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(54) Titre : SYSTEMES PUR 2K AQUEUX

(54) Title: AQUEOUS 2K PUR SYSTEMS

(57) **Abrégé/Abstract:**

The invention relates to aqueous 2K PUR systems consisting of specially modified vinyl polymer polyol dispersions and polyisocyanates, to a method for producing aqueous 2K PUR systems of this type with improved resistance to aqueous chemicals and to their use as 2K PUR coatings for coating any given substrates.



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(54) Title: AQUEOUS 2K PUR SYSTEMS

(54) Bezeichnung: WÄSSRIGE 2-K-PUR-SYSTEME

(57) Abstract: The invention relates to aqueous 2K PUR systems consisting of specially modified vinyl polymer polyol dispersions and polyisocyanates, to a method for producing aqueous 2K PUR systems of this type with improved resistance to aqueous chemicals and to their use as 2K PUR coatings for coating any given substrates.

(57) Zusammenfassung: Die Erfindung betrifft wässrige 2K-PUR-Systeme aus speziell modifizierten Vinylpolymerpolyoldisersionen und Polyisocyanaten, ein Verfahren zur Herstellung solcher wässriger 2 K-PUR-Systeme mit verbesserter Stabilität gegen wässrige Chemikalien und deren Verwendung als 2K-PUR-Lacke bei der Lackierung von beliebigen Substraten.

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Aqueous 2-K PU systems

The invention relates to aqueous 2K PU systems comprising specially modified vinyl polymer polyol dispersions and polyisocyanates, to a process for preparing such
5 aqueous 2 K PU systems with improved stability toward aqueous chemicals, and to their use as 2K PU coating materials in the coating of any desired substrates such as, for example, wood, metal, plastic.

EP-A 0 358 979, EP-A 0 496 210, and EP-A 0 557 844, and other patent literature,
10 describe the preparation of aqueous 2 K PU dispersions for which both what are termed secondary dispersions and what are termed primary dispersions are used as polyol components, together with suitable polyisocyanates. The term "secondary dispersions" comprehends those aqueous dispersions which are first polymerized in a homogeneous organic medium and then redispersed in an aqueous medium with
15 neutralization, generally without the addition of external emulsifiers.

The term "primary dispersions" comprehends polyol dispersions which are prepared directly in aqueous phase by the technique of emulsion polymerization. They generally do contain external emulsifiers; these emulsifiers bring about electrostatic
20 or steric stabilization.

On account of their relatively low molar weights M_n usually below 5000 g/mol and M_w usually below 30000 g/mol, and in view of their balanced hydrophilic/lipophilic character, the secondary dispersions are outstandingly suitable for stably emulsifying
25 not only hydrophilicized but also hydrophobic, non-self-dispersing polyisocyanates in an aqueous medium and at the same time for functioning as a reactive component (cf. EP-A 0 358 979). Owing to the relatively low molar weights of the polyol secondary dispersions, however, such aqueous 2 K PU systems usually lack adequate suitability as regards their physical initial drying on wood substrates. The drying
30 times (dry to touch and dust-free dried) are too long and are unsuitable for industrial coating.

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Better suited to use as rapidly physically drying aqueous 2 K PU systems are generally those comprising primary dispersions as polyol components. These generally have molar weight figures of M_n well above 5000 g/mol and M_w figures of usually above 30000 g/mol. These primary dispersions can generally be combined
5 only with (partly) hydrophilicized polyisocyanates; this produces the possibility of relatively uncomplicated dispersion with relatively simple stirring units or by hand and glass rod (cf. EP-A 0 557 844). When, for example, polyisocyanates hydrophilicized nonionically with polyether groups are used as crosslinkers and such coatings are applied, for example, to wood substrates, drying thereof produces
10 coating films which, after exposure to coloring substances such as red wine, coffee, tea, mustard, etc. for example, retain permanent discolorations which, particularly on pieces of furniture, may result in permanent marking. Moreover, such coating systems display instances of slight film clouding. If, alternatively, polyisocyanates containing external or internal anionic groups are used, there is admittedly an
15 improvement in the marking resistance, but the level of resistance which can be achieved is still not sufficient. Furthermore, there is a deterioration in the compatibility of the components with one another in such systems, and the clouding of the films is increased.

20 The technical problem was therefore to provide polyol dispersions, as components for 2K PU systems, which together with polyisocyanate components containing anionic groups form marking-resistant and clouding-free coating films.

It has been possible to solve this problem by combining aqueous polyol dispersions,
25 preferably of the primary dispersion type prepared in the presence of special nonionic surfactants, with polyisocyanates possessing both a hydrophobic character and a nonionically or anionically hydrophilicized character. This fact was all the more surprising because, as already mentioned earlier on above, polyol dispersions without such special nonionic surfactants lead, in combination with nonionically
30 hydrophilicized polyisocyanates, to coating materials lacking adequate stabilities toward aqueous coloring liquids, and it must have been expected that, following further addition of nonionic surfactants to the 2K PU systems, resistance properties markedly poorer still would result.

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The invention therefore provides aqueous 2K PU coating materials having improved resistance to aqueous chemicals, characterized in that the aqueous 2K PU systems used as binders are composed of

- 5 a) polyol dispersions of the vinyl polymer polyol type,
- b) polyisocyanates, and
- c) customary additives for water-based coating technology,

10

where

the polyol dispersions a)

- 15 have hydroxyl numbers of between 10 and 264 mg KOH/g resin solids, acid numbers (calculated based on the sum of the neutralized and nonneutralized acid groups) of between 0 to 55 mg KOH/g resin solids, molecular weights M_n of at least 5000 g/mol or M_w of at least 30000 g/mol, glass transition temperatures of at least 20°C, and average particle diameters of not more than 300 nm, and have been prepared in the
- 20 presence of

- a1) from 0.1 to 10.0% by weight, based on the sum of the solids contents of polymer polyol and polyisocyanate, of nonionic surfactants of the polyether type,

25

the polyisocyanates b)

- possess viscosities of not more than 12000 mPa*s and either a nonhydrophilicized or nonionically and/or anionically hydrophilicized character, and where component b)
- 30 is added to component a) in an amount which corresponds to an equivalents ratio of isocyanate groups of component b) to hydroxyl groups of component a) of from 0.2:1 to 5:1.

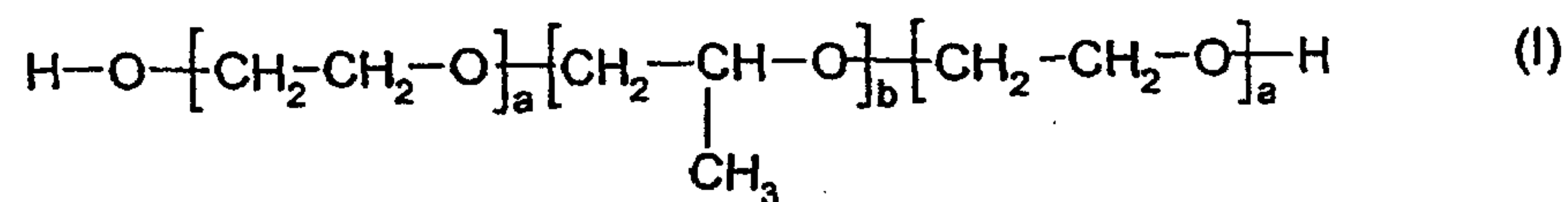
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The invention also provides a process for preparing such aqueous 2K PU systems.

Preferably, component a) is prepared in the presence of

- 5 a1) from 1.0 to 8.0% by weight of the nonionic surfactant of the polyether type.

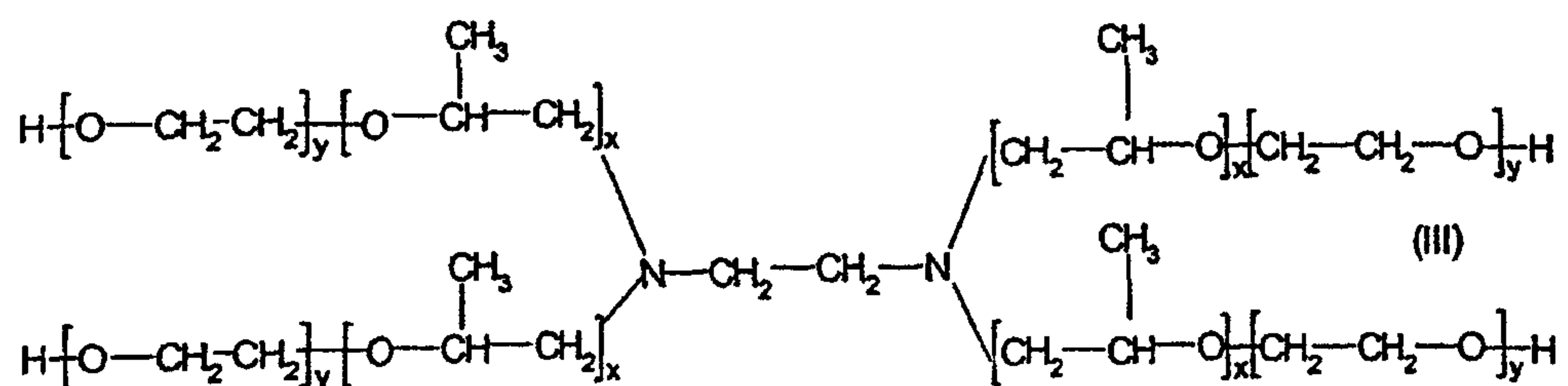
As surfactant components a1) it is preferred to prepare and/or use block polymers of ethylene oxide and propylene oxide in accordance with formula (I)



10

and/or block and random ethylene oxide/propylene oxide copolymers based on fatty alcohol (II),

- 15 and/or block polymers of the type according to formula (III), which can be obtained by polycondensation of propylene oxide and ethylene oxide with ethylenediamine:



- 20 and/or adducts of polyethylene oxide with fatty alcohols (IV)

and/or polyether/polyester block polymers (V).

- 25 Particularly preferred surfactant components a1) are the surfactant classes (I), (II), (III) and (IV). These nonionic surfactants generally have a polyethylene oxide

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content of from 5 to 80% by weight, molar weights of up to 10000 g/mol, and hydroxyl contents of between 0.3 and 8.0% by weight.

Component a) is prepared such that the nonionic surfactant a1) is added even before
5 or during the polymerization. It is preferred to employ a1) before the preparation of a), in the initial polymer charge.

The polyisocyanate component b) may in this case exhibit both

- 10 b1) nonhydrophilicized character and
- b2) nonionically hydrophilicized character or else
- b3) anionically hydrophilicized character.

Where a nonhydrophilicized, water-insoluble or non-water-dispersible
15 polyisocyanate component b1) is used in the aqueous 2K PU system, effective dispersing is possible only by means of a highly effective dispersing unit, e.g., with a nozzle jet disperser in accordance with EP-A 0 685 544. Only by this means are the maximum possible chemical resistance properties of the 2K PU system achieved after the film has been applied.

20

Where, however, the polyisocyanate component b) is to be dispersed effectively and finely with the polyol component using more simple dispersing units such as, for example, a dissolver or through the use of a 2-component unit with preatomizer nozzle or possibly even manually, nonionic or anionic hydrophilicization of b) (as
25 components b2) and/or b3)) is absolutely necessary.

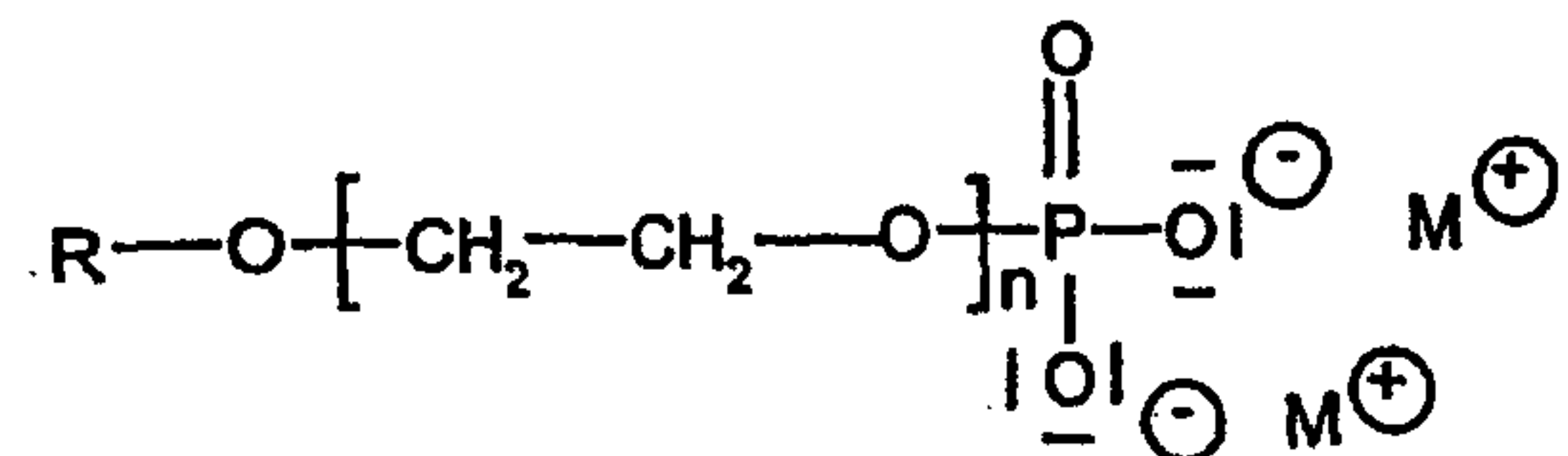
Nonionic hydrophilicization of the polyisocyanate component b) generally takes place by modification of an unmodified hydrophobic polyisocyanate with a polyether monoalcohol in accordance, for example, with EP-A 0 540 985 and EP-A 0 959 087.

30

Anionic hydrophilicization of component b) takes place preferably by using from 0.2 to 5.0% by weight (based on the sum of the solids contents of polymer polyol and polyisocyanate) of surfactants containing sulfate, sulfonate or phosphate groups to

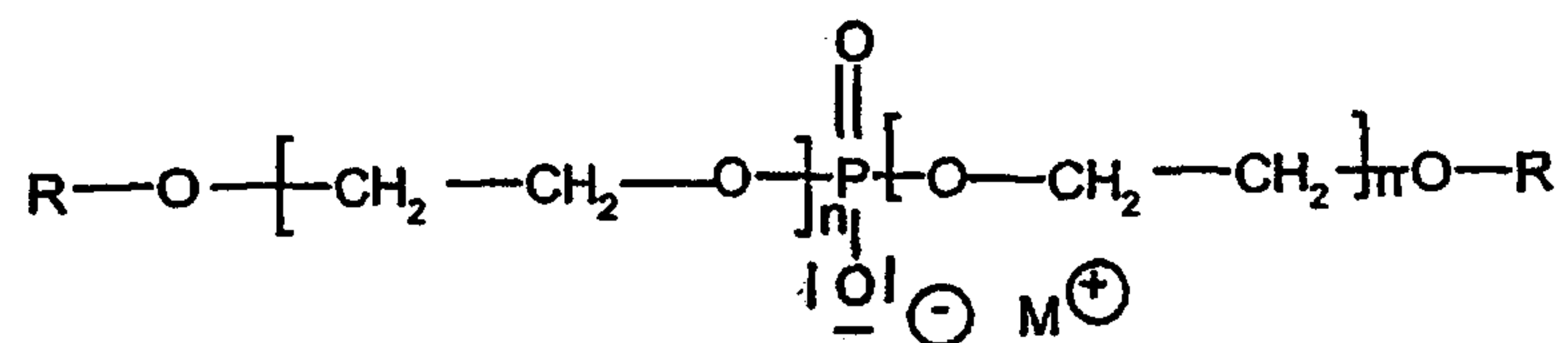
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the unmodified, hydrophobic polyisocyanate component b). The particularly preferred surfactants containing phosphate groups here have the following structures:



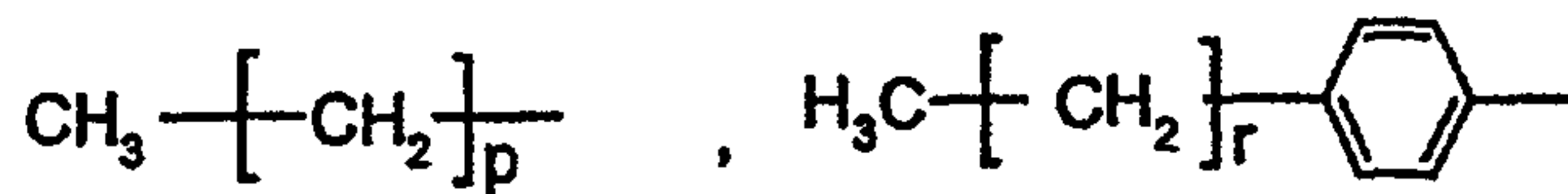
5

and/or



10

with R:



$n = 1 - 100$; $p = 5 - 20$; $r = 5 - 15$; $M = \text{alkali metal or (substituted) ammonium ion}$

The sole use of phosphate surfactants in the above structure in aqueous 2K PU systems is admittedly already known (cf., e.g., WO 98/38196 and WO 98/38231), but in the case of the mixture of the polyisocyanate component b) with the polyol component a) which has not been prepared in the presence of the nonionic surfactant component a1) leads to unusable results: the polyisocyanates b) are significantly more difficult to incorporate into the polyol a), less stable 2-component dispersions result, and the dried coating films are much more cloudy, as is also shown in the present examples; additionally, the stability of the dried 2-component film on wood substrates, for example, with respect to aqueous chemicals such as, for example, coloring substances (red wine, coffee, tea, and mustard), is significantly reduced.

Anionic hydrophilicization of component b) may take place not only by means of phosphate groups but also by means of sulfate or sulfonate groups. In this case it is

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- preferred to use sulfonate groups which are present in chemically bonded form in the polyisocyanate. These can be prepared, for example, by reacting polyisocyanates with compounds which contain sulfonate groups and which additionally bear at least one group which is reactive toward NCO groups. By way of example of such substances mention may be made of reaction products of polyisocyanates with 3-(cyclohexylamino)propanesulfonic acid. The sulfonic acid groups and/or sulfonate groups may be present in amounts of 0.1 – 5.0% by weight, based on the polyisocyanate.
- Also possible in principle is the use of polyisocyanates with mixed hydrophilicization, as is described, for example, in EP-A 0 510 438. In this case hydrophilicization is carried out simultaneously with nonionic polyether groups and with (potentially) anionic carboxyl groups.
- The polymers a) comprise hydroxy-functional copolymers with hydroxyl numbers in the range from 10 to 264 mg KOH/g resin solids, acid numbers in the range from 0 to 55 mg KOH/g resin solids, which further contain an amount of chemically bonded carboxylate and/or sulfonate groups totaling from 0 to 97 milliequivalents per 100 g of solids. The acid number here is based not only on the free, unneutralized acid groups, especially carboxyl groups, but also on the acid groups, especially carboxylate groups, which are present in neutralized form. The copolymers generally have a molecular weight M_n , as determinable by the method of gel permeation chromatography using polystyrene as standard, of from 5000 to 300000 preferably from 10000 to 200000, and a molecular weight M_w of from 30000 to 2000000, preferably from 40000 to 500000, g/mol.

The copolymers a) are preferably those formed from

- A) from 0 to 7%, preferably from 1 to 5%, by weight of acrylic acid and/or methacrylic acid,
- B) from 0 to 75% by weight of methyl methacrylate,
- C) from 0 to 75% by weight of styrene, the sum of B + C making from 10 to 85% by weight,

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- D) from 3 to 40% by weight of one or more C₁₋₈ alkyl acrylates and/or C₂₋₈ alkyl methacrylates,
- E) from 2 to 74% by weight of one or more monohydroxy-functional alkyl acrylates or alkyl methacrylates
- 5 F) from 0 to 15% by weight of other olefinically unsaturated monomers,

the sum of A) to F) making 100% by weight, in which, additionally, from 5 to 100% of the acid groups present in copolymerized form are present in a form in which they have been neutralized with aliphatic amines or with ammonia, so that the amount of

10 anionic saltlike groups in the copolymers corresponds to the figures given above.

The unsaturated acids A) and, where appropriate, F) present in copolymerized form are, as stated, at least partly neutralized, so that the resultant anionic groups ensure or at least facilitate the solubility or dispersibility of the copolymers in water. Where

15 only small concentrations of saltlike groups are present, the solubility or dispersibility of the copolymers in water can be facilitated by using external emulsifiers as well. In any case it is necessary to ensure that the copolymers are dilutable in water, either as a dispersion or as a colloidal to molecularly disperse "solution".

20

The monomers B) and C) can be varied so that in the sum B) + C) of from 10 to 85% by weight only one of the monomers is present, in which case styrene is preferred.

Suitable C₁₋₈ alkyl acrylates D) include, for example methyl acrylate, ethyl acrylate,

25 n-propyl acrylate, iso-propyl acrylate, n-butyl acrylate, iso-butyl acrylate, n-hexyl acrylate, n-octyl acrylate, 2-ethylhexyl acrylate. Preference is given to n-butyl acrylate, n-hexyl acrylate, 2-ethylhexyl acrylate, especially n-butyl and/or 2-ethylhexyl acrylate.

30 Suitable C₂₋₈ alkyl methacrylates D) include, for example, ethyl methacrylate, n-butyl methacrylate and/or 2-ethylhexyl methacrylate.

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Suitable hydroxy-functional (meth)acrylates E) include, for example, 2-hydroxyethyl (meth)acrylate, 2-hydroxypropyl (meth)acrylate, 2-hydroxyisopropyl (meth)acrylate, 4-hydroxybutyl (meth)acrylate or any desired mixtures of these monomers. Preference is given to 2-hydroxyethyl methacrylate and to the technical-grade mixture of 2-hydroxypropyl and 2-hydroxyisopropyl methacrylate, referred to generally as hydroxypropyl methacrylate.

The further monomer units F) can be substituted styrene derivatives, such as, for example, the isomeric vinyl toluenes, α -methylstyrene, propenylbenzene, C₅-C₁₂ cycloalkyl (meth)acrylate, isobornyl (meth)acrylate, vinyl esters such as vinyl acetate, vinyl propionate or versatate, vinylsulfonic acid, the total amount of polymerizable acids (carboxylic acids A) plus, where appropriate, the acids specified under F) not exceeding 7% by weight.

Compounds suitable for the at least partial neutralization of the acid groups present in copolymerized form include ammonia or aliphatic amines such as, for example, triethylamine, dimethylethanolamine, diethylethanolamine, triethanolamine, or any desired other aliphatic amines, preferably with molecular weights in the range from 31 to 200.

The copolymers a) may be prepared, for example, by the principle of solution polymerization in organic solvents. Suitable solvents are for example toluene, xylene, technical-grade mixtures of alkylaromatics (Solvesso* 100, Solvesso* 150, etc.), chlorobenzene, ethyl or butyl acetate, methyl or ethyl glycol acetate, methoxypropyl acetate, methoxybutyl acetate, butyl glycol, dioxane, ethylene glycol monoethyl or diethyl ether, dipropylene glycol dimethyl ether, acetone, butanone, methylene chloride or any mixtures of such solvents.

For the preparation of the copolymers a) in solution, such solvents generally used, and the low concentrations at which they are generally used, are such that it is unnecessary to remove them following the end of polymerization and conversion into the aqueous phase.

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Suitable initiators of this free-radical solution polymerization include, for example, aliphatic azo compounds such as azoisobutyronitrile (AIBN) or peroxidic compounds, e.g., benzoyl peroxide, tert-butyl peroctoate, tert-butyl perpivalate, tert-butyl perbenzoate or di-tert-butyl peroxide.

5

Suitable molecular weight regulators include principally sulfur compounds, such as dodecyl mercaptan (dodecanethiol) or thioglycol.

After polymerization is at an end, the solutions or dispersions a) are prepared preferably by adding neutralizing amine directly to the organic polymer solution and then introducing the neutralized solution into the aqueous phase or by metering the organic polymer solution into the water phase, to which neutralizing agent has been added, and homogenizing it therein. Where necessary, the organic solvent present during polymerization may be subsequently removed, in whole or in part, by distillation.

Particularly advantageous, however, is the preparation of the copolymers a) directly in aqueous dispersion by the technique of emulsion polymerization. Particularly suitable free-radical initiators in this case include peroxodisulfates, e.g., potassium or ammonium peroxodisulfate. Where the copolymers a) are prepared in accordance with the principle of emulsion polymerization it is also possible to use external emulsifiers such as, for example, anionic emulsifiers, such as those based on alkyl sulfate, alkylarylsulfonates, alkylphenol polyether sulfates as specified, for example, in Houben-Weyl, Methoden der organischen Chemie, Erweiterungs- und Folgebände, 4th edition, volume E 20, 1987 (part 1, pages 259 to 262) and/or alkyl polyether sulfates, or nonionic emulsifiers, such as the alkoxylation, especially ethoxylation, products of alkanols, phenols or fatty acids, for example, which remain in the system following the preparation of the copolymers and which may be regarded as auxiliaries c). Where such emulsifiers are present it is often sufficient just to neutralize the attendant acid groups to a very small extent in order to ensure the homogeneity of the solutions or dispersions a). The neutralizing agents which are nevertheless used, at least at a low concentration, can be incorporated into the system as early as during the emulsion polymerization. These dispersions generally have

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solids contents of from 20 to 60% by weight, pHs of from 6 to 10, viscosities of from 10 to 5000 mPas, and average particle diameters of from 50 to 300 nm (measured by laser correlation spectroscopy). The glass transition temperatures (determined by means of DSC) of the solid resins are above 20°C, preferably above 40°C.

5

Polyisocyanates suitable as component b) are in particular the so-called "paint polyisocyanates", having aromatically or (cyclo)aliphatically attached isocyanate groups, the latter (cyclo)aliphatic polyisocyanates being particularly preferred.

- 10 Very highly suitable, for example, are "paint polyisocyanates" based on hexamethylene diisocyanate or on 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane (IPDI) and/or bis(isocyanatocyclohexyl)methane, especially those based exclusively on hexamethylene diisocyanate. By "paint polyisocyanates" based on these diisocyanates are meant the derivatives of these
- 15 diisocyanates, said derivatives being known per se and containing biuret, urethane, uretdione, allophanate and/or isocyanurate groups, which following their preparation have been freed conventionally, preferably by distillation, from excess starting diisocyanate down to a residual content of less than 0.5% by weight. The preferred aliphatic polyisocyanates for use in accordance with the invention include
- 20 polyisocyanates which meet the abovementioned criteria, contain biuret groups and are based on hexamethylene diisocyanate, such as may be obtained, for example, by the processes described in US-A 3 124 605, US-A 3 358 010, US-A 3 903 126, US-A 3 903 127 or US-A 3 976 622, and which consist of mixtures of N,N',N''-tris(6-isocyanatohexyl)biuret with minor amounts of its higher homologues, and also the
- 25 cyclic trimers of hexamethylene diisocyanate which meet the criteria specified, such as may be obtained in accordance with US-A 4 324 879, and which consist essentially of N,N'',N'''-tris(6-isocyanatohexyl)isocyanurate in a mixture with minor amounts of its higher homologues. Particular preference is given to the mixtures which meet the criteria specified and are composed of polyisocyanates based on
- 30 hexamethylene diisocyanate and containing uretdione and/or isocyanurate groups, such as are formed by catalytic oligomerization of hexamethylene diisocyanate using trialkylphosphines. Particular preference is given to the last-mentioned mixtures with a viscosity at 23°C of from 50 to 500 mPas and an NCO functionality of between 2.2

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and 5.0. Also possible, however, is the use of monomeric polyisocyanates such as 4-isocyanatomethyl-1,8-octane diisocyanate, for example.

5 The aromatic polyisocyanates, which are likewise suitable in accordance with the invention but less preferred, are, in particular, "paint polyisocyanates" based on 2,4-diisocyanatotoluene or its technical-grade mixtures with 2,6-diisocyanatotoluene or based on 4,4'-diisocyanatodiphenylmethane or its mixtures with its isomers and/or higher homologues. Examples of aromatic paint polyisocyanates of this kind are the isocyanates containing urethane groups, such as are obtained by reacting excess
10 amounts of 2,4-diisocyanatotoluene with polyhydric alcohols such as trimethylolpropane and then removing the unreacted diisocyanate excess by distillation. Examples of further aromatic paint polyisocyanates are the trimers of the monomeric diisocyanates exemplified, i.e., the corresponding isocyanatoisocyanurates, which have likewise been freed, following their
15 preparation, from excess monomeric diisocyanates, preferably by distillation.

Also possible in principle, of course, is the use of unmodified polyisocyanates of the type exemplified, provided they are in accordance with the comments made in relation to viscosity.

20

The preferred use of hydrophilicized polyisocyanates b), prepared by nonionic and/or anionic hydrophilicization of the hydrophobic polyisocyanates mentioned above, has been described earlier on above.

25 The polyisocyanate component b) may consist, moreover, of any desired mixtures of the polyisocyanates exemplified.

The 2K PU mixtures for use in accordance with the invention are generally prepared simply by stirring the individual components a) and b) together manually or under
30 the action of a stirrer or else, in the case of difficult-to-disperse 2K systems, with a nozzle jet disperser, the proportions being chosen so as to result in NCO/OH equivalents ratios of from 0.2:1 to 5:1, preferably from 0.7:1 to 3:1.

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In this context it is possible in principle to add the nonionic surfactant component a1) as early as before or during the polymerization of a). If component a1) is incorporated into the polyol component a) even before the polymerization, the addition is made, in the case of an emulsion polymerization, to the aqueous initial
5 charge, generally together with an anionic emulsifier.

If component a1) is introduced during the feed polymerization, then the addition is generally made to the monomer mixture to be fed in or, in the case of sufficient water dispersibility, to the aqueous initiator solution to be fed in. Also possible in principle
10 is the addition of surfactant component a1) to the polyol component a) after the end of polymerization.

The compatibility between the polyisocyanate component b) and the polyol component a), and hence also the easier dispersibility of the aqueous 2K PU system
15 prepared from these components, is considerably increased if the polyisocyanate component has been either nonionically or anionically hydrophilicized. Dispersion can then be carried out in some cases simply manually or with simple stirring units such as, for example, a dissolver.

20 It is possible, where appropriate, to incorporate further auxiliaries and additives c) of coatings technology into the mixtures of a) and b) to be used in accordance with the invention and/or into the individual components used to prepare said mixtures.

These further auxiliaries and additives include, for example, further amounts of water
25 and/or solvents for the purpose of establishing the appropriate processing viscosity in the coating compositions of the invention. In the ready-to-use coating compositions of the invention there are, based on the total weight of all components, generally from 2 to 25% by weight of organic solvents and from 10 to 75% by weight of water. Further auxiliaries and additives c) are, for example, pigments, fillers, leveling
30 agents, thickeners, defoamers, devolatilizers, and the like.

The coating compositions of the invention can be applied by all standard industry methods such as, for example, spraying, dipping or spreading onto any desired

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substrates such as, for example, wood, metal, plastic or else mineral substrates, and dried at from room temperature to about 80°C. Application to wood is particularly preferred.

5 Examples

Polyisocyanate 1

Desmodur® N 100 (Bayer AG, Leverkusen, DE) is a polyisocyanate which has not
10 been hydrophilically modified and which is based on 1,6-diisocyanatohexane, having an NCO content of about 22.0%, an average NCO functionality of about 3.8, a viscosity of about 10000 mPas (23°C), and an NCO equivalent weight of about 191 g/mol. The following components were incorporated into this polyisocyanate: to 70 parts by weight of the 100% polyisocyanate, 7 parts by weight of the emulsifier
15 Rhodafac® RE 610 (Rhône Poulenc Chemicals), 1 part by weight of triethylamine and 22 parts by weight of methoxybutyl acetate.

Polyisocyanate 2

20 850 g (4.39 eq) of a polyisocyanate containing isocyanurate groups and based on 1,6-diisocyanatohexane (HDI), having an NCO content of 21.7%, an average NCO functionality of 3.5 (by GPC), a monomeric HDI content of 0.1% and a viscosity of 3000 mPas (23°C), are introduced under dry nitrogen at 100°C with stirring; over the course of 30 minutes, 150 g (0.30 eq) of a monofunctional polyethylene oxide
25 polyether, prepared starting from methanol and with an average molecular weight of 500, corresponding to an NCO/OH equivalent ratio of 14.6:1, are added; and then stirring is continued at this temperature until, after about 2 hours, the NCO content of the mixture has fallen to a level of 17.2%, which corresponds to complete urethaneization. The allophanatization reaction is started by adding 0.01 g of zinc(II)
30 2-ethyl-1-hexanoate. The temperature of the reaction mixture rises to 106°C owing to the heat of reaction given off. After the evolution of heat has subsided, about 30 minutes after adding the catalyst, the reaction is terminated by adding 0.01 g of benzoyl chloride and the reaction mixture is cooled to room temperature. The

- 15 -

product is a virtually colorless, clear, polyisocyanate mixture having the following characteristics:

	Solids content:	100%
5	NCO content:	16.0%
	NCO functionality:	4.0
10	Viscosity (23°C):	3200 mPas

Polyisocyanate 2 is used as a 70% strength by weight solution in methoxybutyl acetate.

15 **Polyisocyanate 3:**

970 g (5.00 eq) of a polyisocyanate containing isocyanurate groups and based on 1,6-diisocyanatohexane (HDI), having an NCO content of 21.7%, an average NCO functionality of 3.5 (by GPC), a monomeric HDI content of 0.1% and a viscosity of
20 3000 mPas (23°C), are stirred at 80°C for 5 hours under dry nitrogen together with 30 g (0.14 eq) of 3-(cyclohexylamino)propanesulfonic acid, 17.4 g (0.14 mol) of dimethylcyclohexylamine and 254 g of 1-methoxypropyl 2-acetate. The product, after cooling to room temperature, is a virtually colorless, clear solution of a polyisocyanate mixture according to the invention, having the following
25 characteristics:

	Solids content:	80%
	NCO content:	16.0%
30	NCO functionality:	3.4
	Viscosity (23°C):	570 mPas

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Color number: 15 APHA

Sulfonate group content: 0.9%

- 5 In all of Examples 1-12 the dispersions are what are termed primary dispersions, which – apart from the inventive use of the surfactants a1) – were synthesized in analogy to Examples 1 and 2 of EP-A 0 557 844.

10 **Polymer polyol 1 (primary dispersion as comparative example without nonionic surfactant)**

This vinyl polymer polyol primary dispersion is based on the following comonomer mixture:

- 15 Acrylic acid (3.0% by weight), hydroxypropyl methacrylate (16.9% by weight), methyl methacrylate (50.0% by weight) and n-butyl acrylate (28.1% by weight), polymerized in the presence of the emulsifier Agitan® 951 (Bayer AG, Leverkusen, DE) (ammonium salt of dodecyl triethylene oxide monosulfate; approximately 2.0% by weight based on monomer mixture). The following physicochemical
20 characteristics are obtained:

- | | |
|--|--------------------------|
| Solids content: | 42.0% by weight |
| pH: | 7.5 |
| Viscosity (23°C; D = 42s ⁻¹) | 400 mPas |
| 25 Average particle diameter (LCS) | 93 nm |
| Acid number: | 12.4 mg KOH/g dispersion |
| Glass transition temperature Tg: | 55°C |

30 **Polymer polyol 2 (comparative example in analogy to Example 1 with nonionic surfactant added subsequently)**

The polymer polyol was prepared as in the example of the polymer polyol 1; after the end of polymerization, however, a nonionic polyethylene oxide/propylene oxide

- 17 -

block polymer having a molecular weight of 2090 g/mol and an ethylene oxide content of 10% (Synperonic® PE/L 61 (ICI Surfactants); approximately 2% by weight in respect of the dispersion) was added to the finished dispersion. The physicochemical characteristics of this dispersion are similar to those of polymer
5 polyol 1.

Polymer polyol 3

Vinylpolymer polyol dispersion based on the following comonomers:

10

Acrylic acid (3.0% by weight), hydroxypropyl methacrylate (16.9% by weight), styrene (68.4% by weight) and n-butyl acrylate (9.7% by weight) polymerized in the presence of the same emulsifier (approximately 2.0% by weight in respect of monomer mixture) as in the case of polymer polyol 1 and with the addition of the
15 same polyethylene oxide/polypropylene oxide block polymer as in Example 2 (Synperonic® PE/L 61 (ICI Surfactants); approximately 2% in respect of the dispersion) to the initial polymer charge. The dispersion has the following physicochemical characteristics:

20	Solids content:	43.9% by weight
	pH:	7.0
	Viscosity (23°C; D = 42s ⁻¹)	about 80 mPas
	Average particle diameter (LCS)	91 nm
	Acid number:	12.8 mg KOH/g dispersion

25

Polymer polyol 4 (primary dispersion)

Vinylpolymer polyol dispersion based on the same comonomers as for polymer polyol 3; preparation likewise in the presence of the same emulsifier as for polymer
30 polyol 1. During the feed polymerization the monomer mixture fed in is admixed with the block polymer Synperonic® PE/L 61 (ICI Surfactants; approximately 2% in respect of the dispersion).

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The following physicochemical characteristics are obtained:

	Solids content:	43.6% by weight
	pH:	7.2
5	Viscosity (23°C; D = 42s ⁻¹)	90 mPas
	Acid number:	13.9 mg KOH/g dispersion
	Average particle diameter (LCS):	102 nm

10 **Polymer polyol 5 (primary dispersion) as comparative example of a polyol with subsequent addition of a nonionic surfactant)**

15 Vinylpolymer polyol dispersion based on the same comonomers as for polymer polyol 3; preparation likewise in the presence of the emulsifier 951 (ammonium salt of dodecyl triethylene oxide monosulfate; approximately 2.0% by weight based on monomer mixture); after the end of polymerization, in the course of neutralization with ammonia, the dispersion is admixed with the polyethylene oxide/polypropylene oxide block polymer Synperonic® PE/L 61 (approximately 2% in respect of dispersion).

20 The following physicochemical characteristics are obtained:

	Solids content:	43.2% by weight
	pH:	7.5
	Viscosity (23°C; D = 42s ⁻¹)	about 70 mPas
25	Acid number:	13.4 mg KOH/g dispersion
	Average particle diameter (LCS):	75 nm
	Molecular weight Mn [g/mol]:	23300
	Molecular weight Mw [g/mol]:	581000
	Glass transition temperature (DSC)	81°C

Polymer polyol 6

Vinylpolymer polyol dispersion based on the same comonomers as in the case of polymer polyol 3; preparation likewise in the presence of the same emulsifier as in the case of polymer polyol 1. Inserted additionally into the initial charge is a polyethylene oxide/polypropylene oxide block polymer having a molecular weight of 2200 g/mol and an ethylene oxide content of 40% (Synperonic® PE/L 44 (ICI Surfactants); approximately 2% in respect of the dispersion). The aqueous dispersion has the following physicochemical characteristics:

10

Solids content:	43.4% by weight
pH:	7.2
Viscosity (23°C; D = 42s ⁻¹)	< 100 mPa*s
Acid number:	13.3 mg KOH/g dispersion
15 Average particle diameter (LCS):	110 nm

Polymer polyol 7

Vinylpolymer polyol dispersion in accordance with EP-A 0 358 979 based on the same comonomers as in the case of polymer polyol 3; preparation likewise in the presence of the same emulsifier as in the case of polymer polyol 1. Inserted additionally into the initial charge is a polyethylene oxide/polypropylene oxide block polymer having a molecular weight of 2900 g/mol and an ethylene oxide content of 40% (Synperonic® PE/L 64 (ICI Surfactants); approximately 2% in respect of the dispersion). The aqueous dispersion has the following physicochemical characteristics:

20

25

Solids content:	43.4% by weight
pH:	7.1
30 Viscosity (23°C; D = 42s ⁻¹)	< 100 mPa*s
Acid number:	12.9 mg KOH/g dispersion
Average particle diameter (LCS):	125 nm

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Polymer polyol 8

Vinylpolymer polyol dispersion based on the same comonomers as in the case of polymer polyol 3; preparation likewise in the presence of the same emulsifier as in the case of polymer polyol 1. Inserted additionally into the initial charge is a polyethylene oxide/polypropylene oxide block polymer having a molecular weight of 3800 g/mol and an ethylene oxide content of 10% (Synperonic® PE/L 101 (ICI Surfactants); approximately 2% in respect of the dispersion).

10 The aqueous dispersion has the following physicochemical characteristics:

	Solids content:	43.2% by weight
	pH:	7.0
	Viscosity (23°C; D = 42s ⁻¹)	< 100 mPa*s
15	Acid number:	12.8 mg KOH/g dispersion
	Average particle diameter (LCS):	93 nm

Polymer polyol 9

20 Vinylpolymer polyol dispersion based on the same comonomers as in the case of polymer polyol 3; preparation likewise in the presence of the same emulsifier as in the case of polymer polyol 1. Inserted additionally into the initial charge is a polyethylene oxide/polypropylene oxide block polymer having a molecular weight of 4800 g/mol and an ethylene oxide content of 80% (Synperonic® PE/F 38 (ICI Surfactants); approximately 2% in respect of the dispersion).

The aqueous dispersion has the following physicochemical characteristics:

	Solids content:	43.8% by weight
30	pH:	6.7
	Viscosity (23°C; D = 42s ⁻¹)	< 100 mPa*s
	Acid number:	13.0 mg KOH/g dispersion
	Average particle diameter (LCS):	133 nm

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Polymer polyol 10

Vinylpolymer polyol dispersion in accordance with Example 3, prepared in the presence of the same emulsifier. Inserted additionally into the initial charge is a
5 ethylene oxide/propylene oxide block polymer, based on ethylenediamine as starting compound, having a molar weight of 3700 g/mol and an ethylene oxide content of 10% (Synperonic® T/701 (ICI Surfactants); approximately 2% in respect of the dispersion).

10 The aqueous dispersion has the following physicochemical characteristics:

	Solids content:	43.6% by weight
	pH:	7.5
	Viscosity (23°C; D = 42s ⁻¹)	< 100 mPas
15	Acid number:	13.8 mg KOH/g dispersion
	Average particle diameter (LCS):	105 nm

Polymer polyol 11

20 Vinylpolymer polyol dispersion in accordance with Example 3, prepared in the presence of the same emulsifier. Inserted additionally into the initial charge is a polyether, prepared starting from a fatty alcohol, having the approximate molecular weight of 630 g/mol (Synperonic® LF/RA 30 (ICI Surfactants); approximately 2%
in respect of the dispersion).

25

The aqueous dispersion has the following physicochemical characteristics:

	Solids content:	44.5% by weight
	pH:	8.5
30	Viscosity (23°C; D = 42s ⁻¹)	< 100 mPas
	Acid number:	14.2 mg KOH/g dispersion
	Average particle diameter (LCS):	95 nm

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Polymer polyol 12

Vinylpolymer polyol dispersion consisting of the same comonomers as in Example 1 in the same proportions by weight, prepared in the presence of the same emulsifier as
5 for polymer polyol 1. Inserted additionally into the initial charge is the same nonionic surfactant as for polymer polyol 2 (Synperonic® PE/L61; approximately 2% in respect of the dispersion).

The aqueous dispersion has the following physicochemical characteristics:

10	Solids content:	43.6% by weight
	pH:	7.5
	Viscosity (23°C; D = 42 /s)	150 mPa*s
	Acid number:	14.5 mg KOH/g dispersion
	Average particle diameter (LCS):	121 nm

15

Performance Examples**Examples 1-6**

20 In Examples 1 – 6 the polymer Example 12 and the Comparative Examples 1 and 2 are investigated in respect of the ease of incorporation of polyisocyanates 1 and 2.

All numerical figures are parts by weight.

25 First of all pigment pastes were prepared from these 3 polymer dispersions. For that purpose the polymer dispersions were admixed first with Disperbyk® 190 and BYK® 024 (both Byk Chemie, Wesel, DE) and the Tronox® R-KB-2 (Kerr McGee Pigments GmbH, Krefeld, DE) and Aerosil® 200 (Grace, DE) were incorporated by dispersion using a dissolver. Dispersion was continued at 2000 rpm for about 15
30 minutes.

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A) Pigment pastes

	Pigment paste 1	Pigment paste 2	Pigment paste 3
Polymer polyol 12	208.0		
Polymer polyol 2		168.0	
Polymer polyol 1			168.0
Tronox R-KB-2 ¹⁾	200.0	200.0	200.0
Aerosil 200 ²⁾	1.0	1.0	1.0
Disperbyk 190 ³⁾	8.0	8.0	8.0
BYK 024 ⁴⁾	2.0	2.0	2.0

- 1) Tronox® R-KB-2: titanium dioxide pigment (Kerr McGee Pigments, Krefeld)
- 2) Aerosil® 200: silicon dioxide as antisetling agent (Grace)
- 3) Disperbyk® 190: wetting and dispersing agent (Byk Chemie, Wesel)
- 4) BYK 024: defoamer (BYK Chemie, Wesel)

10 These pigment pastes were used to formulate 6 paints in accordance with the table below. These paints were drawn down using a box-type coating bar in a wet film thickness of approximately 200 μm onto an Erichsen test chart and were dried at 18 – 24°C for 24 hours. Thereafter the gloss (angle 60°) was measured [value 1: black card/value 2: white card].

15

B) Paint formulation

Example	1	2	3	4	5	6
Pigment paste 1	42.0			42.0		
Pigment paste 2		38.0			38.0	
Pigment paste 3			38.0			38.0
Polymer polyol 12	34.0			34.0		
Polymer polyol 2		38.0			38.0	
Polymer polyol 1			38.0			38.0
BYK 024	1.0	1.0	1.0	1.0	1.0	1.0

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Butyl glycol / water (1:1)	16.0	16.0	16.0	16.0	16.0	16.0
Water	7.0	7.0	7.0	7.0	7.0	7.0
Polyisocyanate 1	10.0	10.0	10.0			
Polyisocyanate 2				10.0	10.0	10.0
Gloss on test card 60°	69/70	62/64	62/68	49/52	36/38	36/36

Examples 1 and 4 show that through the inventive incorporation of the nonionic surfactants into the polyol dispersion it is much easier to incorporate polyisocyanates and thus a marked increase in gloss is occasioned as compared with the noninventive 2K systems 2, 3, 5, and 6.

Examples 7-10

In this test series the polymer Example 3 with the polymer comparison 5 was investigated in pigmented paint systems for chemical resistance in accordance with DIN 68861. The paints were prepared as in the case of Examples 1-6.

	Example 7	Example 8	Example 9	Example 10
Polymer polyol 3	56		56	
Polymer polyol 5		56		56
Disperbyk 190	1.7	1.7	1.7	1.7
Tego Foamex* 810 ⁵⁾	0.02	0.02	0.02	0.02
Aerosil* 200	0.1	0.1	0.1	0.1
Tronox* R-KB-2	14.2	14.2	14.2	14.2
Butyl glycol : water 1:1	16	16	16	16
Serad* FX 1010 5% Strength in water ⁶⁾	7	7	7	7
BYK 024	1	1	1	1
Water	4	4	4	4
Polyisocyanate 2	10	10		
Polyisocyanate 1			10	10
EA/BA 1:1 10 s	0	0	0	0

*trade-mark

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Acetone 10s	1/1	1/1	0	0
Ammonia 2 min	0	0	0	0
Sodium carbonate 2 min	0	0	0	0
Ethanol 50% 1 h	0	0	0	0
Water 16 h	0	0	0	0
Red wine 5 h	1/1	2/2	1/0	2/1
Mustard 5 h	1/0	3/3	1/0	2/2
Coffee 16 h	1/1	3/3	0	3/3

5) Tego Foamex 810: defoamer (Tego Chemie, Essen)

6) Serad FX 1010: thickener (Servo Delden BV, Delden, Netherlands)

- 5 These examples show that through the inventive incorporation of the nonionic surfactants there is a marked improvement in the chemical resistances, particularly with respect to coloring liquids (rating 0: highly stable to chemicals; 5: completely unstable).

Examples 11 - 17

10

In this test series clearcoat systems are produced from the polymer dispersions 3 and 6 – 11 and are investigated for the development of gloss and for chemical resistance in accordance with DIN 68861.

Example	11	12	13	14	15	16	17
Polymer polyol 6	62						
Polymer polyol 7		62					
Polymer polyol 8			62				
Polymer polyol 9				62			
Polymer polyol 10					62		
Polymer polyol 11						59	
Polymer polyol 3							62
BYK 024	1	1	1	1	1	1	1

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Serad FX 1010, 5% strength in water	7	7	7	7	7	7	7
Butyl glycol	8	8	8	8	8	8	8
Water	22	22	21.5	22.5	22	25	22.5
Polyisocyanate 2	10	10	10	10	10	10	10
Gloss 60° cardboard b/w	95/99	90/96	95/99	97/100	95/97	98/101	95/100
EA/BA 1:1 10 s	1/0	1/0	1/0	0	1/0	0	1/0
Acetone 10 s	2/0	1/0	2/0	1/0	2/0	2/0	1/0
Ammonia 2 min	0	0	0	0	0	0	0
Sodium carbonate 2 min	0	0	0	0	0	0	0
Ethanol 50%, 1 h	0	0	0	0	0	1/0	0
Water 16 h	0	0	0	0	0	0	0
Red wine 5 h	0	0	0	0	0	0	0
Mustard 5 h	0	0	0	0	1/0	1/0	0
Coffee 16 h	0	0	0	0	0	0	0

The curing agent was incorporated by dispersion using a dissolver at a rate of approximately 2000 rpm for 2 minutes. Thereafter, wet films were applied in a film thickness of 150 μm to an MDF board coated with a melamine resin sheet. The test area was initially dried at about 20°C for 15 minutes, then cured at 60°C for 60 minutes and also stored at approximately 20°C for 7 days. The chemical resistances were tested in accordance with DIN 68861.

Note: where 2 measurements are listed, the 1st figure denotes an immediate assessment and the 2nd figure the assessment after 3 days.

This coat was also drawn down onto a test chart in a wet film thickness of 200 μm and after 24 hours (storage at approximately 20°C) the degree of gloss was measured (at an angle of 60°).

15

This test series shows that with the OH-containing dispersions of the invention, high-gloss varnish systems with outstanding chemical resistances are obtained.

Example 18

Paints were produced in accordance with Example 9 and were applied using an airless unit (2-component unit from Graco) to sheet-coated MDF boards. Application tests were conducted with the following nozzle combinations:

Application example 1: spray nozzle (0.32 mm); no preatomizer nozzle

Application example 2: spray nozzle (0.32 mm); preatomizer nozzle (0.38 mm)

Application example 3: spray nozzle (0.32 mm); preatomizer nozzle (0.28 mm)

10 Application example 4: spray nozzle (0.32 mm); preatomizer nozzle (0.23 mm)

The paints were applied in a wet film thickness of about 200 g/m², dried initially at 22°C for about 30 minutes and postcured at approximately 60°C for about 1 hour. The appearance of these coatings was assessed.

15

While application Examples 1 and 2 led to matt, non-uniform coatings, application Example 4 in particular gave a high-gloss, uniform coating. Application Example 3 showed a slight deterioration in comparison with application Example 4. These application examples show that the dispersions of the invention can be applied under conditions in accordance with those in practice on a customary 2-component spraying unit using simple preatomizer nozzles which possess a smaller aperture than the spray nozzle.

20

Example 19

25

In Example 19, a pigment paste is first of all incorporated by dispersion, using a dissolver, with the additives indicated below in the table. Dispersion was continued at 2000 rpm for approximately 15 minutes.

Pigment paste	
Polymer polyol dispersion 3	168.0
Tronox R-KB-2	200.0

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Aerosil 200	1.0
Byk 024	2.0
Disperbyk 190	4.0+4.0

This pigment paste was used for paint formulation in accordance with the table below, in analogy to Examples 1-6.

Make-up	
Pigment paste	38.0
Polymer polyol dispersion	38.0
3	
Byk 024	1.0
Butyl glycol	8.0
Water	15.0
Polyisocyanate 3	10.0

5

The performance results are listed in the table below. Parameters investigated here were the drying behavior, the development of hardness, by means of pendulum damping, the gloss, and the chemical resistance (latter in accordance with DIN 68861 1B, drying: 15' RT, 60' 60°C, 7 days RT).

10

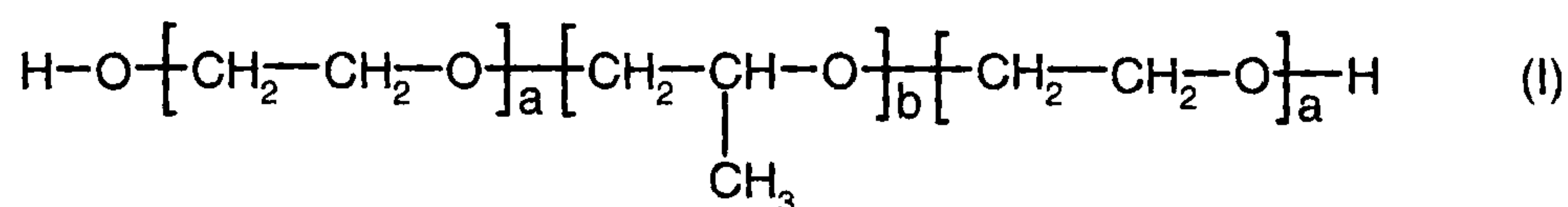
Testing	
Drying	66'
Pendulum damping	
Immediate	12"
After 1 day	47"
After 2 days	59"
After 7 days	77"
Gloss 60° b/w cardboard	86/86
EA/BA 1:1	1/0
Acetone	1/1
Ammonia	0

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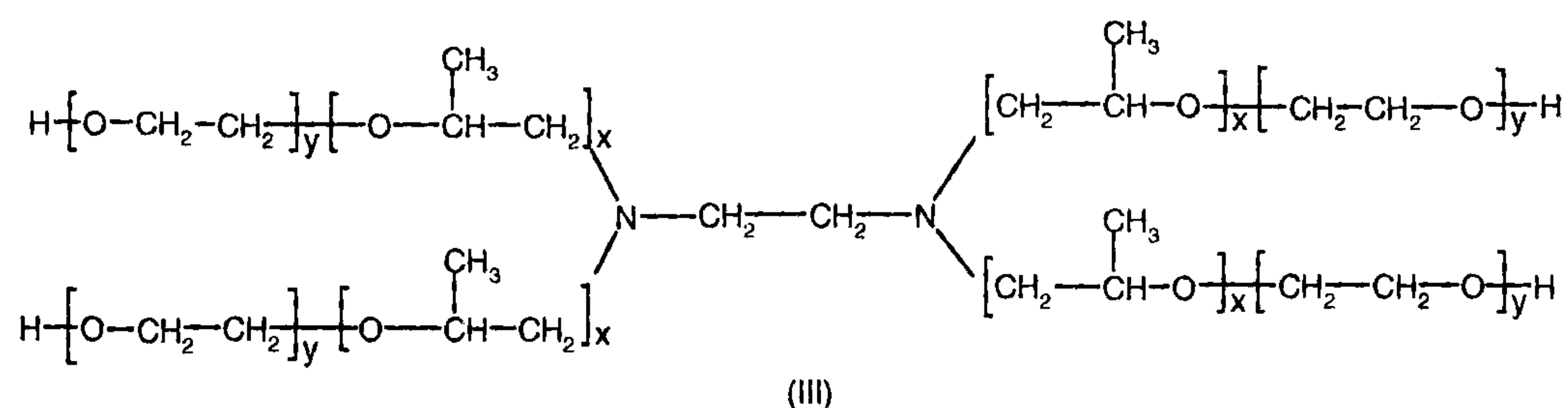
Sodium carbonate	0
Ethanol	1/0
Water	0
Red wine	0
Mustard	1/0
Coffee	0

1. An aqueous 2-component polyurethane (PU) coating composition wherein the binder comprises

- a) a vinyl polymer polyol dispersion, having a hydroxyl number between 10 and 264 mg KOH/g solid resin, an acid value (calculated from the sum of neutralized and non-neutralized acid groups) between 0 and 55 mg KOH/g solid resin, a number-average molecular weight M_n of at least 5000 g/mol and a weight average molecular weight M_w of at least 30,000 g/mol, a glass transition temperature of at least 20°C and an average particle diameter of no greater than 300 nm, which is prepared in the presence of 0.1 to 10.0 wt.%, relative to the sum of the solids contents of polymer polyol and polyisocyanate, of a non-ionic polyether surfactant, selected from the group consisting of block polymers of ethylene oxide and propylene oxide of formula (I)



20 block and random ethylene oxide/propylene oxide
copolymers based on fatty alcohols, block polymers of
formula (III), which can be obtained by polycondensation of
propylene oxide and ethylene oxide on ethylene diamine,



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adducts of polyethylene oxide on fatty alcohols and
polyether/polyester block polymers,

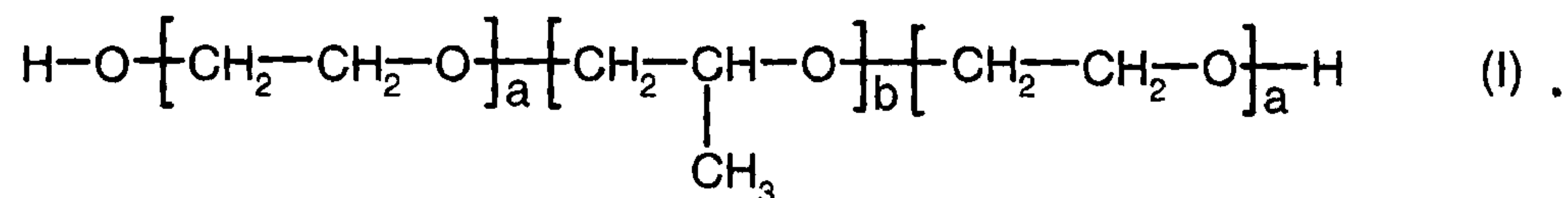
and

- 5 b) a polyisocyanate having a viscosity at 23°C of no more than 12,000
 mPa*s and either a non-hydrophilic or a non-ionically or
 anionically hydrophilic character,

wherein the equivalent ratio of isocyanate groups of component b) to hydroxyl
groups of component a) is 0.2:1 to 5:1.

- 10 2. The coating composition of Claim 1, wherein component a) is
 prepared in the presence of 1.0 to 8.0 wt. % of the non-ionic polyether surfactant.

3. The coating composition of Claim 1, wherein the non-ionic
polyether surfactant comprises a block polymer of ethylene oxide and propylene
15 oxide of formula (I):



4. The coating composition of Claim 1, wherein the non-ionic
20 polyether surfactant comprises a block and random ethylene oxide/propylene
oxide copolymer based on fatty alcohols.

5. The coating composition of Claim 1, wherein the non-ionic
polyether surfactant comprises a block polymer of propylene oxide and ethylene
25 oxide on ethylene diamine of formula (III):

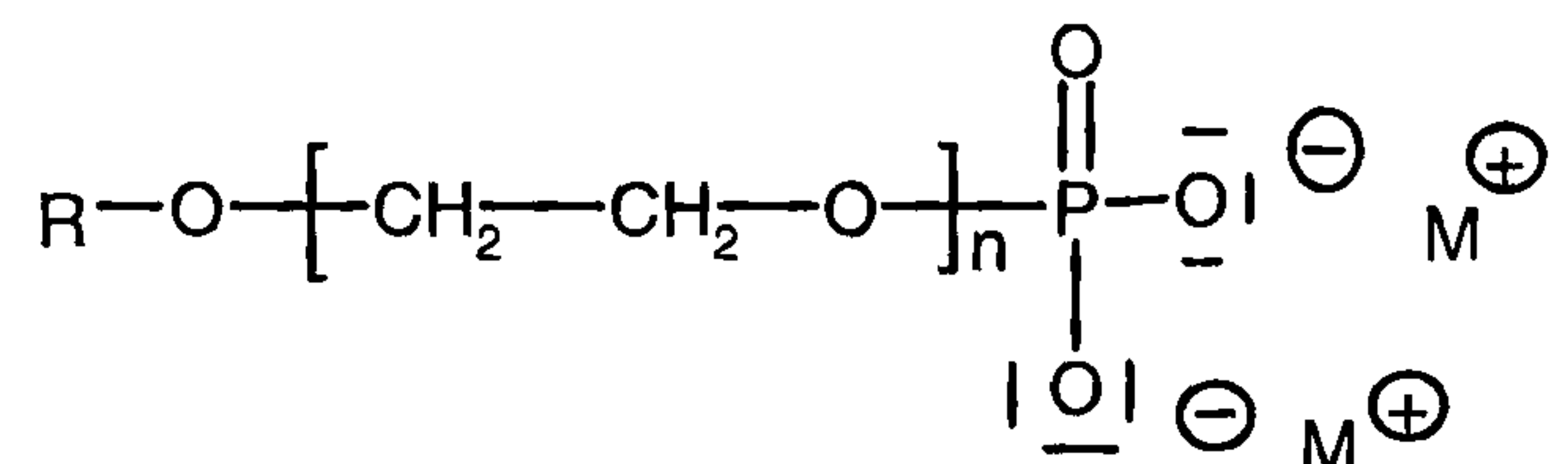
$$\begin{array}{c} \text{H}-\left[\text{O}-\text{CH}_2-\text{CH}_2\right]_y-\left[\text{O}-\overset{\text{CH}_3}{\underset{|}{\text{CH}}}-\text{CH}_2\right]_x \\ | \\ \text{N}-\text{CH}_2-\text{CH}_2-\text{N} \\ | \\ \text{H}-\left[\text{O}-\text{CH}_2-\text{CH}_2\right]_y-\left[\text{O}-\overset{\text{CH}_3}{\underset{|}{\text{CH}}}-\text{CH}_2\right]_x \end{array} \quad \begin{array}{c} \text{CH}_3 \\ | \\ \left[\text{CH}_2-\text{CH}-\text{O}\right]_x-\left[\text{CH}_2-\text{CH}_2-\text{O}\right]_y\text{H} \\ | \\ \text{CH}_3 \\ | \\ \left[\text{CH}_2-\text{CH}-\text{O}\right]_x-\left[\text{CH}_2-\text{CH}_2-\text{O}\right]_y\text{H} \end{array}$$

(III)

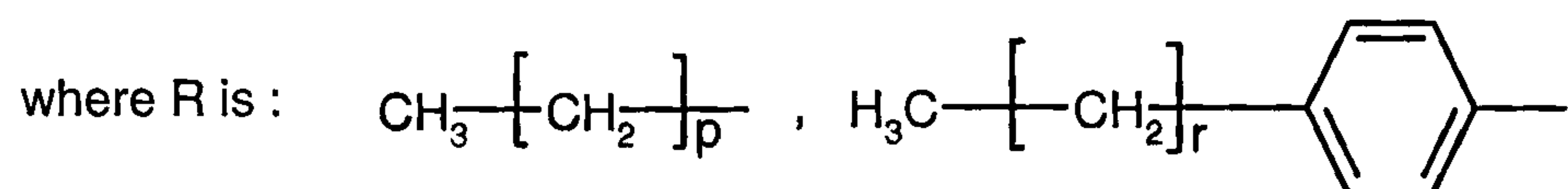
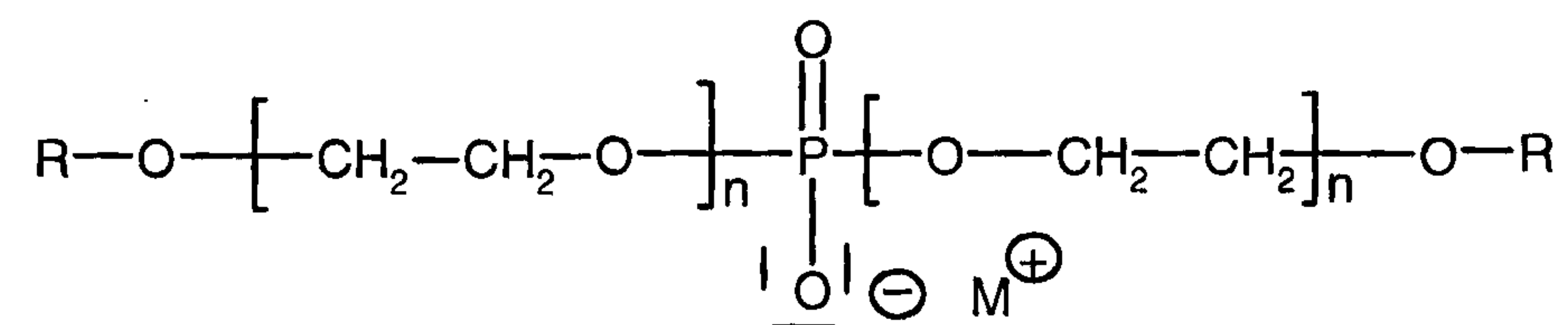
13. The coating composition of Claim 1, wherein the polyisocyanate b) contains anionic groups.

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14. The coating composition of Claim 13, wherein the polyisocyanate
b) comprises 0.2 to 5.0 wt. %, relative to the sum of solids contents of polymer
polyol and polyisocyanate, of at least one phosphate group-containing surfactant
5 selected from the group consisting of



and/or



$n = 1 - 100$; $p = 5 - 20$; $r = 5 - 15$; M = alkali- or (substituted) ammonium ion .

15. The coating composition of Claim 13, wherein the polyisocyanate
b) comprises sulfonate groups in chemically incorporated form.

16. A wood, metal, plastic or mineral substrate painted with the coating
composition of Claim 1.

17. A method of coating a substrate comprising painting thereupon the
composition according to Claim 1.

18. The method of Claim 17, wherein the substrate is selected from the
group consisting of wood, metal, plastic or mineral.