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Bezeichnung der Erfindung: Method for preparing a two-component polyurethane
Title of invention: lacquer atomisable from a pressurised container
Titre de l'invention :

Klassifikation / Classification / Classement : C09D 3/72

ENTSCHEIDUNG / DECISION

vom / of / du 20 January 1988

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet : Hermann Wiederhold GmbH

Einsprechender / Opponent / Opposant : Kurt Vogelsang GmbH

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Article 56

Kennwort / Keyword / Mot clé : Inventive step (no) - process of trial and error, no inventive selection

Leitsatz / Headnote / Sommaire

Case Number : T 259/85

DECISION
of the Technical Board of Appeal 3.3.2
of 20 January 1988

Appellant : (Opponent)	KURT VOGELSANG GmbH D-6954 Haßmerstein
Representative :	Wilhelm, Hans-Herbert, Dr.-Ing. Wilhelm & Dauster Patentanwälte Hospitalstrasse 8 D-7000 Stuttgart 1
Respondent : (Proprietor of the patent)	HERMANN WIEDERHOLD GmbH Postfach 940 D-4010 Hilden/Rhld.
Representative :	Patentanwälte TER MEER - MÜLLER - STEINMEISTER Mauerkircherstrasse 45 D-8000 München 80
Decision under appeal :	Decision of the Opposition Division of the European Patent Office dated 28 August 1985 rejecting the opposition filed against European patent No. 0 030 840 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : P. Lançon
Members : S. Schoedel
E. Persson

Summary of Facts and Submissions

I. European patent 30 840 was granted on 18 May 1983 with 8 claims in response to European patent application 80 304 447.8 filed on 9 December 1980 claiming priority of an earlier application of 14 December 1979. The main claim reads:

"Method for the preparation of a product, atomisable from a pressurised atomising container, which is a two-component polyurethane lacquer in which a prepolymer comprising a copolymer containing 1 to 7 wt.% of hydroxyl groups based upon styrene, vinyltoluene, methacrylic acid esters and/or acrylic acid esters with 1 to 8 carbon atoms in the alcohol residue as well as hydroxyalkyl acrylates and/or methacrylates with 2 to 4 carbon atoms in the alcohol residue, or mixtures of such copolymers, as the first component, which may contain also inorganic and/or organic pigments, flow agents, anti-settling agents, thixotropic agents, reaction accelerators, matting agents and other additives, is dissolved in an organic solvent and is filled together with a propellant into a first pressurised container and in which furthermore a hardener based upon a polyisocyanate containing 10 to 25 wt.% NCO-groups, dissolved in an organic solvent, is filled as second component together with a propellant into a second pressurised container, and wherein for preparation of the ready-for-use lacquer the one component is transferred from its pressurised container into the pressurised container of the other component and mixed with the said other component, characterised in that

(i) for a prepolymer containing up to 2 wt.% hydroxyl groups there is used as propellant propane, butane, monofluorotrichloromethane, difluorodichloromethane, dimethyl ether, nitrogen or mixtures thereof and for a prepolymer with more than 2 wt.% hydroxyl groups there is used as propellant dimethyl ether or a mixture of dimethyl ether, propane, and/or butane,

(ii) for the hardener, nitrogen is used as propellant, and

(iii) the amounts of the prepolymer present in the first pressurised container and of the polyisocyanate present in the second pressurised container are so proportioned that on combination of the two components a mixture results which contains 60 to 96 wt.% prepolymer and 4 to 40 wt.% hardener."

II. On 27 January 1984 the Appellant filed an Opposition citing, **inter alia**,

(3) US-A-3 343 718

and requesting the revocation of the patent for lack of inventive step.

III. By the decision dated 28 August 1985 the Opposition Division maintained the patent unamended; it held that the invention clearly met the requirements of Article 56 EPC.

IV. On 19 October 1985 an appeal was lodged against this decision by the Appellant; the appeal fee was paid simultaneously. The statement setting out the grounds of appeal was submitted on 20 December 1985. The Respondent's reply was received on 15 September 1986.

Later on by communication dated 9 November 1987 the Rapporteur, on behalf of the Board, informed the parties that

(2) FR-A-1 376 585

mentioned in the search report, might be of some relevance in evaluating inventive step.

An oral hearing was appointed for 20 January 1988.

V. The Appellant argued in the written proceedings and at the oral hearing substantially as follows:

As far as the hardener and the hydroxy-acrylate-copolymer were concerned, the claimed two-component polyurethane (PUR) lacquer was nothing but an abridged version of the teaching of the document

(1) DE-A-1 247 006.

Such lacquers were now being proposed in the form of aerosol products packaged in mother-daughter containers, the alleged peculiarity being the specific propellants applied.

Means and methods for using various polymerizable materials such as urethane-prepolymers to form sprayable coating compositions when combined with agents, which may cause polymerization, were well known in the art (3). As exemplified in (3), starting from Desmophen$^{(R)}$ 900 (polyester polyol) and Desmodur$^{(R)}$ TH (polyisocyanate) and using R11/R12 and propane as propellants one arrived at a cured PUR product; the two components were packaged in pressurised containers of the mother-daughter type and combined before use. It was exactly this system which was copied by the Respondent.

As to the alleged selection of the propellants: it was true that in (3) a list of chemical compounds was given suitable for the aforementioned purposes; however, it was clear that those gases which were reactive, toxic, flammable or malodorous had to be sorted out first; what remained to be tested were the common propellants such as hydrocarbons (propane, butane) fluorocarbons (R11, R12) and particularly nitrogen - all of them being applied in the Respondent's lacquer compositions. Solvency problems with the polymers, if there were any, could have been easily recognised and also overcome in the same way simply by testing the relevant solutions and propellants. Apparently no problems occurred either during storage or upon application of the lacquer compositions from (2), which comprised Desmophen$^{(R)}$ 2300 (polyester polyol), Desmodur$^{(R)}$ L (polyisocyanate) and R11/R12 as the propellants.

In the absence of information on experimental details the comparative data submitted must be considered to be irrelevant.

Altogether, no inventive matter could be seen in the claims of the disputed patent.

VI. The Respondent contested the applicant's conclusions and put forward essentially the following arguments:

The invention was based on the selection of a particular combination of propellant and prepolymer on the one hand and of propellant and hardener on the other hand. While the propellant for the hardener was consistently nitrogen, the propellants for the prepolymers varied in dependency on the OH-contents of the acrylate copolymers. These combinations achieved unexpected advantages in terms of the storage stability of the two active ingredients, when kept separately in pressurised containers and in terms of the pot-life of the compositions which were formed upon mixing. Ultimately the resulting lacquers were of superior quality to those obtainable with hydroxy polyesters or polyethers (1). It was the Respondent's merit to have recognised how the difficulties arising could be overcome.

The method and the lacquer compositions claimed were not made obvious by any of the cited documents. In (3) compositions of the general type with which the present invention was concerned were disclosed; in an actual example the preparation of a Desmophen$^{(R)}$ 900/ Desmodur$^{(R)}$ TH based PUR-adhesive was described; even if adapted to the two-component PUR lacquer of the present claims the respective formulation would not arrive at the invention. Moreover, the test results submitted on 18 October 1984, made it clear that example V of (3) was completely inoperable.

No useful information as to a solution of the existing problems could be taken from (2), which document related to one-component PUR lacquers; with these, polymerization was effected between NCO-groups and water; this reaction started as soon as the Desmophen$^{(R)}$ 900/Desmodur$^{(R)}$ TH - prepolymer was atomized and contacted with air humidity.

VII. The Appellant requests that the decision under appeal be set aside and the patent revoked.

The Respondent requests that the appeal be dismissed and the patent maintained unamended.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is therefore admissible.

2. The patent-in-suit concerns a method for the preparation of a two-component PUR lacquer, which is available in the form of an aerosol product.

3. Two-component PUR lacquers of this type are already known from (1), which document is referred to in the introductory part of the present description. The OH-containing acrylate-styrene-copolymers (having an OH content of 0.01 to 10%, preferably from 2 to 10%) and the polyisocyanates (e.g. the condensation product of 3 Mol TDI and 1 Mol trimethylolpropane, having an NCO content of about 17%) are dissolved in an organic solvent (such as an acetic acid ester, aromatics, halogenated hydrocarbons), mixed together and after a short period of pre-reaction applied in the usual manner to the substrate. The pot-life in one of the examples is indicated as being 15 hours.

These lacquers display good application and film properties, such as mechanical and chemical resistance, and are suited, therefore, both as a first coat and as a repair lacquer.

According to the Respondent, attempts to make available such lacquers as an aerosol product have remained unsuccessful, because both the individual components in separate pressurised containers and the mixture of the two components in a single aerosol container have an inadequate storage stability for practical purposes. Up to now these lacquers have been applied by a spray gun (present description, page 2, paragraph 2).

4. The technical problem underlying the patent-in-suit vis-à-vis this prior art can be seen in making available an aerosol type two-component PUR lacquer, the components of which are capable of being stored separately in their own pressurised containers and when being combined in a single dispenser give a mixture having a pot-life adequate for practical use.

4.1 The Respondent's proposal for a solution of the said problem is set out in Claim 1 of the patent-in-suit. Briefly it consists in providing

a basic lacquer component (which is an acrylate copolymer having 1-7 wt.% OH groups; OH-acrylate prepolymer); and

a hardener component (which is a polyisocyanate having 10-25 wt.% NCO groups);

each of the components being separately dissolved in an organic solvent and filled together with a propellant into a separate pressurised container; and

transferring the one component from its container into the container of the other component in order to prepare the ready-for-use mixture;

whereby the propellant for the basic lacquer component is dependent on the OH content of the prepolymer (the sub-ranges being 1-2 and 2-7 wt.%), while the propellant for the hardener is always nitrogen.

4.2 The arrangement of the two pressurised containers follows the principle of the mother-daughter package which means that the contents of the one container being under a higher pressure are transferred to the other container, which has also the function of the spray can, and the liquid mixture formed is kept dispensible over a predictable period following combination of the contents of the two containers.

4.3 Having regard to the results obtained in the Examples of the patent-in-suit, the Board is satisfied that the aforementioned technical problem has been plausibly solved. The Appellant's criticism of the additional experimental data on present Examples 2 and 3, submitted with letter of 18 October 1984 by the Respondent, is not substantiated and need not, therefore, be considered.

5. The claimed method for the preparation of a sprayable two-component PUR lacquer is not to be found in any of the prior art documents so that the subject-matter of Claim 1 of the patent-in-suit is acknowledged to be novel under Article 54 EPC. Since the Appellant has never raised the question of novelty no further discussion of the matter is necessary.

6. Turning now to inventive step it has to be examined whether the present Claim 1 satisfies the requirements of Article 56 EPC. Confronted with the aforementioned

technical problem the expert in the field of resin coating is considered to be aware of the following facts which are reflected for example by (1):

6.1 The NCO-hardener and the OH-acrylate prepolymer after being combined and applied in the form of the liquid composition are converted to a solid film of PUR lacquer.

The OH content of the acrylate prepolymer is important for the calculation of the amount of the NCO-hardener theoretically necessary for crosslinking; as a rule the NCO/OH ratio should be about 1.

Crosslinking of the system is influenced by the reactivity of the individual components, especially of the NCO-hardener but also by that of the prepolymer and to some extent also by the nature of the solvent used.

The pot-life of the liquid composition and consequently the period of time during which the said system can be used are limited (the pot-life is defined as the length of time before the liquid composition, at room temperature, has polymerized to an unworkable state achieved by precipitation or gel formation).

Gel formation should be observed via viscosity tests. The viscosity of the liquid system is therefore of high a relevance.

During storage and when handling the NCO-hardener care must be taken to keep away humidity; however, no specific precautions need to be taken with the OH-acrylate-prepolymer.

Spray guns usually run with compressed air, which contains 80% nitrogen.

Lacquers formed from NCO-hardenable OH-polyacrylates, which are addition polymers, are superior in quality (especially as to their chemical resistance) to those based on OH-containing polyesters or polyethers, which are condensation products ((1), column 3, paragraph 6).

6.2 Atomisable PUR lacquers having a pot-life of "a longer period of time" are known from (2). They are obtained by dissolving Desmophen$^{(R)}$ 2300 (a polyester polyol based on adipic acid, diethyleneglycol and a triol having 1.5 wt.% OH groups) and Desmodur$^{(R)}$ L (an aromatic polyisocyanate having about 17 wt.% NCO groups) in a higher ketone (methyl ethyl ketone, ethyl amyl ketone) the propellants being R11/R12 (monofluorotrichloromethane/difluorodichloromethane). Further potential propellants mentioned are aliphatic hydrocarbons. These formulations polymerise when being dispensed and contacted with air humidity by reaction of the excess NCO groups with water. This type of polymerisation - according to the Respondent - clearly points to **one** component and not to **two** component PUR lacquers, the latter being the subject-matter of the patent-in-suit alone.

6.3 A dispensing system of broad applicability, which relates to sprayable coating compositions having a polymerizable base is disclosed in (3). This system includes a pressurized "catalyst container" and a "dispensing container" arranged as a mother-daughter-package. The two active constituents are kept apart and stored separately under the protective atmosphere of the propellant - thus excluding air and/or moisture and avoiding premature polymerization. Before dispensing the lacquer, a composite composition having a predetermined polymerization period is formed by discharging the contents of the container held under a higher pressure to the other container which is the aerosol dispenser. The gas at the higher pressure ensures completeness of the transfer. A typical example is directed to a sprayable PUR adhesive which is based on a Desmophen$^{(R)}$ 900/Desmodur$^{(R)}$ TH mixture (polyester polyol/TDI derived polyisocyanate).

An element of vital importance for the functioning of such aerosol systems is the nature of its propellant. In (3) there is a list indicating a number of compressible as well as liquefiable gases appropriate for use in the spray dispenser and in the supply container. Compressible gases of this type are, e.g. nitrogen, nitrous oxide, carbon dioxide and air (3, column 2, last paragraph).

Liquefiable gases having vapour pressure suitable for the purpose are saturated aliphatic hydrocarbons such as propane, butane and isobutane, and fluorinated or chlorinated or fluorochlorinated hydrocarbons such as 1,1-difluoroethane, 1-monochloro-1,2-difluoroethane, R11, R12, 1,2-dichloro-1,1,2,2-tetrafluoroethane and the like. Under atmospheric conditions these are in the gaseous state but at higher pressure they condense to form a liquid (3, column 3, paragraph 3).

It is clear that the propellant used must be broadly speaking compatible with the respective components with which it is in contact. Any interaction between the various constituents must be at a minimum. Incompatibility may be caused with non-polar liquid gases if present in too high a concentration and may result in polar polymerizable materials and the polar resin coming out of solution.

Aerosol mixtures comprising liquefied propellants retain their initial pressure even after use. The proportion expelled is compensated by subsequent evaporation of the liquid phase. In contrast to this, compressed gases are less soluble in the liquid composition and therefore do not greatly influence the solvency characteristic of the liquid phase. The function is more that of a scavenger or transfer gas. Upon use, the pressure of such systems decreases.

7. Considering the prior art teachings and common general knowledge the person skilled in the art was in the Board's view able to solve the technical problem without exercising inventive skill.

7.1 Thus, it was obvious to provide the mother-daughter container arrangement (3) with all the concomitant advantages for practical use sought for by the skilled person and to adapt the PUR lacquer compositions known from (1) for being dispersed from that arrangement. There is a close relationship between the Desmodur$^{(R)}$ TH/Desmophen$^{(R)}$ 900 based PUR adhesive (3) and Desmodur$^{(R)}$ L/OH-acrylate-prepolymer two component PUR lacquers as to the mechanism of the NCO/OH cross-linking reaction and there is no fundamental change in properties (especially alkali resistance) of the corresponding PUR lacquers, whether an OH-acrylate prepolymer is used alone, or as a blend with OH-containing polyesters or polyethers in the respective compositions.

7.2 By using a process of trial and error the way was further open to arrive at sprayable, NCO-hardenable OH-polyacrylate two component lacquers having an extended pot-life (of at least to 24 hours; present description, page 4, line 49). By means of conventional tests of various compositions in the normal manner using starting materials as disclosed in

(1) and measuring the viscosity, it would have been possible on the basis of normal professional skill to establish the pot life of the resulting compositions. Mixtures unsuitable for the intended use could thus have been singled out.

7.3 As regards the propellants, a closer analysis of the list of gases set out in (3) reveals that most of them could have been disregarded for the reasons given by the Appellant under Section V. Only a few have actually been applied in the examples of the prior art document, most of them being directed to similar coating systems. These propellants are R11/R12 (including R12), hydrocarbons C_3, C_4), nitrogen and carbon dioxide in that order of frequency, the first two being liquefiable gases and the last two compressible gases and all of them being known to be freely, commercially available.

7.4 Thus, the number of propellants in (3) being of real interest in the actual context was relatively small. It would have been quite a normal approach not requiring inventive ingenuity to have regarded each of the propellants just mentioned as suitable for spray testing.

These considerations are reflected in one particular embodiment falling within the scope of present Claim 1 where the propellant is nitrogen for both the NCO-hardener and the (1-2 wt.%) OH-acrylate prepolymer.

As could have been expected nothing extraordinary occurs either in the storage stage or in the ready-for-use stage when nitrogen is present as the sole propellant and - contrary to the Respondent's view - no inventive selection can therefore be recognised.

To determine the other features necessary for the functioning of the spray system, such as the viscosity of the ultimate composition to be sprayed or the appropriate prepolymer/NCO-hardener proportion in the mixture was merely a matter of routine (present description, page 3, last paragraph).

As to the figure "2 wt.%", which appears under (i) of present Claim 1 in connection with the OH content of the acrylate prepolymer, it seems that this figure is nothing but a guide value already known as being of some significance for the preparation of high performance lacquers rather than a critical threshold beyond which propellants such as hydrocarbons definitely cannot be applied (1, column 3, line 49).

7.5 As the Respondent's only request was the maintenance of the patent-in-suit in unamended form, it can be left undecided whether Claim 1 embraces subject-matter which might possibly be patentable.

8. The Respondent's attempt to rework that PUR adhesive example of (3) failed (letter dated 18 October 1984). The stated reason for this was that no solution, dispersion or emulsion was formed upon mixing the reactants as prescribed (Desmophen$^{(R)}$ 900/acetone/R11, R12; Desmodur$^{(R)}$ TH/propane). The Appellant has not put forward any counter-evidence.

As is known, a consistently homogeneous liquid system is, however, a prerequisite for further handling such materials in aerosol containers ((3), column 2, paragraph 2, column 3, paragraph 2). All that can be concluded from that example is, that the instructions given are either incomplete or wrong. The negative result of the test as it stands is, therefore, of minor importance for assessing inventiveness and can by no means question the general validity of the concept taught in (3), which is documented by numerous other examples.

9. It is therefore the Board's judgement that the subject-matter of Claim 1 of the patent-in-suit does not involve an inventive step as required by Article 56 EPC and consequently the claim is not allowable. Sub-claims 2-8, being dependent on an invalid Claim 1, are not allowable either.

Order

For these reasons, it is decided that:

1. The impugned decision is set aside.

2. The patent is revoked.

The Registrar: The Chairman: