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Veröffentlichung im Amtsblatt	~~ja~~/Nein
Publication in the Official Journal	~~Yes~~/No
Publication au Journal Officiel	~~Oui~~/Non

Aktenzeichen / Case Number / N° du recours : T 36/89 - 3.3.1

Anmeldenummer / Filing No / N° de la demande : 80 301 861.3

Veröffentlichungs-Nr. / Publication No / N° de la publication : 0 022 617

Bezeichnung der Erfindung: Prepolymers, polyisocyanate compositions prepared
Title of invention: therefrom and their use in the preparation of
Titre de l'invention : polyurethane foams

Klassifikation / Classification / Classement : C0G 18/48

ENTSCHEIDUNG / DECISION
vom / of / du 10 July 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet : Imperial Chemical Industries PLC

Einsprechender / Opponent / Opposant : OI Bayer AG
OII BASF AG
OIII Dow Chemical Company

Stichwort / Headword / Référence : Polyurethane foams/ICI

EPÜ / EPC / CBE Article 56

Schlagwort / Keyword / Mot clé : "Inventive step (confirmed) - cited prior art not relevant to the underlying technical problem"

Leitsatz / Headnote / Sommaire

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Case Number : T 36/89 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 10 July 1990

Appellant : (Opponent OII)
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Respondent : (Proprietor of the patent)
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Decision under appeal :
Interlocutory decision of the Opposition Division of the European Patent Office dated 28 November 1988 concerning maintenance of European patent No. 0 022 617 in amended form.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
M. Auz Castro

Summary of Facts and Submissions

I. The mention of the grant of European patent No. 0 022 617, in respect of European patent application No. 80 301 861.3 filed on 4 June 1980 and claiming priority of 11 July 1979 from a prior application filed in the United Kingdom, was announced on 15 February 1984 (cf. Bulletin 84/7).

II. Notices of opposition were filed on 24 September 1984, 26 October 1984 and 15 November 1984 requesting the revocation of the patent on the grounds that its subject-matter lacked novelty and did not involve an inventive step. Opponent OII also maintained that its subject-matter extended beyond the content of the application as filed and Opponent OIII alleged that the patent did not disclose the invention in a manner sufficiently clear and complete for it to be carried out by the skilled person.

III. By an interlocutory decision dated 28 November 1988, the Opposition Division maintained the patent in amended form on the basis of Claims 1 to 4 filed on 11 January 1988.

The Opposition Division held that the proposed solution to the problem of providing polyisocyanate compositions having improved storage stability was novel and inventive. The Opposition Division also concluded that the other objections raised by the Opponents were unfounded.

IV. Appeals were lodged against this decision on 7 and 25 January 1989 and the appeal fees duly paid. Statement of grounds of appeal were filed on 14 and 21 March 1989. Oral proceedings, to which all parties were duly summoned, but at which Appellant OII was not represented, were held on 10 July 1990.

Of the documents cited during the opposition proceedings, only the following ones were referred to during the appeal proceedings:

(5) DE-A-2 513 796
(6) GB-A-2 007 690
(9) US-A-4 137 200 and
(11) US-A-3 793 241.

In his written submissions, Appellant OII alleged that the invention could not be carried out throughout the disclosed scope since the ratio of prepolymer to crude diphenlymethane diisocyanate (MDI) and the isocyanate content of the prepolymer were not defined. This Appellant also contended that the invention was not disclosed in a manner sufficiently clear and complete for it to be carried out by the skilled person and that it was not inventive in the light of the disclosure of document (6).

In his written submission and during the oral proceedings, Appellant OIII argued that the claimed subject-matter did not involve an inventive step in the light of the disclosure of document (5) combined with that of document (9) and/or document (11). This Appellant also alleged that the disputed patent was insufficient insofar as it would appear that undisclosed measures were necessary to obtain a polyol in which the ethylene oxide (EO) residues were distributed in a random manner and which gave the desired stability of the polyisocyanate composition comprising the prepolymer prepared from it and crude MDI.

V. In his reply to the statements of grounds of appeal and during the oral proceedings, the Respondent argued that the invention resided in finding that the use of random copolymers in the preparation of prepolymers provided

compositions with desirable new properties. The Respondent also maintained that the results of the tests filed on 13 November 1987 and those filed with his response to the statements of grounds clearly refuted Appellant OIII's allegation that the storage stability of the polyisocyanate composition was unsatisfactory, regardless of the nature of the polyol used to prepare the prepolymer.

The Respondent further submitted that the expression "distributed in a random manner" was a "clear and complete" disclosure to the skilled person in view of the state of the art.

VI. The Appellants requested that the decision under appeal be set aside and the patent revoked. The Respondent requested that the patent be maintained on the basis of Claims 1 and 2 submitted during oral proceedings. These claims, after the correction of a clerical error in Claim 2, read as follows:

1. A polyisocyanate composition comprising a blend of (a) a prepolymer with an NCO content of up to 29% by weight prepared by reacting a polyoxyalkylene diol or triol with an excess of an isomeric mixture of diphenylmethane diisocyanates, mainly consisting of the 4, 4' and 2, 4' isomers, the isocyanate to hydroxy groups ratio being at least 4:1, said polyoxyalkylene diol or triol having a hydroxyl equivalent weight in the range 1000 to 2000 and containing propylene oxide and ethylene oxide residues, the latter being distributed in a random manner throughout the polyoxyalkylene chain or part thereof and constituting from 50 to 85% by weight of the total alkylene oxide residues, said polyoxyalkylene diol or triol being obtainable by reacting a mixture of ethylene and propylene oxides containing at least 50% by weight of ethylene oxide with a compound containing two or

three active hydrogen atoms in the molecule, and (b) a crude diphenylmethane diisocyanate containing from 40 to 80% by weight of diphenylmethane diisocyanates, the remainder being largely polymethylene polyphenyl polyisocyanates of functionality greater than two.

2. A method for the preparation of cold cure polyurethane foams which comprises intimately mixing:

(a) a polyisocyanate composition according to Claim 1;

(b) a polyoxyalkylene polyol containing oxypropylene and optionally oxyethylene residues;

(c) water, and

(d) a catalyst for foam formation, optionally in the presence of other conventional polyurethane foam ingredients.

VII. At the conclusion of the oral proceedings, the Board's decision to maintain the patent in amended form was announced.

Reasons for the Decision

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

2. There are no formal objections under Article 123 EPC to the present claims since they are adequately supported by the original disclosure and do not extend the protection

conferred. Thus, Claim 1 is based on granted Claim 3 in combination with page 2, lines 33 and 34, 42 to 47 and 51 to 54 of the printed patent specification (cf. also page 2, lines 8 to 14, page 3, lines 3 to 6 and 20 to 29, page 4, lines 6 to 11 and page 5, lines 16 to 20 of the published patent application). Claim 2 is supported by original Claim 1 and Claim 5 as granted in combination with page 6, lines 16 and 17 and page 3, lines 54 and 55 of the published patent application and printed patent specification respectively.

3. The Board is satisfied that the disputed patent discloses the invention in such a manner that it could be carried out by the skilled person.

3.1 With respect to the random copolymers of propylene oxide and ethylene oxide used in the preparation of component (a) of the polyisocyanate composition claimed in Claim 1, such copolymers are commercially available (cf. Respondent's experimental report filed on 7 August 1989 in response to the grounds of appeal) and can be distinguished from block copolymers by means of C^{13}-NMR spectroscopy. Therefore, the skilled person would have no difficulty in selecting polyoxyalkylene diols or triols falling within the definition given in the present Claim 1. Moreover, one method for their preparation is described and exemplified in the disputed patent.

3.2 Although the ratio of the prepolymer to crude MDI in the polyisocyanate composition is not specified, in the Board's judgement suitable ratios, other than those illustrated in Examples of the disputed patent, could be found by the skilled person without undue effort.

4. The patent in suit relates to polyisocyanate compositions and their use in the preparation of cold cure polyurethane foams.

4.1 Although numerous documents have been cited, in the Board's view none of them can be considered to represent closest prior art in the light of which the technical problem underlying the patent in suit should be formulated. The majority of the cited documents are concerned with the storage stability of polyisocyanate compositions and not with the principal problem addressed by the disputed patent of improving the indentation resistance of freshly demoulded cold cure polyurethane foams. Document (9), which discloses the foaming of specified polyisocyanate compositions using a mixture of ice and water (cf. column 8, lines 60 to 64), refers to those properties of the foams, such as resistance to compression set and resilience (cf. column 4, lines 13 to 19), which are determined on foams which have been allowed to stand for about 24 hours after demoulding.

Document 8 (GB-A-2 021 605), which was regarded by the Opposition Division as representing the closest state of the art, was published on 5 December 1989, i.e. after the validly claimed priority date of the disputed patent of 11 July 1979.

4.2 In the absence of any relevant prior art relating to the problem of the markability of freshly demoulded cold cure foams, the Board has adopted the following approach in order to formulate the technical problem underlying the disputed patent.

The cold cure moulding system for the production of polyurethane flexible foams was developed to provide a more economical manufacturing process with respect to process

times and energy requirements. The increase in productivity resulting from the shorter process times is achieved by a decrease in the moulding and demoulding times. However, it was found that compression marks, which could occur during demoulding, did not always disappear and the resulting surface defects led to the rejection of the product.

In the light of the above, the Board sees the technical problem underlying the patent in suit in providing polyisocyanate compositions tailored for in the preparation of cold cure polyurethane foams in which, having regard to the intended application, a satisfactory compromise is reached between the resistance to compression indentation immediately after demoulding (markability) and the mechanical properties of the fully cured products.

According to the patent in suit, this technical problem is essentially solved by providing the polyisocyanate compositions of Claim 1 and their use in the manufacture of cold cure polyurethane foams according to the process of Claim 2.

In view of the fact that the results of the tests filed by the Respondent on 23 May 1986 and 13 November 1987 are credible, the Board is satisfied that this technical problem is solved.

4.3 The Board considers that Appellant's OIII objection that the composition of the MDI, which is reacted with the random polyol, is not specified in the experimental report filed on 13 November 1987 to be groundless, since in the preliminary remarks to this report it is stated that the experiments presented in the Appellant's report dated 17 May 1987 were repeated with certain exceptions. Since the composition of the MDI was not listed among these

exceptions, it is quite clear that the tests were carried out using MDI containing 80 parts of the 4, 4'-isomer and 20 parts of the 2, 4'-isomer.

4.4 In the absence of any prior art document disclosing the manufacture of a cold cure polyurethane foam from a polyisocyanate composition comprising (a) a blend of a prepolymer prepared by reacting a block copolymer of ethylene oxide and propylene oxide with an isomeric mixture of MDI and (b) crude MDI, it is not necessary to consider the comparisons drawn in the various experimental reports between the properties of these cold cure foams and the ones obtained from the present isocyanate compositions.

4.4 With regard to the question of the storage stability of the claimed polyisocyanate compositions, the Board takes the view that this is of secondary importance and cannot be considered to represent the technical problem underlying the disputed patent. During the oral proceedings, the Respondent admitted that polyisocyanate compositions were known, the storage stability of which was better than that of the present composition (cf. the composition disclosed in Example 1 of document (5)). Moreover, in view of the results of the storage tests submitted by Appellant OIII on 27 May 1987, it is doubtful whether, if the problem addressed was concerned with storage stability, the Board would have regarded it as having been solved. Since the technical problem is to be seen as the one defined above, the Appellant OIII's argument that in the light of the combined disclosure with respect to storage stability in documents (5), (9) and (11) a "one-way street" situation existed, cannot be entertained.

5. After examination of the cited prior art, the Board is satisfied that the claimed subject-matter is novel. Since novelty is no longer in dispute, it is not necessary to consider this matter in detail.

6. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.

6.1 Document (5) discloses polyisocyanate compositions comprising (A) from 30 to 85% by weight of a mixture of methylene bridged polyphenyl polyisocyanates containing 30 to 65% by weight of MDI, (B) from 5 to 70% by weight of a reaction product of 1 molar proportion of MDI with 0.005 to 0.6 molar proportions of an alkylene or polyoxyalkylene diol and (C) from 0 to 50% by weight of MDI or a mixture of isomers thereof; the proportions of (A), (B) and (C) being such that the total amount of MDI present in the compositions, including that pre-reacted with the diol, comprises from 55 to 95% by weight of the compositions and their use in the manufacture of polyurethane foams (cf. Claims 1 and 10). According to the second complete paragraph on page 7 (original numbering), the diol may be a simple diol such as, for example, ethylene glycol, propylene-1,3-glycol, butylene-1,3-glycol or hexylene glycol or a polyoxyalkylene diol such as a polyethylene glycol or polypropylene glycol having a molecular weight of, for example, 100 to 1000.

This document, which is mainly concerned with the problem of storage stability, is wholly silent with respect to both copolymers of ethylene oxide and propylene oxide in the component (B) and cold cure foams. In the absence of any disclosure in these respects, it would be of no assistance to the skilled person seeking to solve the problem of minimising the markability of freshly demoulded cold cure foams.

6.2 Document (9) describes polyisocyanate compositions comprising isocyanate capped hydrophilic polyoxyethylene diols having an ethylene oxide content of at least

40 mole %, isocyanate capped polyols having hydroxide functionalities in the range 3 to 8 prior to capping and polyisocyanates having isocyanate functionalities in the rnage 2 to 2.8 (cf. Claim 9). The ratio of isocyanate groups to hydroxyl groups used for capping the polyoxyethylene diols is between about 1:1 to 4:1 (cf. column 2, lines 31 to 34).

These compositions may be foamed by the addition of 6.5 to 390 moles of water, possibly in the form of a mixture of ice and water, for each mole of unreacted isocyanates (cf. Claim 1 and column 8, lines 60 to 64).

In order to improve certain properties such as low temperature flexibility, resistance to compression set and resiliency, it is possible to incorporate various amounts of relatively hydrophobic comonomers into the ethylene oxide based polymerisation products. Provided the copolymers remain hydrophilic, comonomers such as propylene oxide or butylene oxide may be copolymerised as a random or block copolymer (cf. column 4, lines 10 to 34).

However, the above-mentioned properties are in respect of the fully cured product and, therefore, would not be relevant to the skilled person interested in the problem of the markability of freshly demoulded cold cure polyurethane foams. The teaching of this document, either alone or combined with that of document (5), would not provide the skilled person with any indication which would lead him to the claimed solution to the technical problem underlying the disputed patent.

6.3 Document (11) discloses the preparation of prepolymers by reacting polyoxyalkylene polyols, such as polyethylene glycols or oxyethylene-oxypropylene block or random

copolymers, with polyisocyanates (cf. column 6, lines 44 to column 7, line 8). The only isocyanate specifically mentioned in connection with these prepolymers is toluene diisocyanate. The polyfunctional aromatic isocyanate compounds referred to in column 7, lines 16 to 33, including MDI and crude MDI, are said to be suitable for blending with the prepolymer and not for forming the prepolymer.

In the absence of any reference to the indentation of freshly demoulded foams, this document would not suggest to the skilled person that the solution to the problem underlying the disputed patent lies in preparing cold cure foams from the polyisocyanate compositions claimed in the present Claim 1.

6.4 Document (6) is concerned with storage stable aqueous solutions of polyurethanes obtained by admixing crude MDI with less than the stoichiometric amounts of polyethylene glycols (M.Wt. 600 to 3000) or polypropylene glycols capped with 15 to 85% ethylene oxide (M.Wt. 1000 to 3500) and, within a short time of completing the admixture, but during the period where said mixture is completely soluble in water, dissolving said mixture in water (cf. Claim 1). In the absence of any reference to cold cure polyurethane foams this reference is completely irrelevant with respect to the technical problem underlying the disputed patent.

7. Therefore, in the Board's judgement, the claimed solution to the technical problem underlying the patent in suit is not obvious in the light of the cited prior art. The subject-matter of Claims 1 and 2 involves an inventive step and the claims are, therefore, allowable.

8. The Board has examined the document DE-A-2 425 657, which was cited for the first time by Appellant OII in his

statement of grounds of appeal, and decided that consideration of this document would not lead to a different conclusion with respect to the patentability of the subject-matter of the disputed patent. Therefore, in the exercise of its discretion under Article 114(2) EPC, the Board has disregarded this late filed document.

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 maintain the patent on the basis of Claims 1 and 2 submitted during oral proceedings and a description to be brought into agreement with the amended claims.

The Registrar: The Chairman:

M. Beer K.J.A. Jahn