1-C_4-alkyl, or CON(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.

[0011] 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.

[0012] Preferred compounds of formula (2) are those 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.

[0013] Specific examples of preferred compounds of formula (2) are those having one of the formulae:



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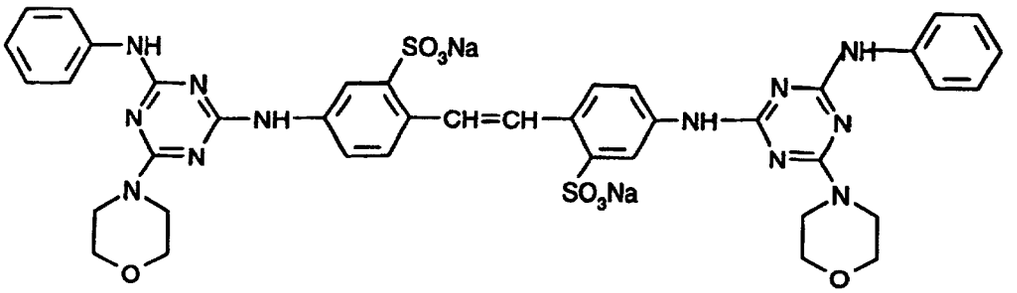

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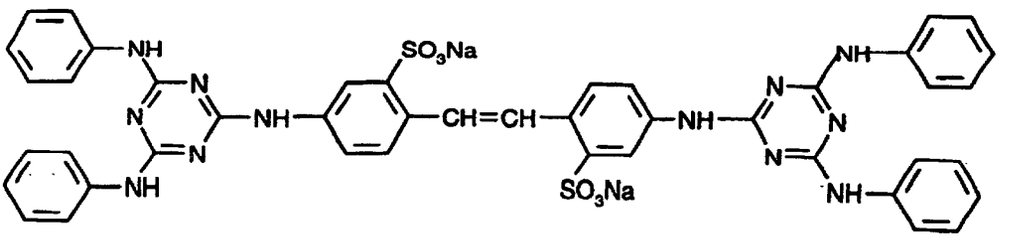

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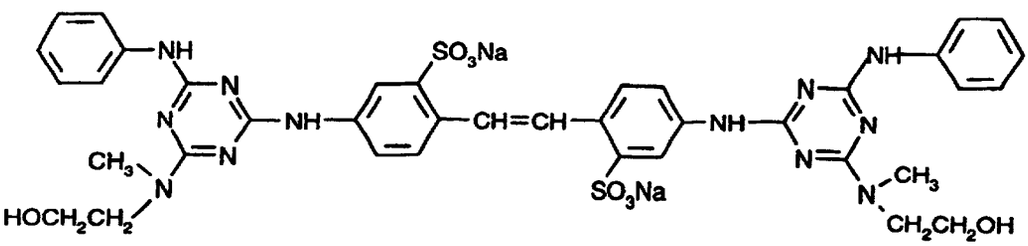

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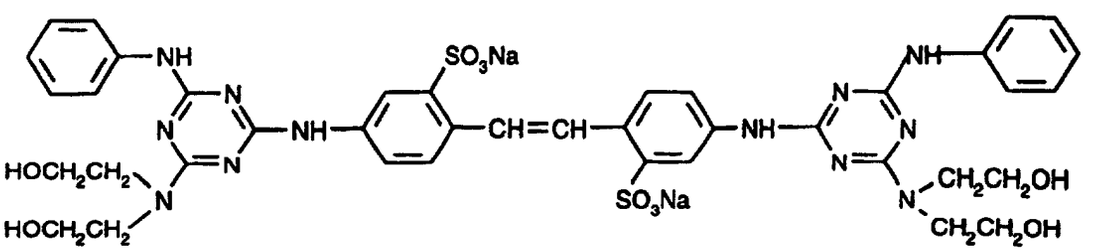

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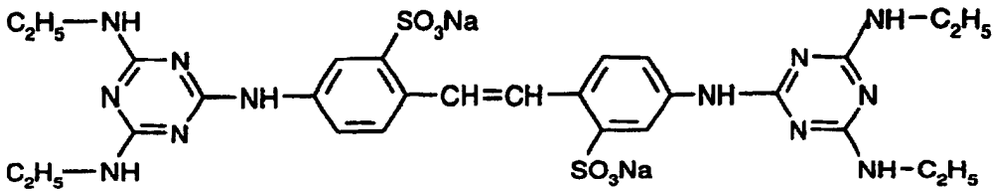
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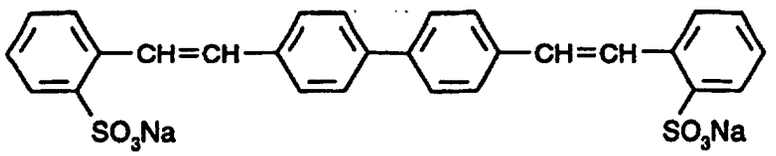
[0014] Preferred compounds of formula (3) are those in which R_4 is H or Cl and p is 1.

[0015] Specific preferred examples of compounds of formula (3) are those having one of the formulae: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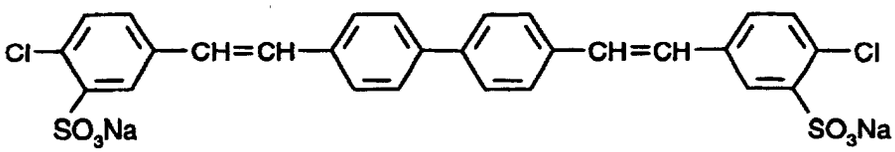
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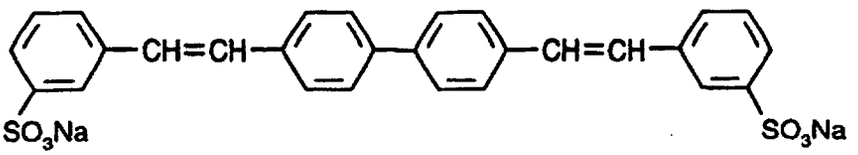

(12).

[0016] Especially preferred is the compound of formula (10) or a crystal modification thereof, as described in EP-A-0 577 557.

[0017] 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.

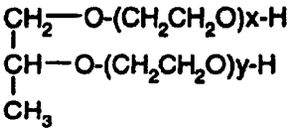
[0018] Component b) of the formulation according to the present invention, is water. If desired, however, water may be used together with one or more polar co-solvents such as an alkylene glycol, e.g. ethylene glycol or 1,2-propylene glycol, or a polyalkylene glycol, especially polyethylene glycol or polypropylene glycol. The amount of any co-solvent preferably ranges from 0-15% by weight, based on the total weight of the formulation.

[0019] The radical R in the compounds of formula (1), when m is 1, is derived from a C_8-C_{18}-alkyl monoalcohol. Examples of such monoalcohols include naturally-occuring 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, C_9-C_{13}-oxoalcohols, tridecyl alcohol, isodecyl alcohol or C_8-C_{18}-linear primary alcohols. Such C_8-C_{18}-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.

[0020] 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 C_8-C_{18}-alkyl monoalcohol. Especially interesting are polyadducts of 3-20 moles of ethylene oxide with 1 mole of a C_{11}-C_{13}-oxoalcohol and polyadducts of 8-20 moles of ethylene oxide with 1 mole of a C_{12}-C_{14}-fatty alcohol.

The radical R in the compounds of formula (1), when m is 2, is derived from a C_2-C_4alkylene glycol residue from which the two hydroxy groups have been removed. Examples of such C_2-C_4alkylene glycols include ethylene glycol, n-butylene glycol and, especially propylene glycol.

[0021] 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 C_2-C_4alkylene 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.

[0022] The alkyl radical R in the compounds of formula (1), when m is 3, is derived from trimethylolpropane.

[0023] 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.

[0024] The alkyl radical R in the compounds of formula (1), when m is 4, is derived from pentaerythritol.

[0025] 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.

[0026] 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.

[0027] The compounds of formula (2) to (12) are known and may be obtained by known methods.

[0028] 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.

[0029] Examples of such stabilisers include, e.g., kaolin, an Mg/Al silicate, especially bentonite, montmorillonite, a zeolite or a highly dispersed silicic acid.

[0030] 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 the mixture so obtained, preferably at an elevated temperature, e.g. at 40-100°C. Mixing is conveniently effected by a suitable stirring device.

[0031] 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.

[0032] 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.

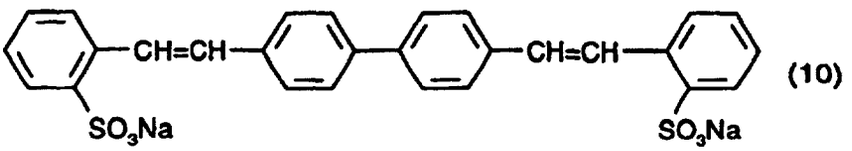
[0033] The following Examples further illustrate the present invention. Parts and percentages shown therein are by weight unless otherwise stated. The viscosity data quoted in the Examples are expressed in mPas and are determined at a shear force of 100/s and at a temperature of 25°C. ± 1°C.

Example 1

[0034] The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of a C_{13}-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:



(10)

[0035] 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.

Example 2

[0036] 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.

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	15	9	55	slightly opalescent	505

Example 3

[0037] The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of a C_{11}-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).

[0038] 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 4

[0039] The following materials are charged into a 1 litre flask:

100g. of deionised water;
420g. of an adduct of 1 part of a C_{13}-oxoalcohol with 8 parts of ethylene oxide;
180g. of an adduct of 1 part of a C_{13}-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).

[0040] 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 5

[0041] The following materials are charged into a 1 litre flask:

150g. of deionised water;
385g. of an adduct of 1 part of a C_{13}-oxoalcohol with 8 parts of ethylene oxide;
165g. of an adduct of 1 part of a C_{13}-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).

[0042] 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 6

[0043] The following materials are charged into a 1 litre flask:

50g. of deionised water;
455g. of an adduct of 1 part of a C_{13}-oxoalcohol with 8 parts of ethylene oxide;
195g. of an adduct of 1 part of a C_{13}-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).

[0044] 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 7 to 10

[0045] The following materials are charged into a 1 litre flask:

100g. of deionised water;
600g. of an adduct of 1 part of a C_{12}-C_{14}-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).

[0046] Using the procedure described in Example 1, a liquid formulation (Example 7) is obtained which is a clear solution at 25°C. and has a viscosity of 1600 mPas.

[0047] Similar good results are obtained when the adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 15 parts of ethylene oxide is replaced by an adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 20 parts of ethylene oxide (Example 8; viscosity 3261 mPas); or by an adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 8 parts of ethylene oxide (Example 9; 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 10; viscosity 2417 mPas).

Examples 11 and 12

[0048] The following materials are charged into a 1 litre flask:

100g. of deionised water;
420g. of an adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 8 parts of ethylene oxide;
180g. of an adduct of 1 part of a C_{12}-C_{14}-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).

[0049] Using the procedure described in Example 1, a liquid formulation (Example 11) is obtained which, after filtration, is a clear solution at 25°C. and has a viscosity of 732 mPas.

[0050] Similar good results are obtained when the amount of the adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 8 parts of ethylene oxide is reduced to 300g. and the amount of the adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 15 parts of ethylene oxide is increased to 300g (Example 12; viscosity 893 mPas).

Examples 13 and 14

[0051] The following materials are charged into a 1 litre flask:

150g. of deionised water;
385g. of an adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 8 parts of ethylene oxide;
165g. of an adduct of 1 part of a C_{12}-C_{14}-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).

[0052] Using the procedure described in Example 1, a liquid formulation (Example 13; viscosity 807 mPas) is obtained which, after filtration, is a clear solution at 25°C.

[0053] Similar good results are obtained when the amount of the adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 8 parts of ethylene oxide is reduced to 275g. and the amount of the adduct of 1 part of a C_{12}-C_{14}-fatty alcohol with 15 parts of ethylene oxide is increased to 275g (Example 14; viscosity 913 mPas).

Examples 15 to 17

[0054] 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 having the formula (10).

[0055] Using the procedure described in Example 1, a liquid formulation (Example 15; viscosity less than 500 mPas) is obtained which is a clear solution at 25°C.

[0056] 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 16; viscosity 446 mPas); or with the adduct of 1 part of trimethylolpropane with 20 parts of ethylene oxide (Example 17; viscosity 558 mPas).

Examples 18 to 20

[0057] 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 having the formula (10).

[0058] Using the procedure described in Example 1, a liquid formulation (Example 18; viscosity less than 500 mPas) is obtained which is a clear solution at 25°C.

[0059] 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 19; viscosity 101 mPas); or with the adduct of 1 part of trimethylolpropane with 20 parts of ethylene oxide (Example 20; viscosity 206 mPas).

Examples 21 and 22

[0060] 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
300g. of a moist presscake (containing 50% by weight of active substance) of the fluorescent whitening agent having the formula (10).

[0061] 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

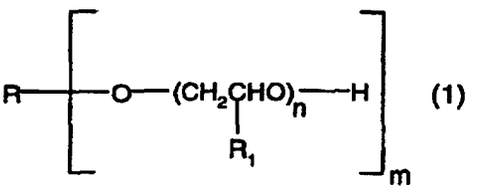
is cooled to 50-60°C. and filtered. A clear yellow solution is obtained having an active substance content of 15% by weight, a water content of 25% by weight and a viscosity of 3000 mPas. The solution remains clear after standing for 2 days.

[0062] Similar results are obtained when the adduct of 1 part of castor oil with 40 parts of ethylene oxide is replaced by an adduct of 1 part of castor oil with 11 parts of ethylene oxide.

Claims

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

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



(1)

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_4alkylene 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.

2. A formulation according to claim 1 in which m is 1, 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 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.

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

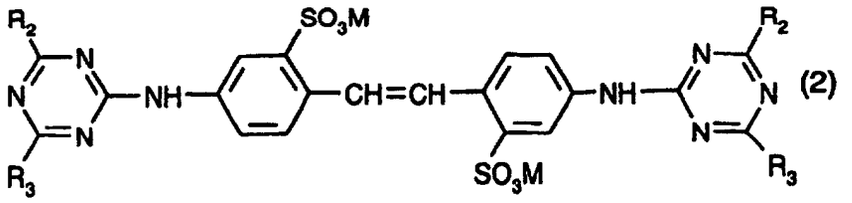
4. A formulation according to claim 3 which has a viscosity of 100 to 1000 mPas.

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.

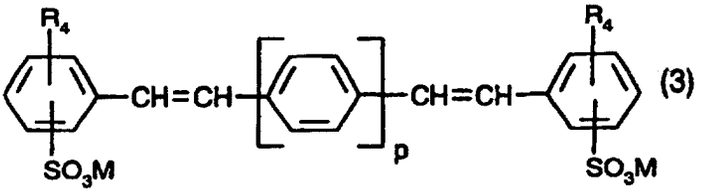
7. A formulation according to any of the preceding claims which contains 50-70% by weight, based on the total weight of the formulation, of a nonionic surfactant having the formula (1).

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



(2)

or

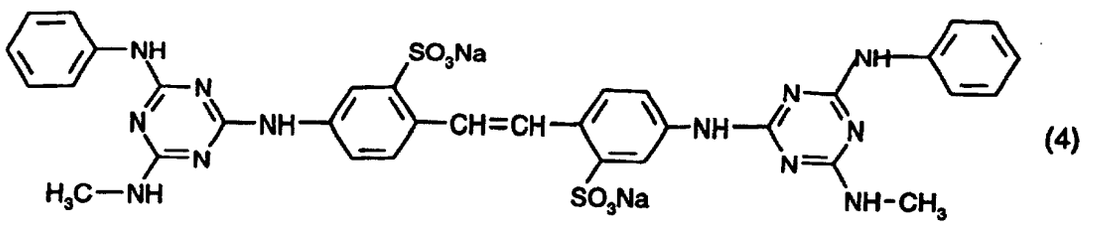


(3)

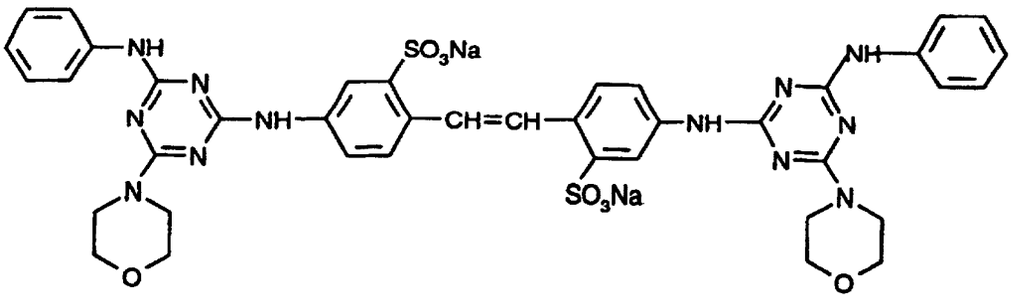
in which R_2 and R_3, independently, are OH, NH_2, O-C_1-C_4-alkyl, O-aryl, NH-C_1-C_4-alkyl, N(C_1-C_4-alkyl)$_2$, N(C_1-C_4-alkyl)(C_1-C_4-hydroxyalkyl), N(C_1-C_4-hydroxyalkyl)$_2$, NH-aryl, morpholino, S-C_1-C_4-alkyl(aryl), Cl or OH; R_4 is H, SO_3M, O-C_1-C_4-alkyl, CN, Cl, COO-C_1-C_4-alkyl, or CON(C_1-C_4-alkyl)$_2$; M is H, U, 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.

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.

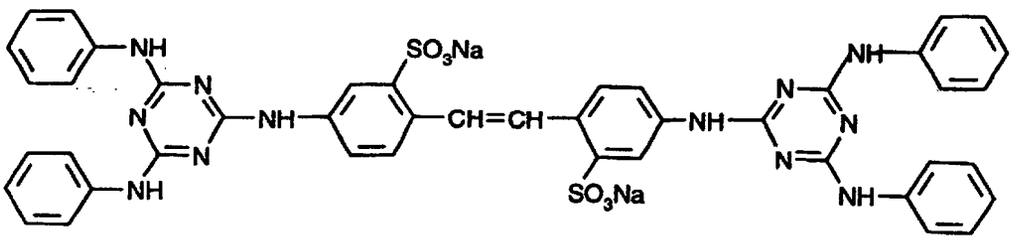
10. A formulation according to claim 8 or 9 in which the compound of formula (2) has one of the formulae:



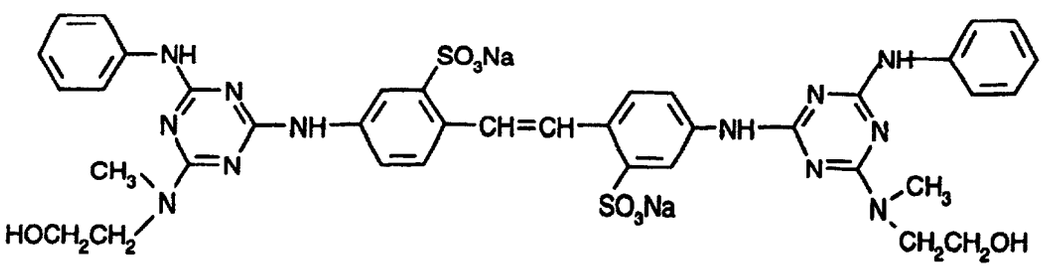
(4)



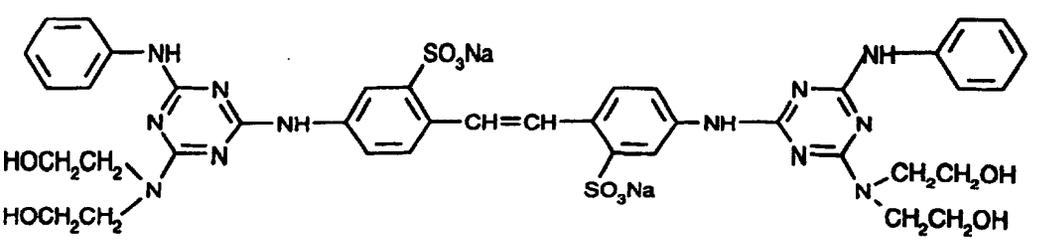
(5)



(6)

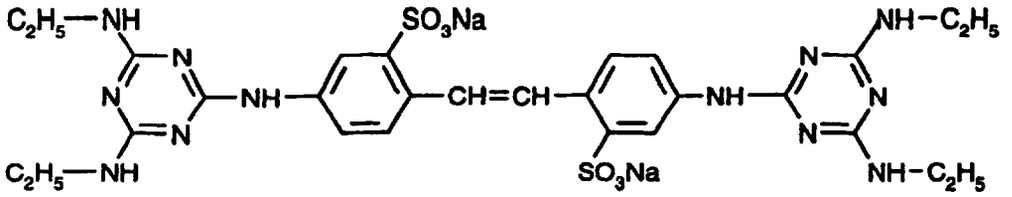


(7)



(8)

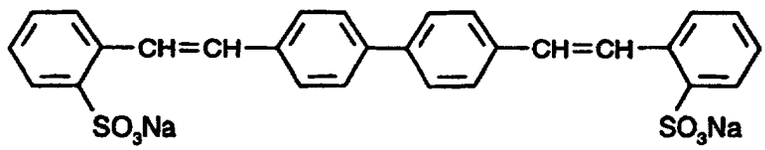
or



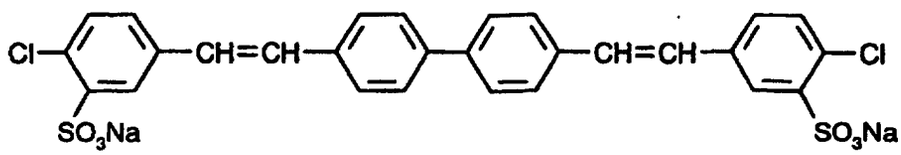
(9).

11. A formulation according to claim 8 in which, in the compounds of formula (3), R_4 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:

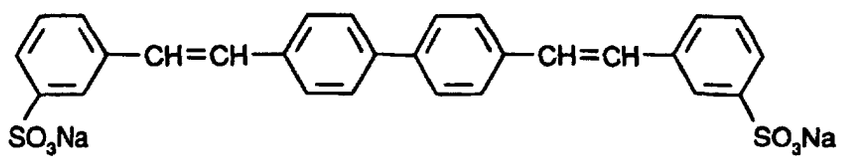


(10)



(11)

or



(12).

13. A formulation according to claim 12 in which the compound of formula (3) is the compound of formula (10) or a crystal modification of the compound of formula (10).

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. A formulation according to claim 1 in which water is used together with one or more polar co-solvents.

16. A formulation according to claim 15 in which the polar co-solvent is an alkylene glycol or a polyalkylene glycol.

17. A formulation according to claim 16 in which the alkylene glycol is ethylene glycol or 1,2-propylene glycol.

18. A formulation according to claim 16 in which the polyalkylene glycol is polyethylene glycol or polypropylene glycol

19. A formulation according to any of claims 15 to 18 in which the amount of co-solvent ranges from 0-15% by weight, based on the total weight of the formulation.

20. 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 C_8-C_{18}-alkyl monoalcohol.

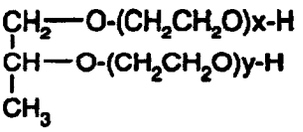
21. A formulation according to claim 20 in which the polyadduct is a polyadduct of 3-20 moles of ethylene oxide with 1 mole of a C_{11}-C_{13}-oxoalcohol or a polyadduct of 8-20 moles of ethylene oxide with 1 mole of a C_{12}-C_{14}-fatty alcohol.

22. A formulation according to any of claims 1 to 19 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 C_2-C_4alkylene glycol.

23. A formulation according to claim 22 in which the C_2-C_4alkylene glycol is ethylene glycol or n-butylene glycol.

24. A formulation according to claim 22 in which the C_2-C_4alkylene glycol is propylene glycol.

25. A formulation according to claim 24 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.

26. A formulation according to claim 25 in which x and y are each an integer within the range of from 1 to 5.

27. A formulation according to claim 26 in which the sum of x and y is 10.

28. A formulation according to any of claims 1 to 19 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.

29. A formulation according to claim 28 in which the compound is a polyadduct of 7-20 moles of ethylene oxide with 1 mole of trimethylolpropane.

30. A formulation according to any of claims 1 to 19 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.

31. A formulation according to claim 30 in which the compound is a polyadduct of 5-20 moles of ethylene oxide with 1 mole of pentaerythritol.

32. 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.

33. 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.

34. 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.

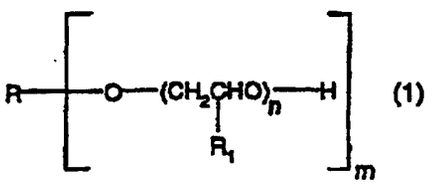
35. A process according to claim 34 in which the process is conducted at a temperature in the range of from 40-100°C.

36. 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.

Patentansprüche

1. Flüssige Fluoreszenz-Weißmacherformulierung, welche eine Lösung mit einer Viskosität von 50 bis 5000 mPas ist, und welche umfaßt:

a) 10-25 Gew.-% mindestens eines anionischen Fluoreszenz-Weißmachers, basierend auf dem Gesamtgewicht der Formulierung;
b) 15-35 Gew.-% Wasser, basierend auf dem Gesamtgewicht der Formulierung; und
c) 45-75 Gew.-%, bezogen auf das Gesamtgewicht der Formulierung, mindestens eines nicht-ionischen Tensids der Formel:



(1)

worin m 1, 2, 3 oder 4 ist, und wenn m 1 ist, R ein C_8-C_{18}-Alkylrest oder ein C_8-C_{18}-Alkylcarboxyrest ist; wenn m 2 ist, R ein C_2-C_4-Alkylenglykolrest ist, von welchem die zwei Hydroxygruppen entfernt worden sind; wenn m 3 ist, R der Rest von Trimethylolpropan ist, von dem die drei Hydroxygruppen entfernt worden sind; und wenn m 4 ist, R der Rest von Pentaerythritol ist, von dem die vier Hydroxygruppen entfernt worden sind; R_1 Wasserstoff, Methyl oder Ethyl ist; und n eine Zahl im Bereich von 1 bis 40 ist.

2. Formulierung nach Anspruch 1, wobei m 1, 3 oder 4 ist und, wenn m 1 ist, R ein C_8-C_{18}-Alkylrest oder ein C_8-C_{18}-Alkylcarboxyrest ist, wenn m 3 ist, R der Rest von Trimethylolpropan ist, von dem die drei Hydroxygruppen entfernt worden sind; und wenn m 4 ist, R der Rest von Pentaerythritol ist, von dem die vier Hydroxygruppen entfernt worden sind; R_1 Wasserstoff, Methyl oder Ethyl ist; und n eine Zahl im Bereich von 1 bis 40 ist.

3. Formulierung nach Anspruch 1 oder 2, welche eine Viskosität von 100 bis 3500 mPas besitzt.

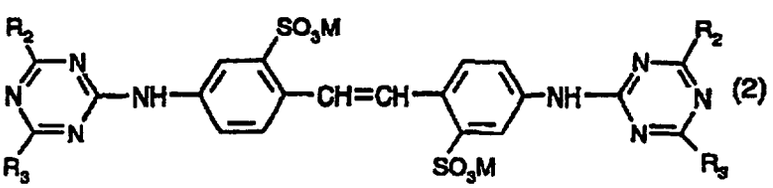
4. Formulierung nach Anspruch 3, welche eine Viskosität von 100 bis 1000 mPas besitzt.

5. Formulierung nach mindestens einem der vorangehenden Ansprüche, enthaltend 15-20 Gew.-% eines anionischen Fluoreszenz-Weißmachers, bezogen auf das Gesamtgewicht der Formulierung.

6. Formulierung nach mindestens einem der vorangehenden Ansprüche, enthaltend 20-30 Gew.-% eines polaren Lösungsmittels, bezogen auf das Gesamtgewicht der Formulierung.

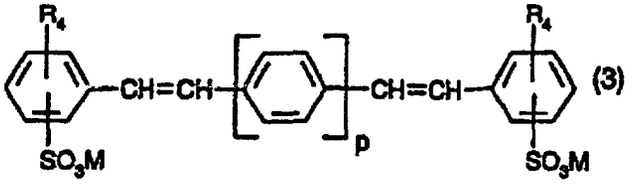
7. Formulierung nach mindestens einem der vorangehenden Ansprüche, enthaltend 50-70 Gew.-%, bezogen auf das Gesamtgewicht der Formulierung, eines nicht-ionischen Tensids der Formel (1).

8. Formulierung nach mindestens einem der vorangehenden Ansprüche, wobei der anionische Fluoreszenz-Weißmacher entweder die Formel



(2)

oder

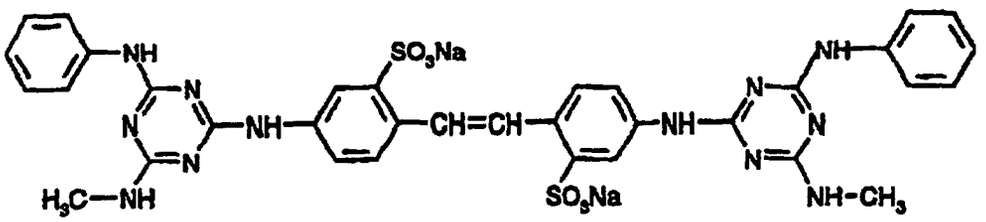


(3)

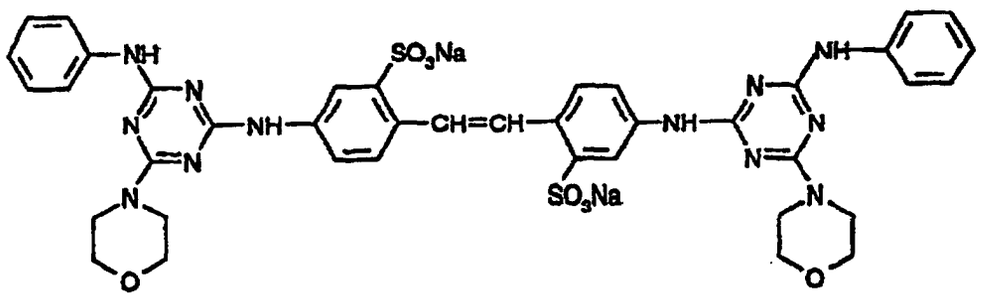
besitzt, worin R_2 und R_3 unabhängig OH, NH_2, O-C_1-C_4-Alkyl, O-Aryl, NH-C_1-C_4-Alkyl, N(C_1-C_4-Alkyl)$_2$, N(C_1-C_4-Alkyl)(C_1-C_4-hydroxyalkyl), N(C_1-C_4-Hydroxyalkyl)$_2$, NH-Aryl, Morpholino, S-C_1-C_4-Alkyl(aryl), Cl oder OH sind; R_4 H, SO_3M, O-C_1-C_4-Alkyl, CN, Cl, COO-C_1-C_4-Alkyl oder CON(C_1-C_4-Alkyl)$_2$ ist; M H, Li, Na, K, Ca, Mg, Ammonium, Mono-, Di-, Tri- oder Tetra-C_1-C_4-alkylammonium, Mono-, Di- oder Tri-C_1-C_4-hydroxyalkylammonium oder Ammonium, welches dioder tri-substituiert ist mit einer Mischung aus C_1-C_4-Alkyl- und C_1-C_4-Hydroxyalkylgruppen; und p 0 oder 1 ist.

9. Formulierung nach Anspruch 8, wobei R_2 und R_3 unabhängig voneinander Methoxy, Phenoxy, NH_2, NH-Methyl, N(Methyl)$_2$, N(Methyl)(hydroxyethyl), NH-Ethyl, N(Hydroxyethyl)$_2$, NH-Phenyl, Morpholino, S-Methyl(phenyl), Cl oder OH sind.

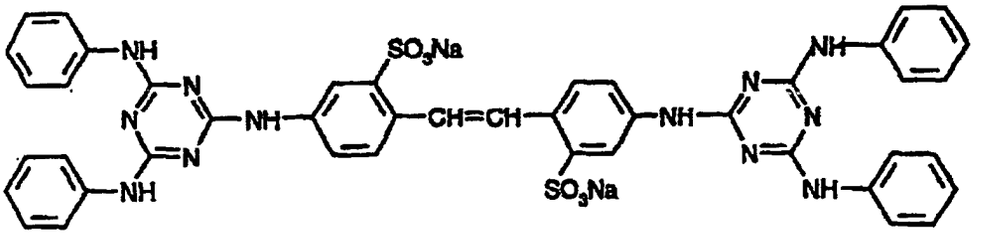
10. Formulierung nach Anspruch 8 oder 9, wobei die Verbindung der Formel (2) eine der folgenden Formeln besitzt:



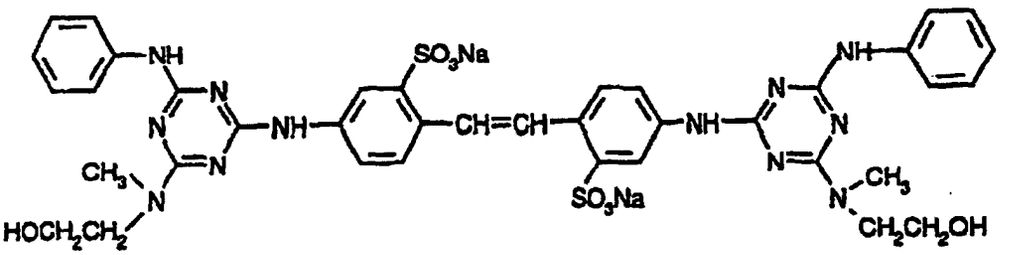
(4)



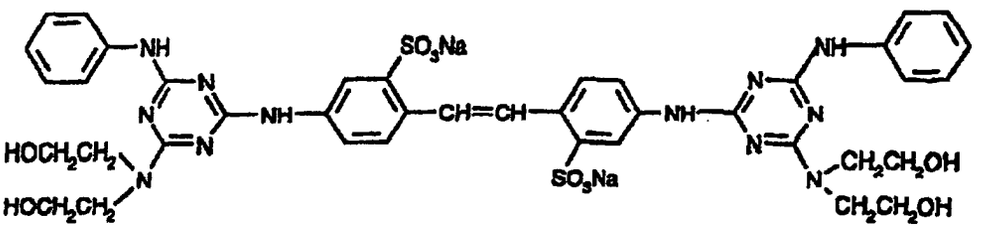
(5)



(6)

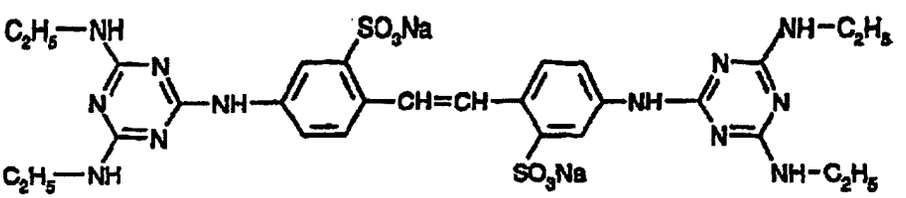


(7)



(8)

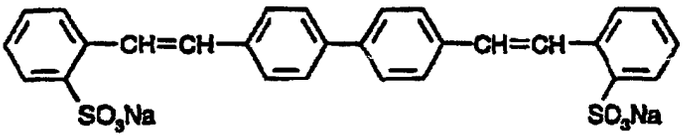
oder



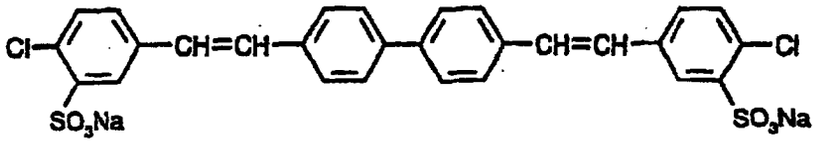
(9).

11. Formulierung nach Anspruch 8, wobei in den Verbindungen der Formel (3) R_4 H oder Cl ist und p 1 ist.

12. Formulierung nach Anspruch 8 oder 11, wobei die Verbindung der Formel (3) eine der Formeln besitzt:

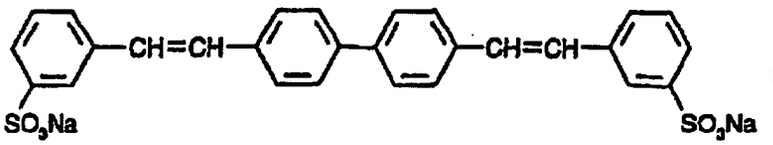


(10)



(11)

oder



(12).

13. Formulierung nach Anspruch 12, wobei die Verbindung der Formel (3) die Verbindung der Formel (10) oder eine Kristallmodifikation der Verbindung der Formel (10) ist.

14. Formulierung nach mindestens einem der vorangehenden Ansprüche, wobei eine Mischung aus zwei oder mehreren anionischen Fluoreszenz-Weißmachern als Komponente a) der Formulierung verwendet wird.

15. Formulierung nach Anspruch 1, wobei Wasser zusammen mit einem oder mehreren polaren Co-Lösungsmitteln verwendet wird.

16. Formulierung nach Anspruch 15, wobei das polare Co-Lösungsmittel ein Alkylenglykol oder Polyalkylenglykol ist.

17. Formulierung nach Anspruch 16, wobei das Alkylenglykol Ethylenglykol oder 1,2-Propylenglykol ist.

18. Formulierung nach Anspruch 16, wobei das Polyalkylenglykol Polyethylenglykol oder Polypropylenglykol ist.

19. Formulierung nach mindestens einem der Ansprüche 15 bis 18, wobei die Menge an Co-Lösungsmittel im Bereich von 0-15 Gew.-% liegt, bezogen auf das Gesamtgewicht der Formulierung.

20. Formulierung nach mindestens einem der vorangehenden Ansprüche, wobei in der Verbindung der Formel (1) m 1 ist und die Verbindung ein Polyaddukt aus 3-20 Molen Ethylenoxid mit 1 Mol eines C_8-C_{18}-Alkylmonoalkohols ist.

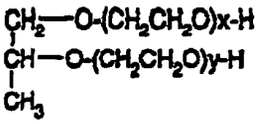
21. Formulierung nach Anspruch 20, wobei das Polyaddukt ein Polyaddukt aus 3-20 Molen Ethylenoxid mit 1 Mol eines C_{11}-C_{13}-Oxoalkohols oder ein Polyaddukt aus 8-20 Molen Ethylenoxid mit 1 Mol eines C_{12}-C_{14}-Fettalkohols ist.

22. Formulierung nach mindestens einem der Ansprüche 1 bis 19, wobei in der Verbindung der Formel (1) m 2 ist und die Verbindung ein Polyaddukt aus 2-20 Molen Ethylenoxid mit 1 Mol C_2-C_4-Alkylenglykol ist.

23. Formulierung nach Anspruch 22, wobei das C_2-C_4-Alkylenglykol Ethylenglykol oder n-Butylenglykol ist.

24. Formulierung nach Anspruch 22, wobei das C_2-C_4-Alkylenglykol Propylenglykol ist.

25. Formulierung nach Anspruch 24, wobei die Verbindung der Formel (1) die Formel besitzt:



worin x und y jeweils eine ganze Zahl innerhalb des Bereiches von 1 bis 10 sind.

26. Formulierung nach Anspruch 25, wobei x und y jeweils eine ganze Zahl innerhalb des Bereichs von 1 bis 5 sind.

27. Formulierung nach Anspruch 26, wobei die Summe aus x und y 10 ist.

28. Formulierung nach mindestens einem der Ansprüche 1 bis 19, wobei in der Verbindung der Formel (1) m 3 ist und die Verbindung ein Polyaddukt aus 3-20 Molen Ethylenoxid mit 1 Mol Trimethylolpropan ist.

29. Formulierung nach Anspruch 28, wobei die Verbindung ein Polyaddukt aus 7-20 Molen Ethylenoxid mit 1 Mol Trimethylolpropan ist.

30. Formulierung nach mindestens einem der Ansprüche 1 bis 19, wobei in der Verbindung der Formel (1) m 4 ist und die Verbindung ein Polyaddukt aus 4-20 Molen Ethylenoxid mit 1 Mol Pentaerythritol ist.

31. Formulierung nach Anspruch 30, wobei die Verbindung ein Polyaddukt aus 5-20 Molen Ethylenoxid mit 1 Mol Pentaerythritol ist.

32. Formulierung nach mindestens einem der vorangehenden Ansprüche, wobei eine Mischung aus zwei oder mehreren der Verbindungen der Formel (1) als Komponente c) der Formulierung verwendet wird.

33. Formulierung nach mindestens einem der vorangehenden Ansprüche, wobei ebenfalls ein oder mehrere Hilfsmittel vorliegen, gewählt aus Stabilisatoren, welche bei der Einstellung der Fließeigenschaften der Formulierung wirksam sind, Antischaummitteln, Alkalimitteln, Textilweichmachern, Anti-Wiederablagerungsmitteln, Antioxidantien, Hilfsgerüststoffen und Duftstoffen.

34. Verfahren zur Herstellung einer Formulierung nach Anspruch 1, umfassend das Vermischen des anionischen Fluoreszenz-Weißmachers, Lösungsmittels und der Verbindung der Formel (1), zusammen mit beliebigen Hilfsmitteln und Homogenisieren der so erhaltenen Mischung.

35. Verfahren nach Anspruch 34, wobei das Verfahren bei einer Temperatur im Bereich von 40-100°C durchgeführt wird.

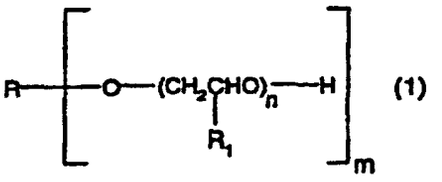
36. Verfahren zur Herstellung eines Detergensmittels, umfassend das Zugeben der erforderlichen Menge der Formulierung nach Anspruch 1 zu einer Detergenszusammensetzung und dann Homogenisieren der so erhaltenen Mischung.

Revendications

1. Formulation liquide d'agent de blanchiment fluorescent qui est une solution ayant une viscosité de 50 à 5000 mPa. s et qui comprend :

a) 10 à 25 % en poids d'au moins un agent de blanchiment fluorescent anionique, par rapport au poids total de la formulation ;
b) 15 à 35 % en poids d'eau, par rapport au poids total de la formulation ; et

c) 45 à 75 % en poids, par rapport au poids total de la formulation, d'au moins un agent tensio-actif non ionique ayant la formule :



(1)

dans laquelle m est 1, 2, 3 ou 4 et, lorsque m est 1, R est un résidu alkyle en C_8-C_{18} ou un résidu alkylcarboxy en C_8-C_{18} ; lorsque m est 2, R est un résidu d'alkylène-glycol en C_2-C_4 duquel les deux groupes hydroxyle ont été enlevés ; lorsque m est 3, R est le résidu de triméthylolpropane duquel les trois groupes hydroxyle ont été enlevés ; et lorsque m est 4, R est le résidu de pentaérythritol duquel les quatre groupes hydroxyle ont été enlevés ; R_1 est l' hydrogène, un groupe méthyle ou éthyle ; et n est un nombre compris entre 1 et 40.

2. Formulation selon la revendication 1, dans laquelle m est 1, 3 ou 4 et, lorsque m est 1, R est un résidu alkyle en C_8-C_{18} ou un résidu alkylcarboxy en C_8-C_{18}, lorsque m est 3, R est le résidu de triméthylolpropane duquel les trois groupes hydroxyle ont été enlevés, et lorsque m est 4, R est le résidu de pentaérythritol duquel les quatre groupes hydroxyle ont été enlevés ; R_1 est l'hydrogène, un groupe méthyle ou éthyle ; et n est un nombre compris entre 1 et 40.

3. Formulation selon la revendication 1 ou 2, qui a une viscosité de 100 à 3500 mPa.s.

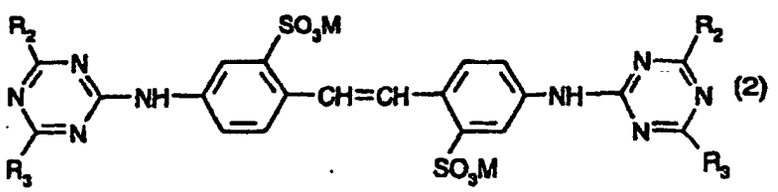
4. Formulation selon la revendication 3, qui a une viscosité de 100 à 1000 mPa.s.

5. Formulation selon l'une quelconque des revendications précédentes, qui contient 15 à 20 % en poids d'un agent de blanchiment fluorescent anionique, par rapport au poids total de la formulation.

6. Formulation selon l'une quelconque des revendications précédentes, qui contient 20 à 30 % en poids d'un solvant polaire, par rapport au poids total de la formulation.

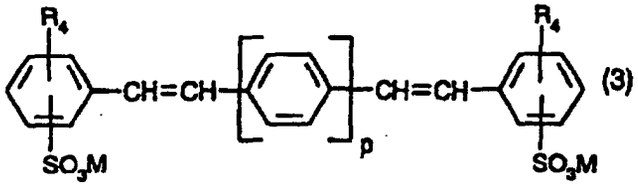
7. Formulation selon l'une quelconque des revendications précédentes, qui contient 50 à 70 % en poids, par rapport au poids total de la formulation, d'un agent tensio-actif non ionique ayant la formule (1).

8. Formulation selon l'une quelconque des revendications précédentes, dans laquelle l'agent de blanchiment fluorescent anionique est représenté par l'une ou l'autre des formules :



(2)

ou

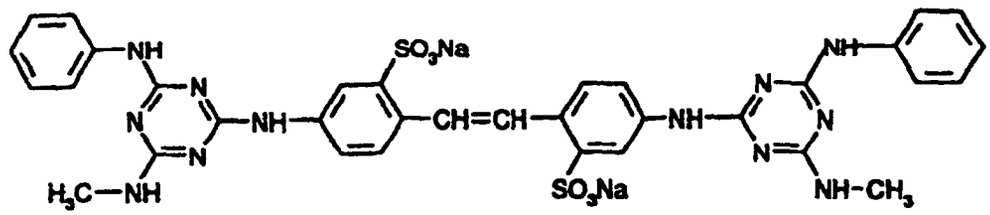


(3)

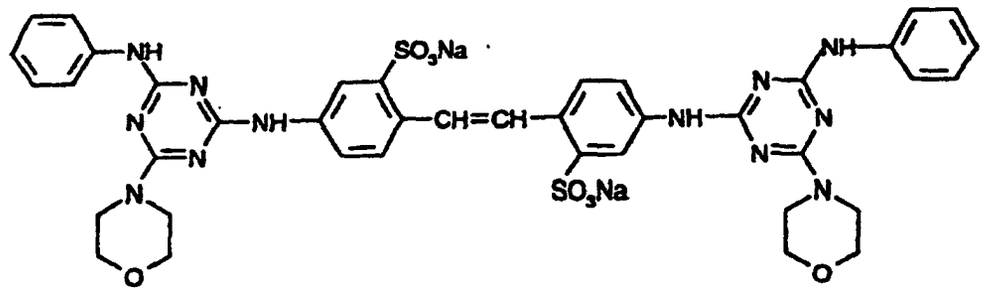
dans lesquelles R_2 et R_3 sont indépendamment des groupes OH, NH_2, O-alkyle en C_1-C_4, O-aryle, NH-alkyle en C_1-C_4, N(alkyle en C_1-C_4)$_2$, N(alkyle en C_1-C_4)(hydroxyalkyle en C_1-C_4), N(hydroxyalkyle en C_1-C_4)$_2$, NH-aryle, morpholino, S-alkyle en C_1-C_4(aryle), Cl ou OH ; R_4 est H, SO_3M, un groupe O-alkyle en C_1-C_4, CN, Cl, COO-alkyle en C_1-C_4 ou CON(alkyle en C_1-C_4)$_2$; M est H, Li, Na, K, Ca, Mg, l'ammonium, un mono-, di-, tri- ou tétra-(alkyle en C_1-C_4)ammonium, un mono-, di- ou tri-(hydroxyalkyle en C_1-C_4)ammonium ou un ammonium qui est di- ou trisubstitué par un mélange de groupes alkyle en C_1-C_4 et hydroxyalkyle en C_1-C_4 ; et p est 0 ou 1.

9. Formulation selon la revendication 8, dans laquelle R_2 et R_3 sont indépendamment des groupes méthoxy, phénoxy, NH_2, NH-méthyle, N(méthyle)$_2$, N(méthyl)(hydroxyéthyle), NH-éthyle, N(hydroxyéthyle)$_2$, NH-phényle, morpholino, S-méthyl(phényle), Cl ou OH.

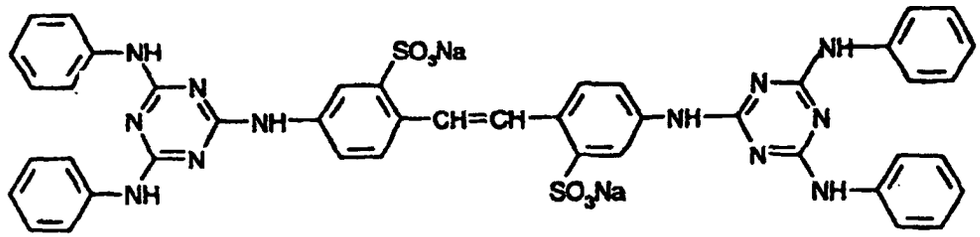
10. Formulation selon la revendication 8 ou 9, dans laquelle le composé de formule (2) répond à l'une des formules :



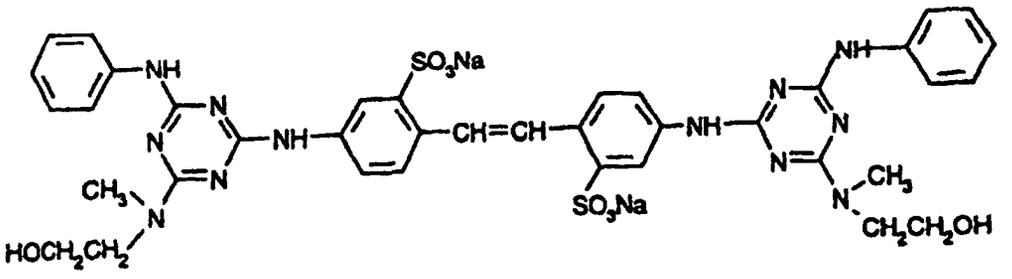
(4)



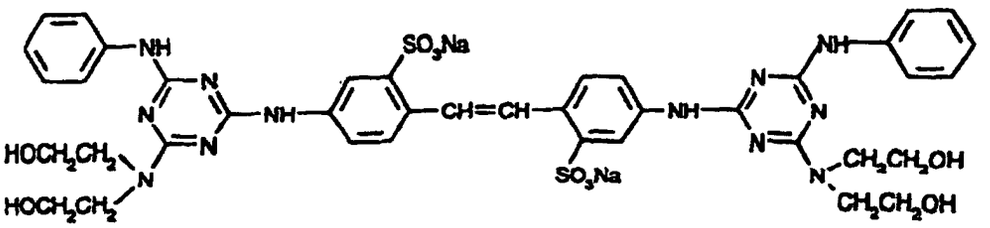
(5)



(6)

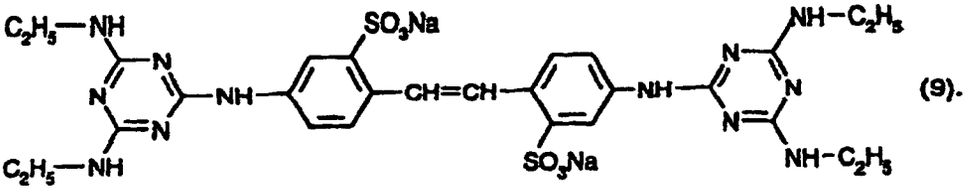


(7)



(8)

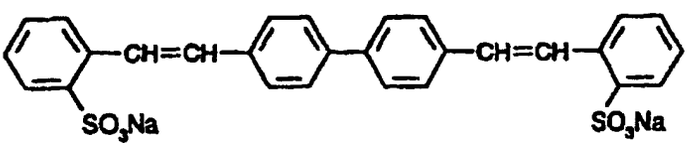
ou



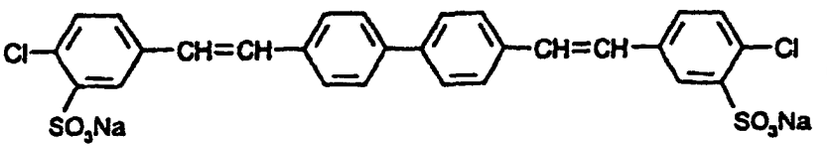
(9).

11. Formulation selon la revendication 8, dans laquelle, dans les composés de formule (3), R_4 est H ou Cl et p est 1.

12. Formulation selon la revendication 8 ou 11, dans laquelle le composé de formule (3) répond à l'une des formules :

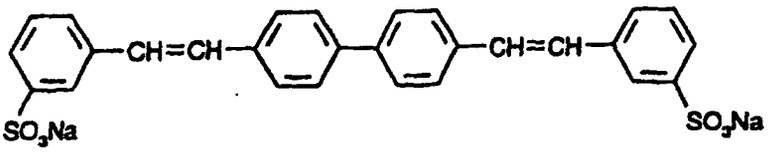


(10)



(11)

ou



(12).

13. Formulation selon la revendication 12, dans laquelle le composé de formule (3) est le composé de formule (10) ou une variété cristalline du composé de formule (10).

14. Formulation selon l'une quelconque des revendications précédentes, dans laquelle on utilise un mélange de deux ou plusieurs agents de blanchiment fluorescents anioniques comme composant a) de la formulation.

15. Formulation selon la revendication 1, dans laquelle on utilise de l'eau conjointement à un ou plusieurs co-solvants polaires.

16. Formulation selon la revendication 15, dans laquelle le co-solvant polaire est un alkylène-glycol ou un polyalkylène-glycol.

17. Formulation selon la revendication 16, dans laquelle l'alkylène-glycol est l'éthylène-glycol ou le 1,2-propylène-glycol.

18. Formulation selon la revendication 16, dans laquelle le polyalkylène-glycol est un polyéthylène-glycol ou un polypropylène-glycol.

19. Formulation selon l'une quelconque des revendications 15 à 18, dans laquelle la quantité de co-solvant est comprise entre 0 et 15 % en poids par rapport au poids total de la formulation.

20. Formulation selon l'une quelconque des revendications précédentes, dans laquelle, dans le composé de formule (1), m est 1 et le composé est un produit de polyaddition de 3 à 20 moles d'oxyde d'éthylène avec 1 mole d'un monoalcool alkylique en C_8-C_{18}.

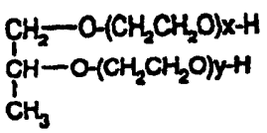
21. Formulation selon la revendication 20, dans laquelle le produit de polyaddition est un produit de polyaddition de 3 à 20 moles d'oxyde d'éthylène avec 1 mole d'un oxoalcool en C_{11}-C_{13} ou un produit de polyaddition de 8 à 20 moles d'oxyde d'éthylène avec 1 mole d'un alcool gras en C_{12}-C_{14}.

22. Formulation selon l'une quelconque des revendications 1 à 19, dans laquelle, dans le composé de formule (1), m est 2 et le composé est un produit de polyaddition de 2 à 20 moles d'oxyde d'éthylène avec 1 mole d'un alkylène-glycol en C_2-C_4.

23. Formulation selon la revendication 22, dans laquelle l'alkylène-glycol en C_2-C_4 est l'éthylène-glycol ou le n-butylène-glycol.

24. Formulation selon la revendication 22, dans laquelle l'alkylène-glycol en C_2-C_4 est le propylène-glycol.

25. Formulation selon la revendication 24, dans laquelle le composé de formule (1) répond à la formule :



dans laquelle chacun de x et y est un nombre entier compris dans l'intervalle de 1 à 10.

26. Formulation selon la revendication 25, dans laquelle chacun de x et y est un nombre entier compris dans l'intervalle

de 1 à 5.

27. Formulation selon la revendication 26, dans laquelle la somme de x et y est égale à 10.

28. Formulation selon l'une quelconque des revendications 1 à 19, dans laquelle, dans le composé de formule (1), m est 3 et le composé est un produit de polyaddition de 3 à 20 moles d'oxyde d'éthylène avec 1 mole de triméthylolpropane.

29. Formulation selon la revendication 28, dans laquelle le composé est un produit de polyaddition de 7 à 20 moles d'oxyde d'éthylène avec 1 mole de triméthylolpropane.

30. Formulation selon l'une quelconque des revendications 1 à 19, dans laquelle, dans le composé de formule (1), m est 4 et le composé est un produit de polyaddition de 4 à 20 moles d'oxyde d'éthylène avec 1 mole de pentaérythritol.

31. Formulation selon la revendication 30, dans laquelle le composé est un produit de polyaddition de 5 à 20 moles d'oxyde d'éthylène avec 1 mole de pentaérythritol.

32. Formulation selon l'une quelconque des revendications précédentes, dans laquelle on utilise un mélange de deux ou plusieurs composés de formule (1) comme composant c) de la formulation.

33. Formulation selon l'une quelconque des revendications précédentes, qui contient également un ou plusieurs agents auxiliaires choisis parmi des stabilisants qui sont efficaces pour régler les propriétés d'écoulement de la formulation, des agents antimousse, des agents alcalins, des assouplissants pour tissus, des agents anti-redéposition, des antioxydants, des adjuvants de détergence auxiliaires et des parfums.

34. Procédé de production d' une formulation selon la revendication 1, comprenant les étapes consistant à mélanger l'agent de blanchiment fluorescent anionique, le solvant et le composé de formule (1), avec tous les agents auxiliaires éventuels, et à homogénéiser le mélange ainsi obtenu.

35. Procédé selon la revendication 34, lequel procédé est conduit à une température comprise dans l' intervalle de 40 à 100°C.

36. Procédé de production d'un détergent, comprenant les étapes consistant à ajouter la quantité requise de la formulation de la revendication 1 à une composition de détergent, puis à homogénéiser le mélange ainsi obtenu.