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(54) Titre : COMPOSITIONS D'AGENTS DE BLANCHIMENT FLUORESCENTS
(54) Title: FLUORESCENT WHITENING AGENT COMPOSITIONS

(57) **Abrégé/Abstract:**

The invention relates to a composition suitable for optically whitening paper or board, wherein the composition contains specified bis-triazinylamino-stilbene fluorescent whitening agents with carboxylic acid and/or sulfonic acid groups. The composition can be used for whitening paper in the pulp, size press or by coating.

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(54) **Title:** FLUORESCENT WHITENING AGENT COMPOSITIONS

(57) **Abstract:** The invention relates to a composition suitable for optically whitening paper or board, wherein the composition contains specified bis- triazinylamino-stilbene fluorescent whitening agents with carboxylic acid and/or sulfonic acid groups. The composition can be used for whitening paper in the pulp, size press or by coating.

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Fluorescent whitening agent compositions

5 The present invention relates to fluorescent whitening agent compositions containing specific bis-triazinylamino-stilbene fluorescent whitening agents with carboxylic acid and/or sulfonic acid groups for whitening paper or board.

10 It is well known that the whiteness of paper and board can be improved by the addition of fluorescent whitening agents (FWAs). The most important fluorescent whitening agents used in the paper and board industry are anilino-substituted bistriazinyl derivatives of 4,4'-diaminostilbene-2,2'-disulfonic acid (flavonic acid). From these fluorescent whitening
15 agents disulfo-, tetrasulfo- and hexasulfo-types are known. The disulfo-type fluorescent whitening agents with no sulfonic acid groups at the aniline rings have a low solubility in water and a high affinity for cellulose fibres. They are especially suitable for use at the wet-end of paper making process. The hexasulfo-type fluorescent whitening agents with
20 two sulfonic acid groups at each aniline ring have a high solubility in water and a low affinity for cellulose fibres. They are more specialty products when very high whiteness is desired. The tetrasulfo-type fluorescent whitening agents with one sulfonic acid group at each aniline ring exhibit a behaviour between the disulfo- and hexasulfo-type fluorescent
25 whitening agents and are most commonly used for whitening paper or board.

For ease of handling and metering, the paper and board industry demands fluorescent whitening agents to be supplied in a liquid form, preferably
30 as a concentrated aqueous solution, which should be stable to prolonged storage over a wide temperature range. Due to the low solubility of disulfo-type fluorescent whitening agents in water, currently solubilising aux-

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iliaries such as urea, triethanolamine or diethylene glycol are added in amounts of up to 30% to provide storage stability for concentrated aqueous solutions of disulfo-type fluorescent whitening agents. These solubilising agents have no affinity to cellulose and contaminate the effluent from the paper mill, thus being undesired. EP-A-1 752 453 teaches storage stable solutions of disulfo-type fluorescent whitening agents which contain specific counter-ions for the sulfonic acid groups, which counter-ions are derived from specific aminoalkanols. WO 02/055646 A1 discloses concentrated aqueous solutions containing a mixture of two specific disulfo-type fluorescent whitening agents. Alternatively, slurries or dispersions of disulfo-type fluorescent whitening agents in water are known, e.g. from EP 0 884 312 B1. However, in order to enable the metering of homogenous preparations into the papermaking process, usually stirring is required.

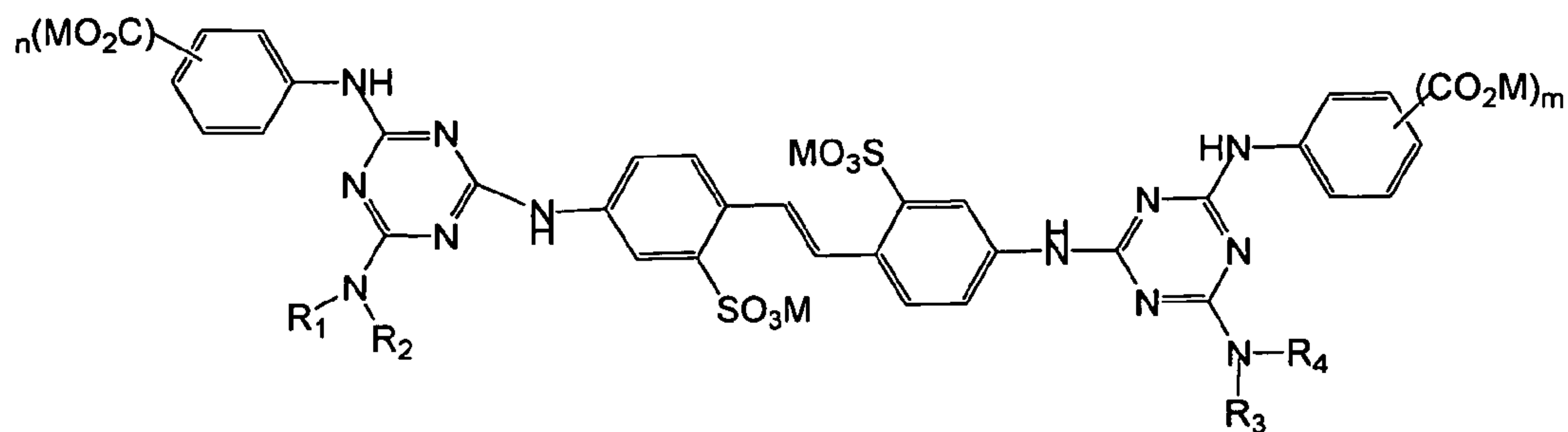
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Surprisingly, it has been found that problems of the prior art can be overcome by using mixtures or combinations of specific bis-triazinylaminostilbene fluorescent whitening agents having carboxylic acid and/or sulfonic acid groups at the terminal phenyl rings. These mixtures or combinations when used for whitening paper or board yield paper or board of improved whiteness. Further, the production process of those fluorescent whitening agents is more cost-effective, compared to that of the commonly used disulfo-type fluorescent whitening agents, since it dispenses with laborious isolation and filtration steps. Moreover, the disulfo-type fluorescent whitening agents thereof enable stable concentrated aqueous preparations or solutions to be formed, without addition of solubilising auxiliaries.

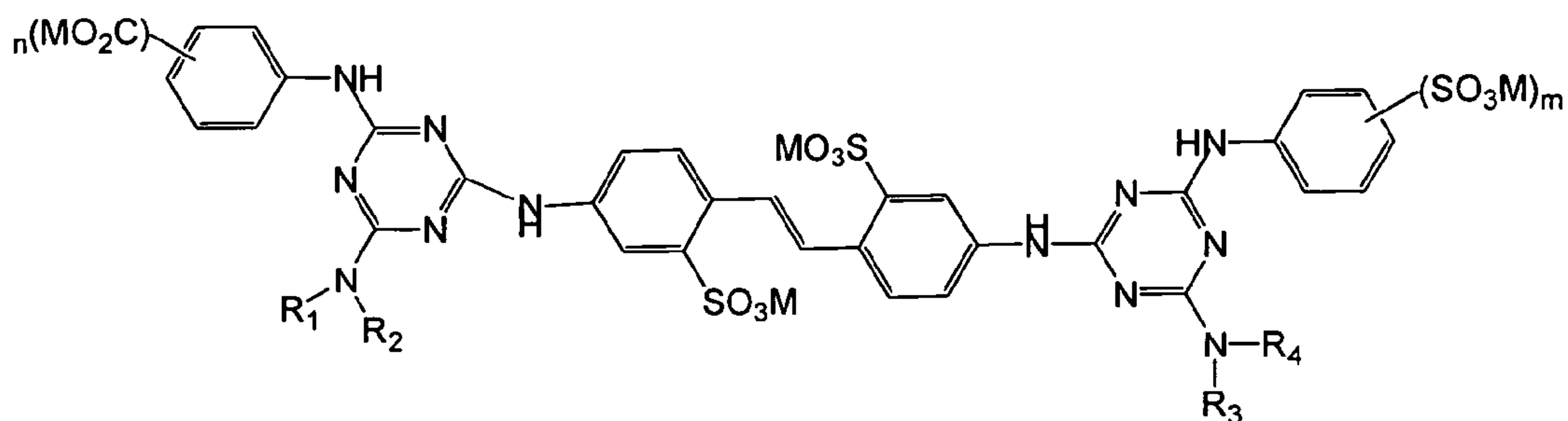
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Therefore, the present invention relates to fluorescent whitening agent (FWA) compositions suitable for optically whitening paper or board, wherein the composition contains at least two fluorescent whitening agents selected from the fluorescent whitening agents of the formula (1), formula (2) and formula (3)

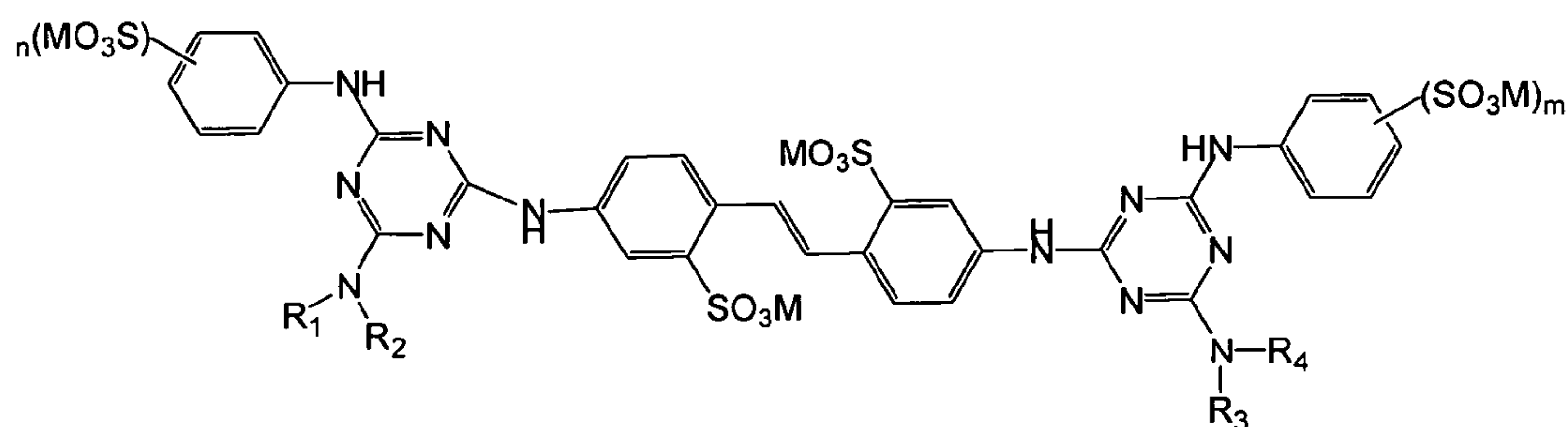
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(1)



(2)



(3)

wherein

n and m are, independently of each other, 0, 1, or 2, with the proviso that
 5 n and m are not both 0;

R₁, R₂, R₃ and R₄ represent, independently of each other, hydrogen,
 cyano, C₁-C₄ alkyl, C₂-C₄ hydroxyalkyl, C₂-C₄ cyanoalkyl or C₁-C₄
 alkoxyalkyl, or the residue of an amino acid from which a hydrogen atom
 has been abstracted from the amino group; or R₁ and R₂, or R₃ and R₄,
 10 independently of each other, together with N atom form morpholine, pi-
 peridine or pyrrolidine ring; or -(CH₂)_l-SO₃M, where l is 1, 2 or 3; or -
 (CH₂)_i-COOR, -(CH₂)_i-CONHR, -(CH₂)_i-COR, where i is an integer from
 1 to 4, R is C₁-C₃ alkyl or has the same meaning as M;

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M represents hydrogen, or one equivalent of a cation, in particular Li, Na, K, Ca, Mg, ammonium, or ammonium which is mono-, di-, tri- or tetra-substituted by C₁-C₄ alkyl or C₂-C₄ hydroxyalkyl.

5 The invention also refers to the fluorescent whitening agent of formula (2) and the use of that fluorescent whitening agent and of the fluorescent whitening agent (FWA) compositions for optically whitening paper or board, e.g. in the pulp. Further, the invention relates to a process for optically whitening paper and to paper obtainable by that process. Preferred
10 embodiments of the invention are described in the description hereinafter, and the claims.

In a preferred embodiment of the invention, the fluorescent whitening agent composition is a wet-end composition, and the process is a process
15 for whitening paper, wherein a pulp or pulp suspension is brought into contact with said composition. In another preferred embodiment, the fluorescent whitening agent composition is used for preparing a size press liquor or a coating composition.

20 According to the invention the composition or mixture contains at least two of the bis-triazinylamino-stilbene compounds of the above defined formulae (1), (2) and (3). In the context of the invention, in the formulae (1), (2) and (3) the alkyl group can be linear or branched, and the possible substituents of the alkyl group, which are alkoxy, cyano, and/or hydroxyl groups, can be attached at any position of the alkyl chain. In the
25 present invention, C₁-C₄ alkoxyalkyl means C₁-C₄ alkyl substituted with C₁-C₄ alkoxy. In a preferred embodiment, R₁, R₂, R₃ and R₄ represent, independently of each other, C₂-C₄ hydroxyalkyl, C₁-C₄ alkoxyalkyl, or C₁-C₄ alkyl, preferably C₂-C₄ hydroxyalkyl or C₁-C₄ alkoxyalkyl, in particular hydroxyethyl or hydroxyisopropyl. Most preferably, R₁, R₂, R₃
30 and R₄ represent hydroxyethyl.

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In a preferred embodiment, n and m are, independently of each other, 1 or 2. In another most preferred embodiment, n and m are both 1. Alternatively, preferably n is 1 and m is 2, or n is 2 and m is 1. The groups -SO₃M and -CO₂M can be at any position of the terminal phenyl rings.

5 Preferred compounds in the composition are those where the groups -SO₃M and -CO₂M are, independently of each other, in *ortho*- or *para*-position, preferably in *para*-position. If n and/or m is/are 2, the group -SO₃M and/or the group -CO₂M are preferably in *ortho*- and *para*-position. A preferred compound is a compound of formula (2), wherein n

10 and m are both 1, the group -SO₃M is in *meta*- or *ortho*-position, and the group -CO₂M is in *para*-position. Another preferred compound is a compound of formula (2), wherein n and m are both 1, the group -SO₃M is in *para*-position, and the group -CO₂M is in *ortho*-position.

15 Preferred embodiments of M are hydrogen, Na, K, Ca, Mg, in particular M is Na, K or hydrogen, most preferred is Na. The fluorescent whitening agents are used as free acids or as salts thereof, preferably alkali metal salts.

20 The fluorescent whitening agents of formulae (1), (2) and (3) and the mixtures thereof can be prepared by known procedures. Generally, the compounds are prepared by reacting cyanuric chloride with 4,4'-diaminostilbene-2,2'-disulfonic acid or a salt thereof, aminobenzoic acid and/or aminosulfonic acid and/or the corresponding aniline compound

25 with two carboxy acid or sulfonic acid groups, and substituted aliphatic amines or heterocyclic compounds. The ratio of aminobenzoic acid, aminosulfonic acid and/or aniline compound with two carboxy acid or sulfonic acid groups can be selected such that a desired ratio of the fluorescent whitening agents of formulae (1), (2) and/or (3) is obtained. PL patent

30 61710 discloses the preparation of some specific fluorescent whitening agents with one carboxylic acid group in p-position of each aniline ring without sulfonic acid group. The purification of the fluorescent whitening agents of formulae (1), (2) and (3) is easier and thus more

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cost-effective than for commonly used disulfo-type fluorescent whitening agents, since isolation and formulation steps can be avoided. The purification could be carried out by, for example, membrane filtration.

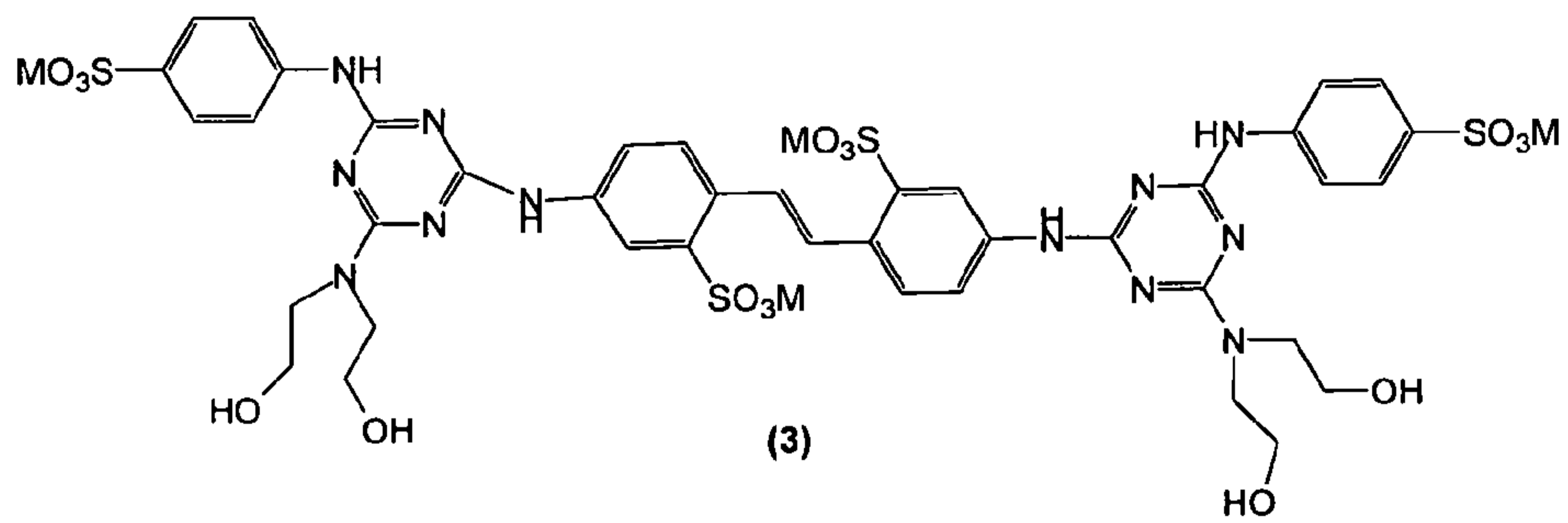
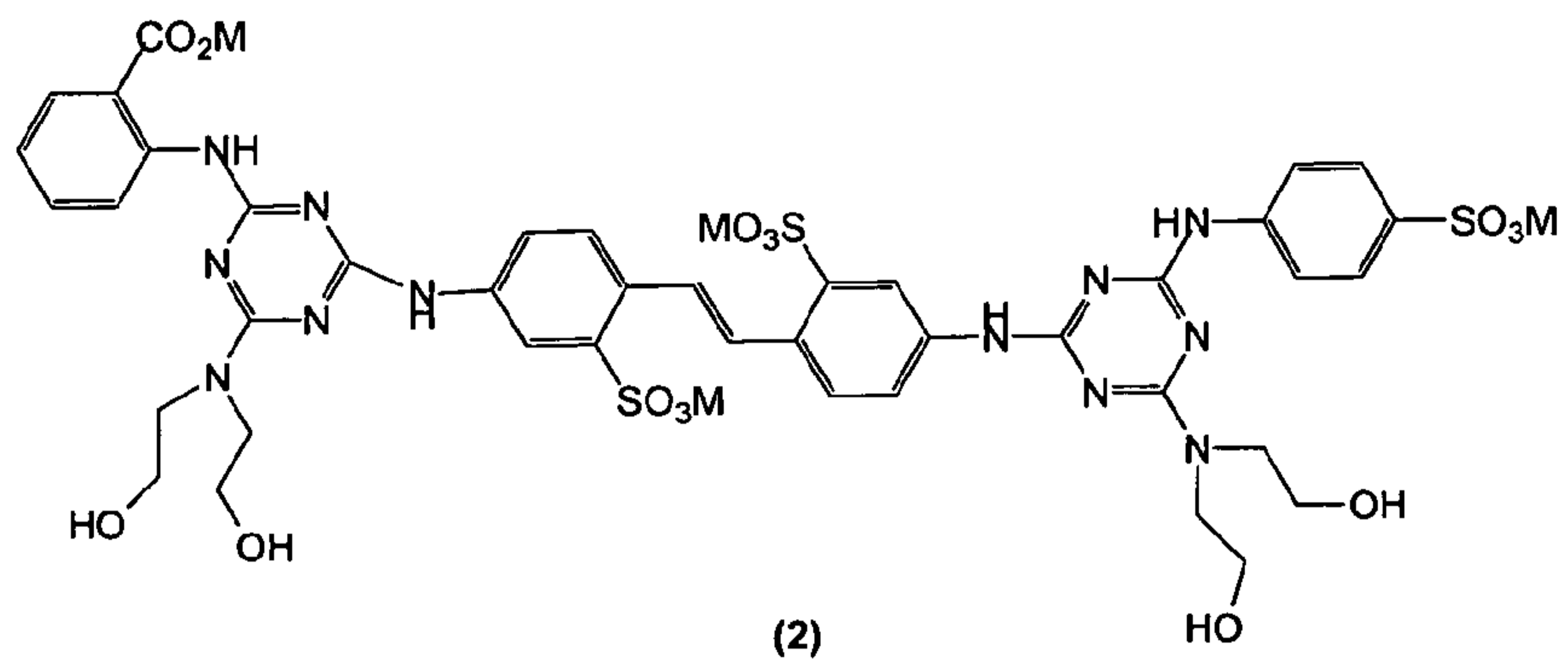
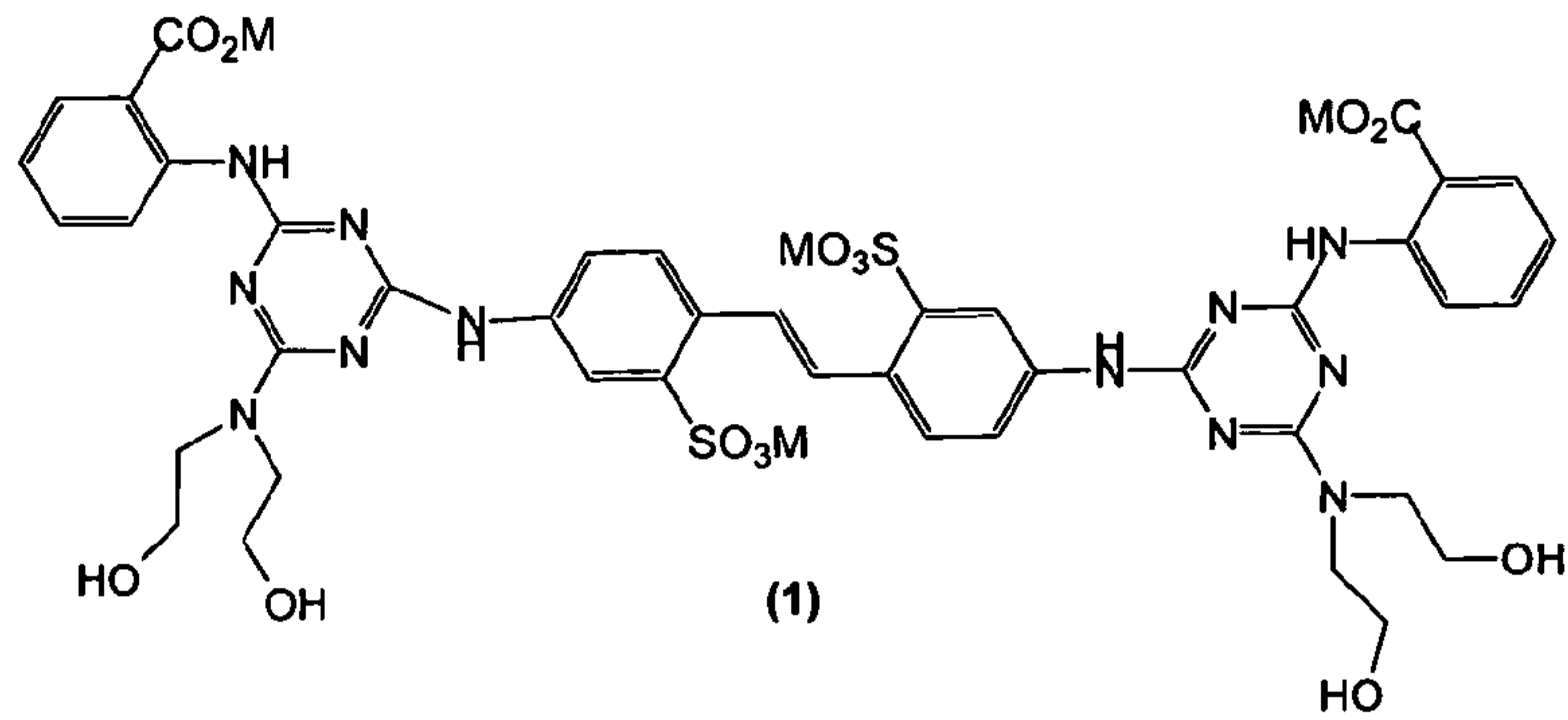
5 The composition of the fluorescent whitening agents of formulae (1), (2) and/or (3) can be produced in form of a mixture with the desired ratio of the fluorescent whitening agents of formulae (1), (2) and/or (3). Alternatively, the composition can be produced by preparing separately the fluorescent whitening agents of formulae (1), (2) and/or (3) by the methods
10 known in the art and as described above, and then blending or mixing together in the desired ratio after their preparation.

The composition of the invention contains at least two, in particular two or three, fluorescent whitening agents selected from the fluorescent whitening agents of formulae (1), (2) and (3). In a more preferred embodiment, the composition comprises the fluorescent whitening agents of the
15 formulae (1) and (2). In a further more preferred embodiment, the composition comprises the fluorescent whitening agents of the formulae (2) and (3). In another preferred embodiment, the composition comprises the fluorescent whitening agents of the formulae (1) and (3). In a most preferred embodiment, the composition contains at least two fluorescent whitening agents selected from the fluorescent whitening agents of formulae (1), (2) and (3), and one of those fluorescent whitening agents is the compound of formula (2). In another most preferred embodiment, the
20 composition comprises the fluorescent whitening agents of the formulae (1), (2) and (3). The composition can also contain one or more of each of a fluorescent whitening agent of the formulae (1), (2) and/or (3). In addition, the composition can contain one or more known bis-triazinylamino-stilbene or distyryl-biphenyl based fluorescent whitening agents.

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Preferred compositions of the invention are shown below in the Embodiments 1 and 2, wherein M is as described above.

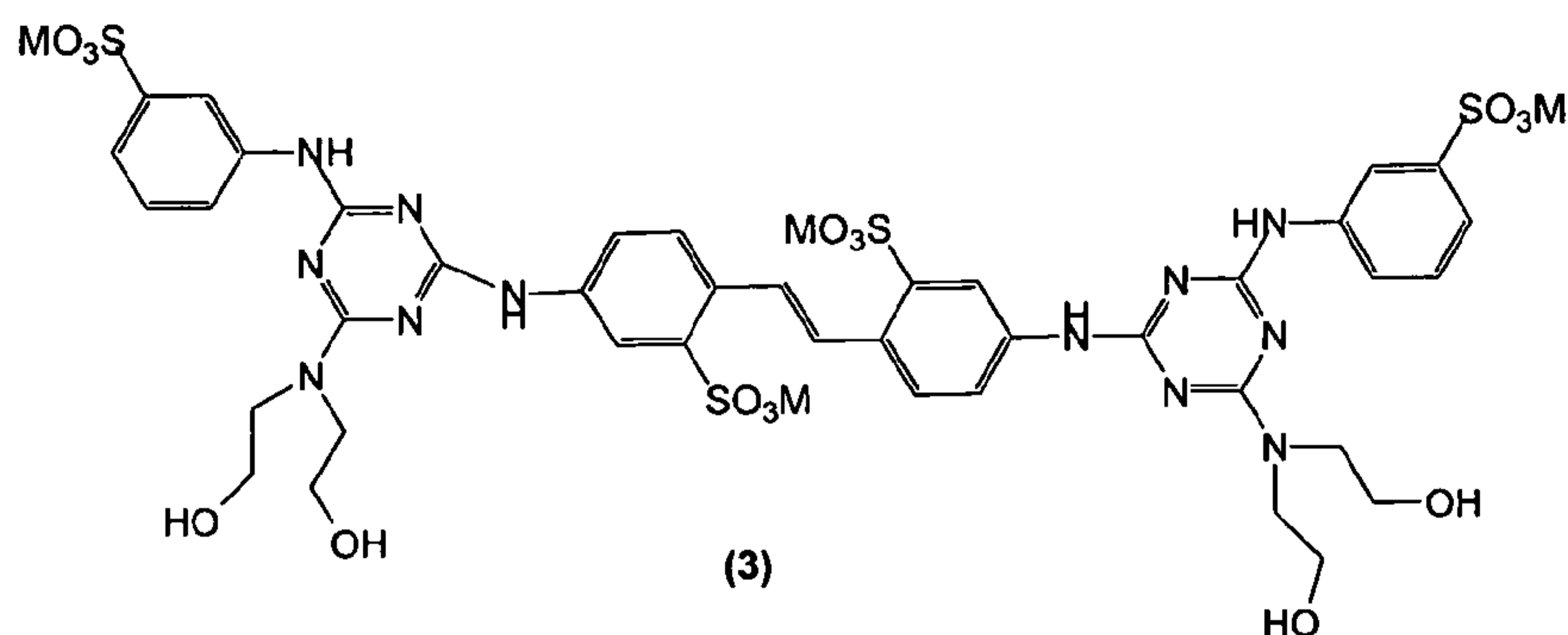
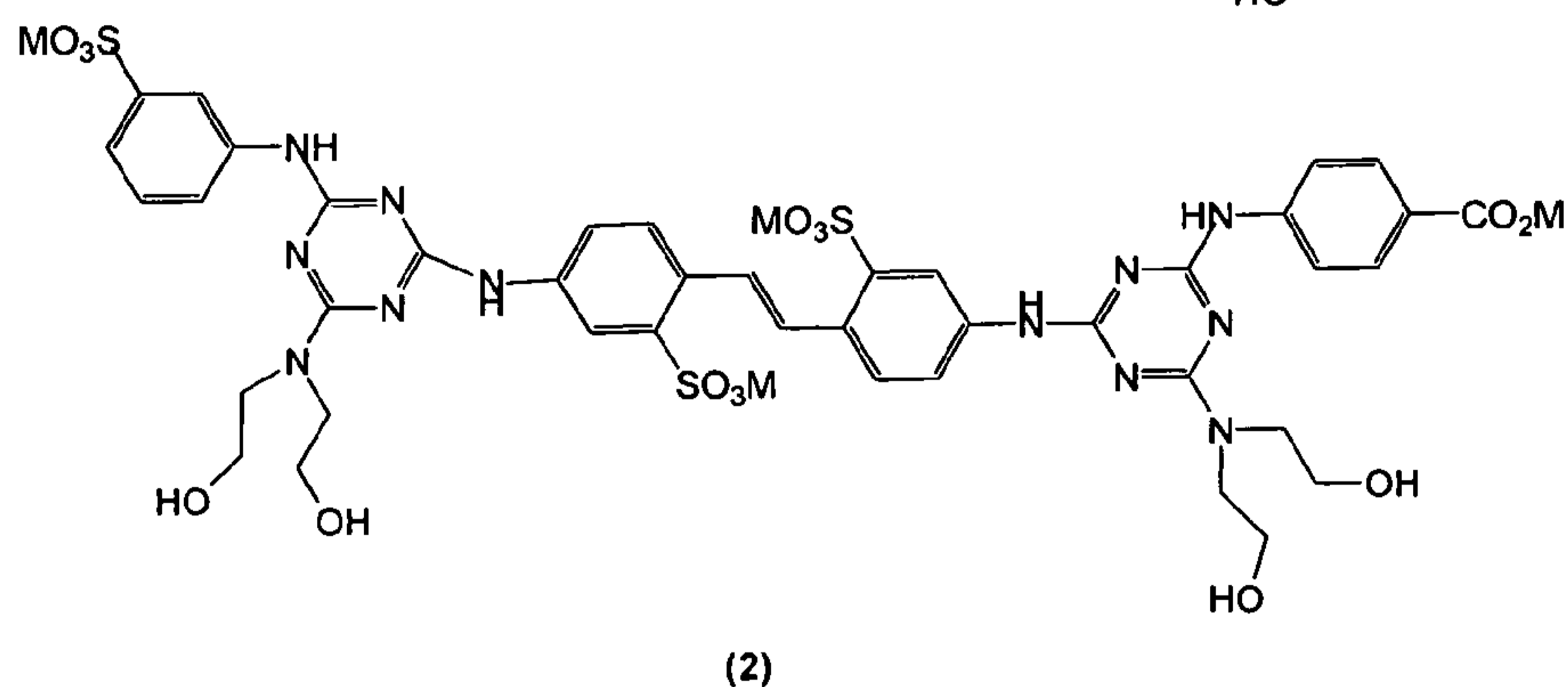
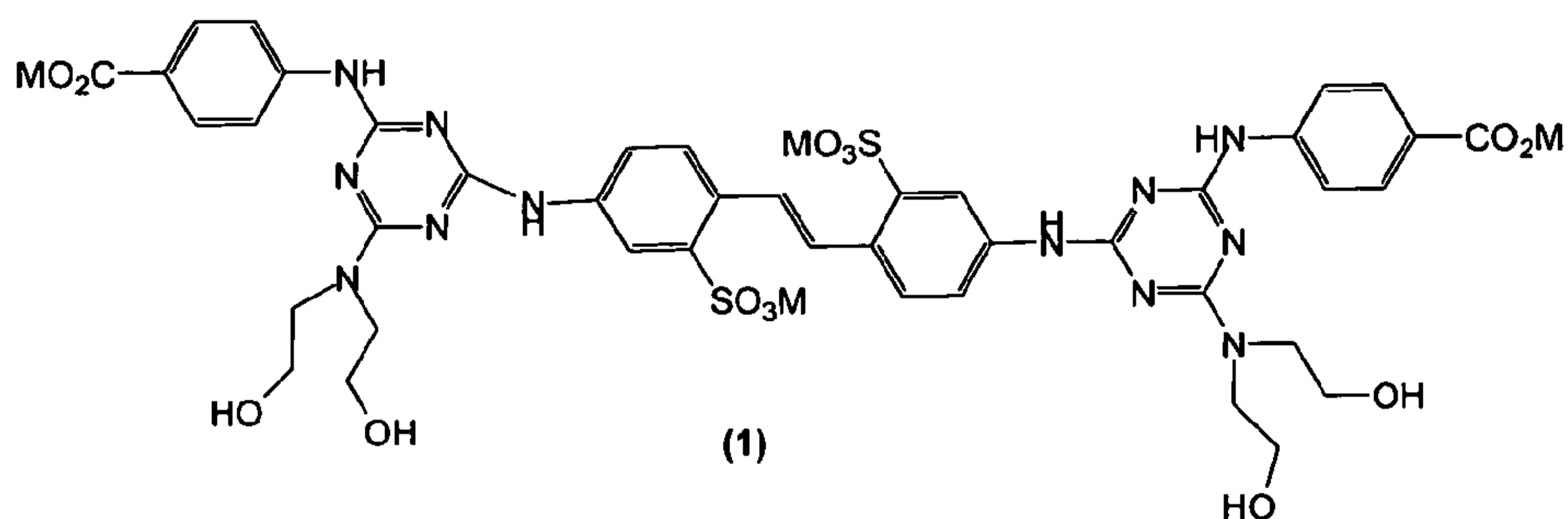
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Embodiment 1

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Embodiment 2



5 The amounts of the fluorescent whitening agents present in the composition depend on the number of fluorescent whitening agents present and which fluorescent whitening agents are present. According to the invention the composition contains preferably at least one fluorescent whitening agent of formula (1) in an amount of 0 to 99 weight-%, preferably 2 to 90 weight-%, most preferably 5 to 80 weight-%; at least one fluorescent whitening agent of formula (2) in an amount of 0 to 99 weight-%, preferably 2 to 90 weight-%, most preferably 5 to 80 weight-%; and at least one fluorescent whitening agent of formula (3) in an amount of 0 to

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99 weight-%, preferably 2 to 90 weight-%, most preferably 5 to 80 weight-%; in each case based on 100 weight-% of the total amount of the present fluorescent whitening agents of the formulae (1), (2) and/or (3). In further embodiments the composition contains preferably at least one fluorescent whitening agent of formula (1) in an amount of 10 to 80 weight-%, in particular 20 to 70 weight-%, at least one fluorescent whitening agent of formula (2) in an amount of 10 to 60 weight-%, in particular 10 to 50 weight-%, and at least one fluorescent whitening agent of formula (3) in an amount of 10 to 80 weight-%, in particular 20 to 70 weight-%, in each case based on 100 weight-% of the total amount of the present fluorescent whitening agents of the formulae (1), (2) and/or (3).

The fluorescent whitening agent composition can be present in liquid form, in particular as a solution, or in form of a powder. In preferred embodiments, the compositions contain water, in particular in an amount of 20 to 90 weight-%, based on 100 weight-% of the total amount of the fluorescent whitening agents and water. Such aqueous fluorescent whitening agent compositions or mixtures are present in liquid form, in particular as a solution. Preferably, those are free of crystalline whitener particles, in particular their hydrate forms.

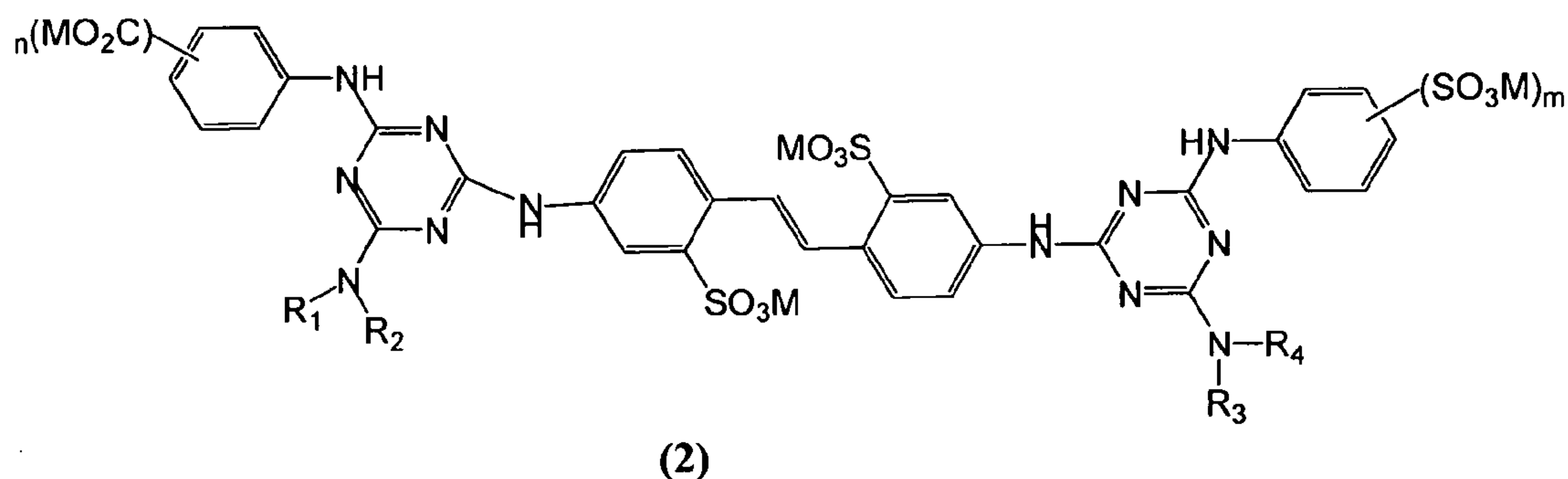
The fluorescent whitening agent compositions, in particular the aqueous compositions, may contain a small amount of auxiliaries. This might be particularly relevant for fluorescent whitening agent compositions containing disulfo-type fluorescent whitening agents and/or if used in cold regions to enhance preparations' cold stability. In a preferred embodiment, the aqueous fluorescent whitening agent composition contains less than 30 % by weight, preferably less than 20 % by weight, more preferably less than 15 % by weight, in particular less than 10 % by weight of components other than the fluorescent whitening agents and water. For example, formulation auxiliaries, such as standardizing agents, surface-active compositions, dispersants, antifoams, thickeners, preservatives, antifreezes, complexing agents, and/or electrolytes may be used. Howev-

er, for ecological reasons, the aqueous fluorescent whitening agent preparation preferably contains only very small amounts of other components, e.g. organic additives or auxiliaries, particularly altogether less than 3 % by weight, in particular less than 1 % by weight, based on 100 % by weight of the aqueous fluorescent whitening agent composition. Particularly preferably, the composition contains no organic co-solvents, and/or urea. In a further preferred embodiment, the composition consists or consists essentially of the fluorescent whitening agents and water.

10 The aqueous fluorescent whitening agent composition is preferably prepared by introducing the fluorescent whitening agents of formulae (1), (2) and/or (3) or their mixture in the desired ratio in form of a powder or a concentrated solution thereof into water. Any auxiliaries can optionally be added during or after preparation of the mixture.

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The invention further refers to the fluorescent whitening agent or compound of formula (2)



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wherein n , m , M , R_1 , R_2 , R_3 and R_4 are as defined above. This compound is not known from the state of the art. It can be prepared by methods known in the art or as described above. Preferred embodiments for R_1 , R_2 , R_3 , R_4 , M , n and m are the same as described above for the composition according to the invention. The compound of formula (2) can be used as described below for the composition according to the invention.

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The composition according to the invention can be used for whitening paper or board, preferably in the pulp or pulp suspension (stock), in particular in the wet-end. Alternatively, the composition can be used for whitening paper at the surface. In wet-end applications the compositions can be added at any point of the pulp circuit, e.g. chests or pipes, before sheet forming. Depending on the papermaking process used, the compositions can be added to the papermaking process also in diluted form, wherein the composition has been diluted to a desired concentration by addition of water and/or auxiliaries. In a preferred embodiment, the, preferably aqueous, fluorescent whitening agent composition is introduced, optionally after dilution with water, to the pulp or pulp suspension. The compositions can be added continuously or discontinuously. The application is beneficial for both wood-containing pulps and wood-free pulps, in particular for wood-containing pulps. In surface application, the compositions can be used for preparing size press liquors, coating compositions or coating slips.

The aqueous fluorescent whitening agent compositions exhibit high storage stability and ease of application. Simultaneously, they provide high affinity (substantivity) to fibres and high whitening performance.

The invention also refers to a process for whitening paper, which comprises providing a pulp or pulp suspension; adding a fluorescent whitening agent composition as described above to the pulp or pulp suspension, preferably in an amount of 0.01 to 5 % by weight, more preferably 0.02 to 2 % by weight, based on 100 % by weight of dry pulp; producing a paper sheet from the pulp; and drying the sheet. In one embodiment of this process, the composition is added, after dilution with water and/or auxiliaries, in particular dilution with water, to the pulp or pulp suspension. In another preferred embodiment of a process for whitening paper, a cellulose sheet is brought into contact with a fluorescent whitening agent

composition as described above. The described processes for whitening paper also can be carried out by using the compound of formula (2).

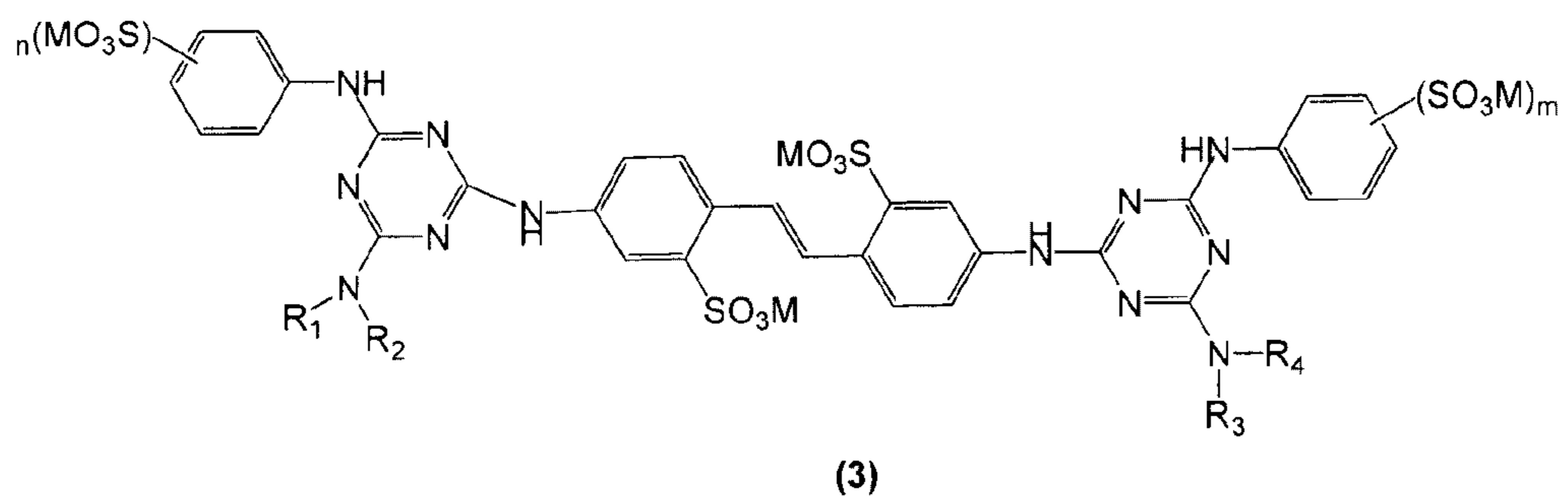
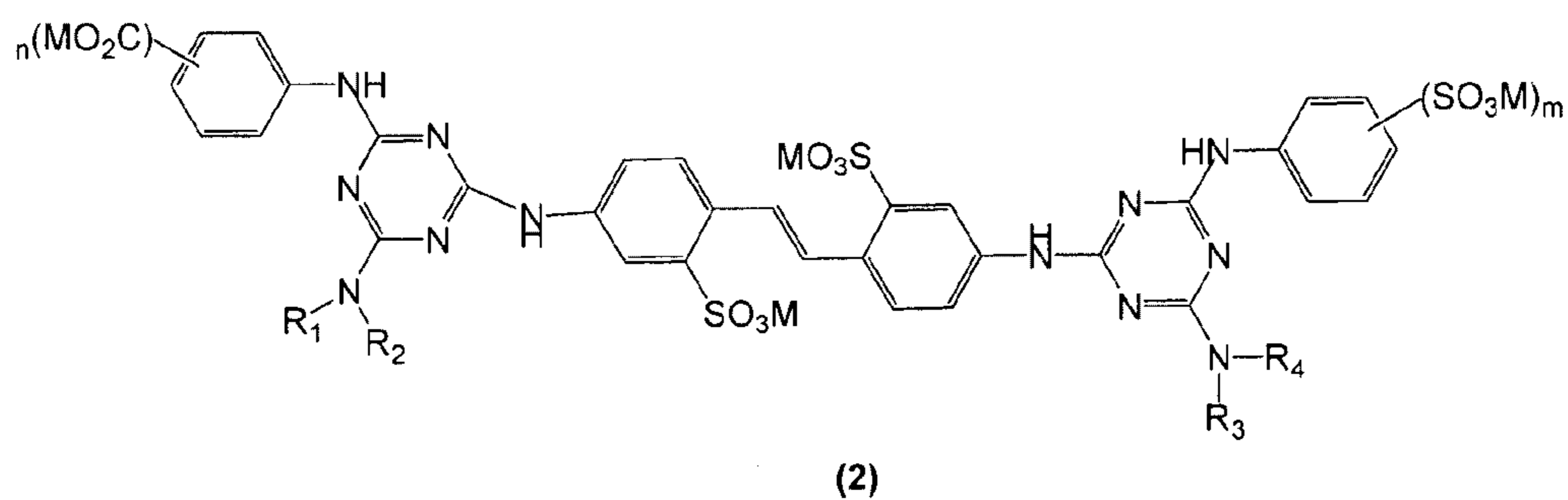
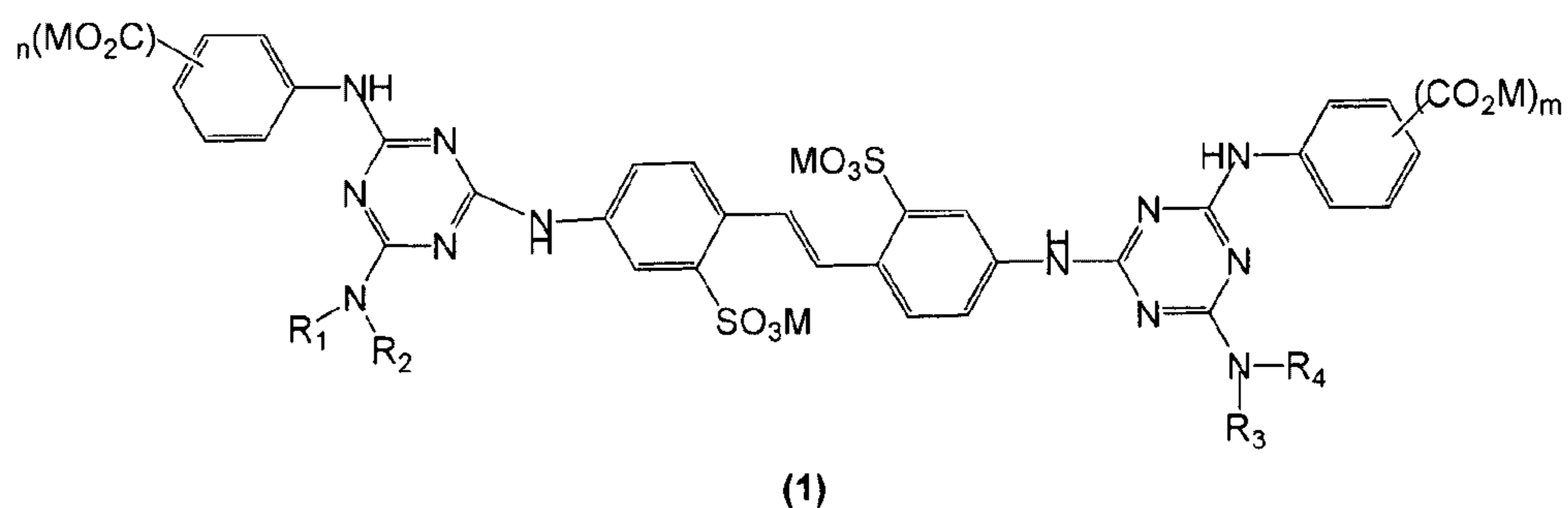
5 The whiteness of the papers produced can be characterized by the CIE whiteness. Different fluorescent whitening agents can be compared to each other with respect to the saturation behaviour when determined according to CIE whiteness. In other words, if a larger amount of fluorescent whitening agent is used and no further increase in whiteness is found, there is saturation behaviour and there may even be adverse effects on the whiteness when using higher amounts. The effect of saturation is also referred to as greening. The greening limit, i.e. the point at which increasing amounts of fluorescent whitening agent used results in virtually no further increase in whiteness, can be derived, for example, from the a^*-b^* diagram, where a^* and b^* are the colour coordinates in the CIE- $L^*a^*b^*$ system.

Paper produced by using the fluorescent whitening agent compositions according to the invention exhibits higher whiteness compared to paper produced using the typically used tetrasulfo-type fluorescent whitening agents. In particular it was found that when aminobenzoic acid (different isomers) is blended with aminosulfonic acid (different isomers) in various ratios in the production process, the produced mixtures have better affinity to cellulose fibres than the corresponding tetrasulfo-type fluorescent whitening agents. Moreover, the performance of the obtained mixtures is superior to that of the corresponding tetrasulfo-type fluorescent whitening agents both in stock and surface applications, thus exhibiting an unexpected synergistic effect.

CLAIMS

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1. A composition suitable for optically whitening paper or board, wherein the composition contains at least two fluorescent whitening agents of formula (1), formula (2) or formula (3)



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wherein

n and m are, independently of each other, 0, 1, or 2, with the proviso that n and m are not both 0;

R₁, R₂, R₃ and R₄ represent, independently of each other, hydrogen, cyano, C₁-C₄ alkyl, C₂-C₄ hydroxyalkyl, C₂-C₄ cyanoalkyl or C₁-C₄ alkoxyalkyl, or the residue of an amino acid from which a hydrogen atom has been abstracted from the amino group; or R₁ and R₂, or R₃ and R₄, independently of each other, together with N atom form morpholine, piperidine or pyrrolidine ring; or -(CH₂)_i-SO₃M, where i is 1, 2 or 3; or -(CH₂)_i-COOR, -(CH₂)_i-CONHR, -(CH₂)_i-COR, where i is an integer from 1 to 4, R is C₁-C₃ alkyl or has the same meaning as M;

M represents hydrogen, or one equivalent of a cation.

2. The composition according to claim 1, wherein M represents Li, Na, K, Ca, Mg, ammonium, or ammonium which is mono-, di-, tri- or tetra-substituted by C₁-C₄ alkyl or C₂-C₄ hydroxyalkyl.
3. The composition according to claim 1 or 2, wherein n and m are 1.
4. The composition according to any one of claims 1 to 3, wherein the groups -CO₂M and -SO₃M are, independently of each other, in *ortho*- and/or *para*-position.
5. The composition according to any one of claims 1 to 4, wherein R₁, R₂, R₃ and R₄ represent, independently of each other, C₂-C₄ hydroxyalkyl, or C₁-C₄ alkoxyalkyl.
6. The composition according to any one of claims 1 to 5, wherein the composition contains a fluorescent whitening agent of the formula (2).

7. The composition according to claim 6, wherein in the compound of the formula (2) n and m are 1, the group $-\text{CO}_2\text{M}$ is in *ortho*-position, and the group $-\text{SO}_3\text{M}$ is in *para*-position.

5 8. The composition according to any one of claims 1 to 7, wherein the composition contains the fluorescent whitening agents of formula (1), formula (2) and formula (3).

9. The composition according to any one of claims 1 to 8, wherein the
10 composition contains the fluorescent whitening agent of formula (1) in an amount of 20 to 95 weight-%, the fluorescent whitening agent of formula (2) in an amount of 0.1 to 60 weight-%, and the fluorescent whitening agent of formula (3) in an amount of 0.1 to 30 weight-%, in each case based on 100 weight-% of the total amount of the present fluorescent whitening agents of the formulae
15 (1), (2) and (3).

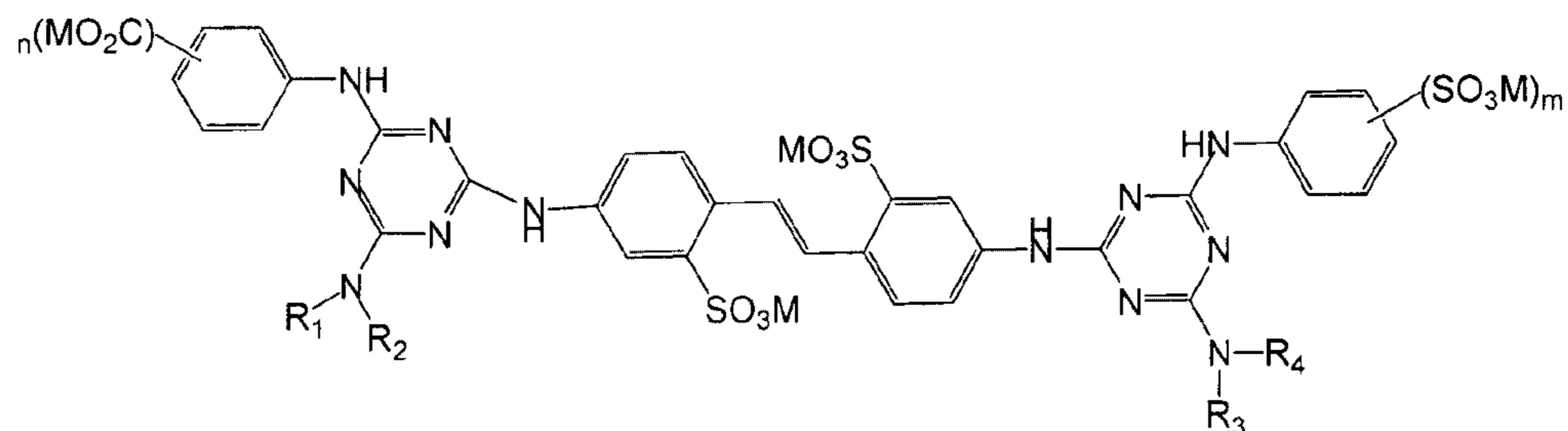
10. The composition according to any one of claims 1 to 9, wherein the composition is an aqueous preparation.

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11. The composition according to claim 10, wherein the composition is a size press liquor, a coating composition or a coating slip.

12. A compound of formula (2)

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(2)

wherein n , m , M , R_1 , R_2 , R_3 , and R_4 are as defined in claim 1.

13. The compound according to claim 12, wherein the compound is as defined in any one of claims 2 to 5 and 7.
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14. Use of a composition according to any one of claims 1 to 11 or a compound according to claim 12 or 13 for optically whitening paper or board.
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15. Use of claim 14, for optically whitening paper or board in a pulp or pulp suspension.
16. A process for optically whitening paper, wherein a pulp or pulp suspension is brought into contact with a composition as defined in any one of claims 1 to 11 or a compound as defined in claim 12 or 13.
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17. A process for optically whitening paper, wherein a cellulose sheet is brought into contact with a composition as defined in any one of claims 1 to 11 or a compound as defined in claim 12 or 13.
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18. A paper obtained by the process according to claim 16 or 17.