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D E C I S I O N
of 4 September 2002

Case Number: T 0542/99 - 3.3.3

Application Number: 95939965.0

Publication Number: 0792316

IPC: C08K 5/3492

Language of the proceedings: EN

Title of invention:

Waterborne compositions containing 1,3,5-triazine carbamates

Applicant:

CYTEC TECHNOLOGY CORP.

Opponent:

-

Headword:

-

Relevant legal provisions:

EPC Art. 56, 83, 84, 123(2)

Keyword:

"Inventive step (yes) - after amendment"
"Disclosure - enabling (yes) - undue burden (no)"
"Claims -clarity (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 0542/99

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 4 September 2002

Appellant: CYTEC TECHNOLOGY CORP.
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Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 1 December 1998
refusing European patent application
No. 95 939 965.0 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: R. Young
Members: A. Däweritz
J. van Moer

Summary of Facts and Submissions

- I. European patent application No. 95 939 965.0, based on International application No. PCT/US95/14837, was filed on 13 November 1995, claiming the priority of 16 November 1994 of an earlier application in the United States of America (340950) and published under No. WO-A-96/15185 on 23 May 1996. The application was refused by a decision of the Examining Division, issued in writing on 1 December 1998, for lack of inventive step and lack of clarity (Article 56 and 84 EPC). The decision was based on a set of 32 claims submitted by letter dated 27 January 1998.

Claims 1 to 14, 28 and 29 of this set related to waterborne coating compositions, Claims 15 to 27 concerned a dispersible composition, Claim 30 defined a process of preparing a waterborne coating composition based on the dispersible composition according to any one of Claims 16 to 28. Claim 31 referred to a method of coating a substrate by applying thereto a composition according to Claim 29, and in Claim 32, a substrate coated with a cured film derived from the composition according to Claim 29 was claimed.

Claims 1, 15, 16 and 28 to 32 read as follows:

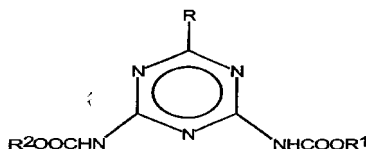
- "1. A waterborne coating composition comprising an aqueous medium having substantially homogeneously dispersed therein (1) a reactive resin component and (2) a crosslinker component, characterized in that:

- (1) the reactive resin component comprises an active hydrogen-containing surface active resin which possesses sufficient anionic hydrophilizing functionality to render the active hydrogen-containing surface active resin water dispersible;
- (2) the crosslinker component comprises a 1,3,5-triazine carbamate crosslinker; and
- (3) it further comprises an aqueous dispersion promoting material."

"15. A dispersible composition comprising a substantially homogenous mixture of (1) a reactive resin component and (2) a crosslinker component, characterized in that:

- (1) the reactive resin component comprises an active hydrogen-containing surface active resin which possesses an amount of anionic hydrophilizing functionality sufficient to render the dispersible composition water dispersible, and
- (2) the crosslinker component comprises a 1,3,5-triazine carbamate crosslinker."

"16. The dispersible composition of claim 15, wherein the 1,3,5-triazine carbamate crosslinker is selected from one or more compounds of the following formula, as well as oligomers thereof:



wherein

R is selected from the group consisting of -NHCOOR³, hydrogen, hydrocarbyl, hydrocarbyloxy, hydrocarbylthio, amido, sulfonamido, amino hydrocarbylamino, dihydrocarbylamino and cyclic amino; and each R¹, R² and R³ is independently selected from the group consisting of hydrocarbyl, hydroxyhydrocarbyl, hydrocarbyloxyhydrocarbyl and hydroxyhydrocarbyloxyhydrocarbyl."

"28. A waterborne coating composition comprising an aqueous medium having the dispersible composition of any one of claims 15-27 substantially homogeneously dispersed therein."

"29. The waterborne coating composition of claim 28, characterized in that it comprises from about 20% to about 75% by weight solids."

"30. A method of preparing a waterborne coating composition comprising the steps of:

(A) preparing a dispersible composition by substantially homogeneously mixing (1) a reactive resin component and (2) a crosslinker component, as set forth in any one of claims 16-28; and

(B) dispersing the dispersible composition in an aqueous medium, whereby prior to or concurrently with step (B), the anion generating functionality on the active hydrogen-containing surface active resin is sufficiently neutralized so as to render the

dispersible composition water dispersible."

"31. A method of coating a substrate comprising the steps of (A) applying to the substrate the waterborne coating composition according to claim 29; and (B) curing the so applied coating."

"32. A substrate coated with a cured film derived from the waterborne coating composition according to claim 29."

The remaining claims were dependent claims which concerned specific elaborations of the subject-matter of the respective antecedent independent claims cited above.

II. These claims had been submitted in reply to a first communication dated 31 October 1997, in which the Examining Division had maintained, under Articles 56 and 84 EPC, objections of lack of inventive step, lack of clarity and lack of conciseness previously raised in an international preliminary examination report (IPER) issued on 12 November 1996. The objection of lack of inventive step had been based on

D1: EP-A-0 604 922,

D2: US-A-5 084 541 and

D3: US-A-5 342 878.

In the contested decision, the Examining Division held that the closest state of the art with respect to the subject-matter of Claims 15 and 28, as well as Claims 16 to 21 (appendant to Claim 15) and 29 (appendant to

Claim 28) was represented by D3. The only difference between the subject-matter of these claims and D3 was seen in the presence of a 1,3,5-triazine carbamate as a crosslinking agent. The technical problem to be overcome with respect to D3 was defined as to provide water-dispersible compositions which could be cured to coatings having an improved weatherability.

Since (i) improved gloss after weathering of coatings obtained from aqueous coating compositions, which comprised active hydrogen-containing resins and 1,3,5-triazine di- or tricarbamates as crosslinking agent, was reported in D1 (Table 4), (ii) D2 taught that 1,3,5-triazine carbamates provided light stability and improved environmental resistance to coatings and that these crosslinking agents could be formulated into water-dispersible compositions, and (iii) the resins of D3 were known to be useful surfactants which could assist the dispersion of the tricarbamates in water, the skilled person interested in providing water-dispersible coating compositions based on water-dispersible anionic resins and having good weatherability would have envisaged the use of the crosslinking agent known from D1 and D2 in compositions according to D3. He would not have expected any particular difficulties in formulating such compositions, since the crosslinking agents of D2 had been used in waterborne compositions and the resins of D3 might have acted as surfactants for the water-insoluble components.

All other features in these claims were either disclosed in D3, constituted preferred embodiments of the crosslinking agents in D1 and D2 or were considered as standard in the art.

Claims 1 to 14 and 22 to 27 (the latter group being appendant to Claim 15) further differed from D3 by the presence of a surface active compound (the aqueous dispersion promoting material of Claim 1), the presence of which - according to the Applicant - would increase shelf life and stability. It was, however, considered evident by the Examining Division that the stability of a dispersion which tended to separate could be increased by incorporation of surface active agents, such as surfactants. Moreover, the concomitant use of the triazine carbamates and a surface active agent had already been contemplated in D2.

It was also considered obvious to first mix a resin, which could serve as a potential surfactant for the water-insoluble crosslinking agent, with this crosslinking agent and then to disperse the resulting composition in water. Claims 31 and 32 did not comprise any technical features, which could have contributed to an inventive step, and therefore shared the fate of the product claims.

The Examining Division further objected to the references in Claims 28 and 29 to Claims 22 to 27 for lack of conciseness (Article 84 EPC), because a waterborne coating composition comprising an aqueous medium and the composition of Claim 22 was already claimed in Claim 1. Claims 1 and 28 were considered as having the same scope.

Claim 29 was objected to for lack of clarity (Article 84 EPC).

III. On 29 January 1999, a Notice of Appeal against the above decision was lodged by the Appellant (Applicant). The

prescribed fee was paid on the same date.

In the Statement of Grounds of Appeal, received on 9 April 1999, and in further letters dated 3 August 2001 and 2 August 2002, which were filed, respectively, in reply to a communication from the Rapporteur dated 29 December 2000 and an annex to summons, dated 17 May 2002, to attend oral proceedings on 4 September 2002, the Appellant requested to grant a patent on the basis of further amended sets of claims, submitted together with each of its letters and replacing the respective previous versions of the claims.

Each of three sets of claims according to a main request and two auxiliary requests, respectively, submitted with the letter dated 2 August 2002, contained independent claims to a waterborne coating composition, a method of preparing a waterborne coating composition, a method of coating a substrate and a substrate coated with a cured film which essentially followed the structure of the wording of Claims 1, 30, 31 and 32 cited above.

These further amended sets of claims were to meet a number of objections against the wording of the claims in their different versions raised in the communication of the Rapporteur and the annex to the summons mentioned above, wherein objections under Article 83 and 84 EPC had been raised, because Claim 1 did not specify all the components necessary to achieve the objects referred to in the introductory part of the description: to avoid or reduce the release of volatile organic compounds (**VOC**) such as formaldehyde (upon curing) and the use of organic solvents. Moreover, high dispersibility and stability were important features of the aqueous coating composition aimed at.

IV. Oral proceedings were held on 4 September 2002. At the onset, the Appellant was informed that the Board had come to the preliminary view that it could neither accept any of the latest versions of claims nor the arguments presented by the Appellant in their support, because the objections previously raised were still valid. In particular, the definitions of the components comprised in the claimed subject-matter, which were in very general and functional terms, were such that the skilled person was faced with a situation of undue burden to find a solution for the aspects of the problem to be overcome and to obtain the promised improvement in water dispersibility and prevention of precipitation. The wording of the independent claims rendered the claimed subject-matter indefinite, because it required only vanishingly small amounts of the active ingredients comprised in components (1) and (2) as referred to in the claims and because of obscurity of the functional criterion of component (3). It was not even clear that it would be these compounds defined in the claim, which would react with each other. The examples on file could at most provide evidence for the usefulness of a specific type of polymer as component (1). In summary, the skilled person had to find out whether any conceivable compound would or would not contribute to the solution aimed at.

V. In view of these objections, the previous requests were replaced in the oral proceedings by a new set of claims reading as follows:

"1. A waterborne coating composition comprising an aqueous medium having substantially homogeneously dispersed therein (1) a reactive resin component and (2) a 1,3,5-triazine carbamate crosslinker

component, characterized in that:

- (1) the reactive resin component is a polymeric surface active resin containing active hydrogens reactive with carbamate groups under cure conditions, which possesses sufficient anionic hydrophilizing functionality to render the active hydrogen-containing surface active resin water dispersible upon neutralisation, said surface active resin being selected from copolymers of (meth)acrylic acid, hydroxyalkyl (meth)acrylates and, optionally, other free-radically polymerizable monomers which, when polymerized, possess the following characteristics:

a number average molecular weight (Mn) of from 1000 to 50000,

an acid number of from 15 to 150 mg KOH/g resin (100% solids basis); and

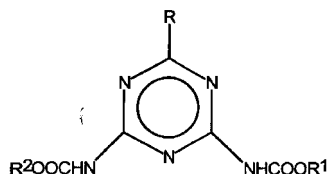
an amount of hydroxyl groups of from about 2.5 wt % to 6 wt % (100% solids basis), and that

- (3) the composition further comprises an aqueous dispersion promoting material, which is non-polymeric, in an amount sufficient to at least double the stability life of the composition, and which does not exceed 40 wt %, based on the combined weight of the resin and crosslinker components, which aqueous dispersion promoting material is

selected from the group consisting of long chain aliphatic alcohols having at least 8 carbon atoms, and a hydroxyalkyl ester of an alkanolic acid having a total of at least 8 carbon atoms,

wherein the active hydrogen-containing surface active resin and the 1,3,5-triazine carbamate crosslinker are present in amounts such that the carbamate:active hydrogen functionality ratio is in the range of from 0.5:1 to 2:1.

2. The waterborne coating composition of claim 1, characterized in that the 1,3,5-triazine carbamate crosslinker is selected from one or more compounds of the following formula, as well as oligomers thereof:



wherein

R is selected from the group consisting of -NHCOOR³, hydrogen, hydrocarbyl, hydrocarbyloxy, hydrocarbylthio, amido, sulfonamido, amino hydrocarbylamino, dihydrocarbylamino and cyclic amino; and each R¹, R² and R³ is independently selected from the group consisting of hydrocarbyl, hydroxyhydrocarbyl, hydrocarbyloxyhydrocarbyl and hydroxyhydrocarbyloxyhydrocarbyl.

3. The waterborne coating composition of claim 1, characterized in that it comprises from 20% to 75%

by weight solids.

4. A method of preparing a waterborne coating composition comprising the steps of:
 - (A) preparing a dispersible composition by substantially homogeneously mixing (1) the reactive resin component and (2) the crosslinker component, and (3) the aqueous dispersion promoting material, all as set forth in claim 1 or 2; and
 - (B) dispersing the dispersible composition in an aqueous medium, whereby prior to or concurrently with step (B), the anion generating functionality on the active hydrogen-containing surface active resin is sufficiently neutralized so as to render the surface active resin water dispersible.
5. A method of coating a substrate comprising the steps of (A) applying to the substrate the waterborne coating composition according to claim 3; and (B) curing the so-applied coating."

VI. The arguments as to patentability submitted by the Appellant can be summarised as follows:

The prior art disclosed a number of formulations of waterborne systems. However, the key to the objects underlying the patent application in suit lay in the finding of a formulation having a sufficiently high dispersion stability and the finding of films derived therefrom which should be hard, resistant and durable.

It was the surface active resin containing both active hydrogen-containing and sufficient anionic hydrophilising functionalities, which was reactive with the 1,3,5-triazine carbamates and allowed to switch from organic solvent-based to waterborne systems due to its contribution to the dispersion in water of these typically hydrophobic and non-dispersible compounds.

The hydroxyl functional acrylic polymer of D3 did not qualify as a resin within the meaning of component (1) as defined in Claim 1, because it had only a very low acid content (< 1 % acid, Claim 3 of D3) and an acid value no greater than 10 (Claim 1 of D3). Moreover, D3 disclosed neither 1,3,5-triazine carbamate crosslinking agents nor an aqueous dispersion promoting material as defined in Claim 1 which served to further improve the dispersion stability.

Although D1 and D2 described coating compositions comprising the above carbamate crosslinking agents and hydrogen containing resins, they failed to disclose the anionic water-dispersible resins of the application in suit and the aqueous dispersion promoting material.

Consequently, even the combination of these documents with D3, as envisaged by the Examining Division, would not result in a composition as defined by Claim 1, because the acrylic polymer according to D3 was not a reactive resin having both functionalities as required by Claim 1. The low content of acid groups would, even if completely neutralised, not be sufficient to disperse the hydrophobic carbamates in water.

Furthermore, it was found that the waterborne coating compositions which were insufficiently stable could be

stabilised by the aqueous dispersion promoting material (page 12, lines 22 to 34 of the application as originally filed). This had been demonstrated in Example 8 with a polymer containing hydroxyl groups in an amount at the lower extreme of the claimed range. It was the dispersion stability, which was to be improved by means of the claimed subject-matter, apart from the other aspects of the problem underlying the claimed subject-matter such as the reduction of VOC such as formaldehyde.

The Appellant concluded that - for the reasons given above - a person skilled in the art would indeed have expected difficulties in formulating a waterborne coating composition comprising the said resin and the said crosslinking agents and therefore he would not have combined any of the documents, ie D3 with D1 or D2. It followed that inventive effort was certainly needed to arrive at the specific combination of compounds as required by the claims under consideration.

VII. The Appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the set of claims 1 to 5 filed at the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. *Wording of the claims*
 - 2.1 Article 123(2) EPC

The amended wording of Claim 1 is based on the following parts of the application documents as originally filed: on Claims 1 and 9; page 2, line 31 to page 3, line 28; page 6, lines 9 to 14, 20 and 29 to 33; page 7, lines 22 to 32; page 8, lines 10 to 13; page 12, lines 22 to 24; page 13, lines 1 to 7, 18 to 20 and 29 to 31; and page 15, lines 9 to 11.

Claim 2 is based on original Claim 2, Claim 3 on original Claim 8, Claim 4 on Claims 31, 16 and 23 and page 6, lines 9 to 14 as originally filed, and Claim 5 is based on original Claim 32.

Consequently, the requirements of Article 123(2) EPC are met by the claims.

2.2 Article 84 EPC

The Board is satisfied that the wording of the claims as submitted at the oral proceedings is clear, because the essential ingredients of the claimed water-borne coating composition, ie components (1), (2) and (3), are now unambiguously defined in terms of their chemical compositions and in terms of their respective amounts. Thus, the amount of component (2) is defined in terms of the ratio of its carbamate groups to the amount of functionalities in component (1) reactive therewith and specified in the definition of component (1). The minimum amount of component (3) which is defined as a functional feature can be determined straightforwardly by the skilled person by comparing the stability against precipitation of dispersions containing this component or not.

Furthermore, these particulars are supported by the

description (see the references to the passages of the description referred to in section 2.1, above) and, in particular, the scope of these claims corresponds to the evidence for the solution of the problems indicated in the description which can be found in the examples of the application in suit. Thus, reference can be made to formulations C and H in Tables 1 and 5, respectively, and to Example 8.

The claims also meet the requirements of Rule 29(2) EPC (as entered into force on 2 January 2002).

3. *Article 83 EPC*

From the above findings, it is evident that the undue burden of experimentation due to the unclear and indefinite scope of the claimed subject-matter in the previous versions of Claim 1 has been lifted from the skilled person, and he is now given the information in a manner sufficiently clear and complete which enables him to carry out the invention with a reasonable expectation of success.

4. *Novelty*

None of the citations referred to in section II, above, discloses a composition comprising an aqueous dispersion promoting material according to the definition of component (3). In fact, novelty had already been acknowledged in the decision under appeal.

Consequently, the Board is satisfied that the requirements of Article 54(1) and (2) EPC are met.

5. *Problem and solution*

The application in suit concerns a waterborne coating composition.

- 5.1 In the decision under appeal, document D3 was considered to represent the closest state of the art. It discloses base neutralised stable aqueous dispersions of hydroxyl functional acrylic polymers and water based coating compositions made therefrom without adding external surfactants. The compositions can be cured to films by curing agents such as aminoplasts and polyisocyanate curing agents which primarily react with the reactive hydroxyl and amide groups of the polymer (column 1, lines 38 to 41). Since coating compositions which contain unreacted carboxylic groups after the reaction with the above curing agents have diminished water resistance, the content of carboxyl groups in the polymer is to be low (column 1, line 66 to column 2, line 16).

Therefore, the hydroxyl functional acrylic polymers are synthesised from a monomer component comprising at least about 50 percent of 2-hydroxyethyl acrylate and at least one other comonomer, but without the direct incorporation of acid functional monomers or the use of externally added surfactants, contain less than 1 % of acid, and have a number average molecular weight of from about 500 to about 4500 and an acid value in the range of from 1.5 to 10. They are prepared by free-radical polymerisation in the presence of a hydroxyl-free and primarily non-polar organic solvent. Depending on the amount of hydroxyethyl acrylate, the presence of a polar solvent such as isopropanol is preferred in order to facilitate the dispersion of the resulting polymer in water. The polymers can form a stable dispersion in water after neutralisation of the minor amount of

residual acid in the polymer with a minor amount of basic material such as an amine. They themselves are useful as surfactants in the preparation of aqueous dispersions and, in the claimed coating composition, become an integral part of the cured film without contributing to humidity and water sensitivity due to their capability of participating in the crosslinking reaction of the composition (Claim 1; column 2, lines 25 to 37; column 3, lines 2 to 11 and 46 to 50; column 4, lines 26 to 41; and column 5, lines 4 to 18).

Although reference is made to the advantageous properties of water based coating compositions in general, such as low VOC content, good coating properties such as ease of application, good gloss and general appearance (column 1, lines 59 to 66), a reduction of formaldehyde release is not mentioned in D3.

- 5.2 In line with the arguments provided by the Appellant (see section VI, above) and a number of passages in the description (page 2, lines 7 to 10 and 24 to 27 and page 12, line 22 to page 13, line 32), the technical problem can be seen in providing waterborne coating compositions which have a significantly improved (at least doubled) stability against the formation of precipitates and are curable with a reduced emission of VOC, in particular formaldehyde.

The solution for this technical problem proposed according to Claim 1 of the application in suit is a composition of a hydroxy-functional acrylic polymer, which, in particular, has a content of carboxylic groups higher than the polymer used in D3, a different crosslinker component and an aqueous dispersion

promoting material.

As demonstrated by the results of formulation C in Table 1, formulation H in Table 5 and in particular Example 8, the technical problem has effectively been solved. Moreover, the films obtained have a good hardness and high resistance against wear.

6. *Obviousness*

It remains to be decided whether the solution disclosed in the application in suit was obvious to a person skilled in the art having regard to the state of the art relied upon in the decision under appeal.

6.1 It is evident from the above considerations, that D3 does not provide an incentive to overcome the above technical problem, let alone in a way so as to arrive at something within the ambit of the subject-matter of Claim 1. In particular, there is no suggestion to (i) increase the content of carboxylic groups in the hydroxy-functional acrylic polymer, (ii) change the crosslinking agent and (iii) add the aqueous dispersion promoting agent.

6.2 Document D2 discloses curable compositions comprising (a) a triazine compound selected from (i) 2,4,6-triisocyanato 1,3,5-triazine, (ii) 2,4,6-tricarbamoyl 1,3,5-triazine, (iii) an oligomer of (i) or (ii), or (iv) a mixture of at least two of the above alternatives (i), (ii) or (iii), and optionally, (b) an active hydrogen-containing material and further optionally (c) a cure catalyst. Preferably, the material (b) contains at least two reactive carboxyl, alcoholic hydroxy, amine or amide groups, or a mixture of such groups. Preferred

examples for such polymers are a hydroxy-functional acrylic resin, a low molecular weight polyester polyol or an alkylene polyamine (D2: column 3, lines 34 to 60).

In practice, the curable compositions can be adapted for use in solvent-based, water-based, and powder coating compositions when the tricarbamoyl triazines are employed. Coating compositions comprising aqueous dispersions are particularly suited to application by electrodeposition (column 7, lines 29 to 36). Such a composition based on cationic polymeric material is exemplified in Example 9 of the document.

However, the document neither discloses the specific active hydrogen-containing anionic copolymer nor refers to the stability of aqueous dispersions against precipitation nor to an improvement of this feature by means of an aqueous dispersion promoting material as defined in Claim 1 of the application in suit. On the contrary, the composition it exemplifies is cationic.

- 6.3 Document D1 aims at curable compositions which avoid too high curing temperatures and show good properties such as good environmental etch resistance and absence of formaldehyde emissions during cure (page 2, paragraphs 1 to 3 and line 26).

The composition is based on curable "polyfunctional hydroxy group containing materials" conventionally used in aminoresin coatings, ranging from polyols and hydroxy-functional acrylic resins to hydroxy-functional polyurethane prepolymers and products derived from the condensation of epoxy resins with an amine. As an example, a commercial acrylic resin is given based on 50% styrene, 20% hydroxypropyl methacrylate and 30%

butyl acrylate having a hydroxyl number of 140, an equivalent weight of 400 and a number average molecular weight of 4000. No reference to anionic hydrophilising groups and/or surface active properties is made (page 3, line 4 *et seq.*). The composition is cured by means of a triazine tris-carbamate crosslinking agent and an acid cure catalyst or by means of a combination of an aminoresin crosslinking agent and the triazine tris-carbamate and the catalyst. These compositions have low cure temperatures and low formaldehyde emissions during cure and the films obtained therefrom exhibit higher environmental etch resistance than conventional aminoresin derived coatings (page 2, lines 41 to 54).

Optionally, a liquid medium may be contained in the composition. Amongst a broad range of organic solvents, water is mentioned as one liquid medium for preparing dispersions, emulsions, invert emulsions or solutions (page 7, lines 11 to 17). In all the examples, the compositions are water-free and contain m-xylene and butyl acetate.

D1 does not contemplate the improvement of the stability of a waterborne coating composition. Therefore, it cannot contribute to the solution of the relevant problem mentioned above.

6.4 In summary, in none of the documents cited in the decision under appeal, has the improvement of the stability of a waterborne coating composition against precipitation been contemplated, let alone by means of a composition comprising the three components (1), (2) and (3) as defined in Claim 1.

6.5 It follows that the subject-matter of Claim 1 does not

arise in an obvious way from the cited state of the art. Consequently, Claim 1 involves an inventive step.

7. Since the other Claims 2 to 5 include the same features and limitations as Claim 1, they are supported by the patentability of this claim and are thus also allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the first instance with the order to grant a patent on the basis of claims 1 to 5 filed at the oral proceedings, after any necessary consequential amendment of the description.

The Registrar:

The Chairman:

E. Görgmaier

R. Young