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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 206/84

Anmeldenummer / Filing No / N° de la demande 80105 092.3

Veröffentlichungs-Nr. / Publication No / N° de la publication : 24 723

Bezeichnung der Erfindung:
Title of invention:
Titre de l'invention : Polyvinyl butyral plasticized with tetraethyleneglycol di-n-heptaoate

Klassifikation / Classification / Classement :

ENTSCHEIDUNG / DECISION

vom / of / du 20 June 1985

~~Anmelder / Applicant / Demandeur :~~ Du Pont

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Einsprechender / Opponent / Opposant : Dynamit Nobel (appellant)

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Art. 52(1) and 54 EPC
"Inventive step", "Improvements"

Leitsatz / Headnote / Sommaire

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Case Number: T 206 / 84

DECISION

of the Technical Board of Appeal 3.3.1

of 20 June 1985

Appellant: (Opponent)
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Representative:

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Decision under appeal:
Decision of the Opposition Division of the European Patent Office dated 2 July 1984 rejecting the opposition filed against European patent No. 24 723 pursuant to Article 102(2) EPC

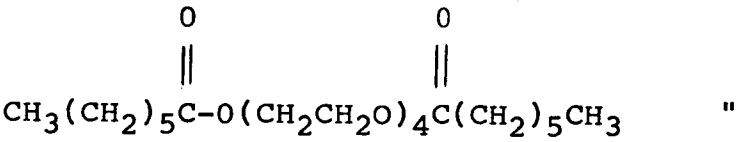
Composition of the Board:

Chairman: K. Jahn
Member: G. Szabo
Member: F. Benussi

Summary of Facts and Submissions

I. European patent No. 24 723 was granted on 16 June 1982 with six claims in response to European patent application No. 80 105 092.3 filed on 27 August 1980 claiming the priority of the earlier application (US - 71 537) of 31 August 1979. Claim 1 reads as follows :

"A plastic compound comprising polyvinyl butyral (hereinafter referred to as PVB) and, in compatible admixture therewith, a plasticizer comprising at least about 10 weight percent tetraethyleneglycol di-n-heptanoate of the general formula :



II

"

II. The opponent filed opposition against the European patent on 16 March 1983, requesting that it be revoked on the ground of lack of inventive step. The opposition was supported by the following four documents :

(1) Gnamm und Sommer "Die Lösungsmittel und Weichmachungsmittel", 1958, especially page 615
(2) K. Thinius "Chemie und Technologie der Weichmacher" VEB Verlag Technik, Berlin 1960, page 604
(3) Römpps Chemie-Lexikon, 7th Edition, 1975, Vol. I, pages 2750/2751
(4) Modern Plastics Encyclopedia, 1978/1979, Vol. I, page 696.

III. The Opposition Division rejected the opposition by a decision of 2 July 1984. The homologues disclosed in the new citations were no closer to the plasticizer in the claim, tetraethyleneglycol di-n-heptoate (4G7), than triethylene-

glycol di-n-heptanoate (3G7) cited in the search report and referred to in the specification by way of comparison. The advantageous properties of 4G7 could not be expected on the basis of the state of the art. This plasticizer provided "outstanding" mechanical properties with PVB at concentrations which are even lower than those required by using 3GH, a popular commercial product. Triethyleneglycol di-2-ethylbutyrate (3GH), 3G7 and triethyleneglycol di-n-octanoate (3G8) were known to be compatible with PVB but the isomer of the last, the di-2-ethylhexanoate (3GO) was partially incompatible. It appeared that it could have been derived from the cited documents that 4G7 would be less compatible than 3G7. There was therefore no good reason to carry out tests with 4G7 to find the true position and to discover that it has better mechanical properties than 3G7 or 3GH. The skilled man seeking improvements had many other possibilities for routine tests. Claim 1 represented an advantageous selection with inventive merit.

IV. The opponent appealed against this decision on 31 August 1984 paying the appropriate fee. The statement of grounds was filed on 2 November 1984. The appellant's submissions during the proceedings and at the oral hearing on 20 June 1984 were substantially as follows :

It was well known that a number of the triethyleneglycol (for definition see (3)) derivatives of aliphatic acids with 6 to 8 carbon atoms were compatible with PVB, including in particular 3GH and 3G7. The argument that 4G7 has a better compatibility than 3G7 disregarded the fact that it was nevertheless worse than 3GH in this respect. In fact 4G7 fell between two known homologues, 4GH (tetraethyleneglycol 2-ethylbutyrate) and 4GO (tetraethylenglycol 2-ethylhexaoate). As regards mechanical properties, the evidence in the specification, in particular Tables II and III, provided no convincing picture. It appeared that the

improved properties only came to light when smaller quantities of the plasticizer were used. The comparisons were unfair and no valid conclusions could be drawn from them.

V. Contesting this view the respondent stated that 4G0 was not compatible with PVB over a broad concentration range (cf. (2) and (4)) and this was further supported by a leaflet from Union Carbide (1965) submitted in the proceedings on 8 February 1985. The prior art suggested a clear tendency of a decrease of compatability when tetraethylene homologues replaced triethylene compounds. There was no reason to test 4G7 in the expectation of advantages. The comparative tests showed improvements of mechanical properties over those of 3GH. The plasticizer according to the invention exhibited a lower tensile creep than commercial compositions and a better Pummel adhesion and edge cloud resistance than 3G7 or 3GH.

VI. The appellant (opponent) requests the cancellation of the contested decision and the revocation of the European patent. The respondent (patentee) requests the rejection of the appeal.

Reasons for the Appeal

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

2. The subject-matter of the patent relates to PVB plasticised with a polyethyleneglycol acylate (i.e. 4G7). The closest state of the art undisputed by the parties in the oral proceedings, is the incorporation of 3G7 in a composition with PVB. Document (4) tabulates 3G7 with its physical properties in comparison with 3GH, 3G0 and 3G8, as well as other types of plasticizers. It is stated there that 3G7 is

compatible with PVB, like 3GH and 3G8, but unlike 3G0 which is only partially compatible. The disclosure establishes 3G7 as a plasticizer for the purpose having at least properties similar to 3GH in this respect. Commercial samples of PVB with 3GH and 3G7 existed (cf. Example 17 of the contested patent).

3. Given the state of the art, therefore, the technical problem was to provide a plasticizer PVB with improved properties. In particular, the limit of compatability with PVB was to be increased within a wide range of hydroxy content in comparison with 3G7, and a sheeting therewith was to be provided with the same or improved mechanical properties and with a plasticizer concentration which is even lower than that required for 3GH. The solution of the problem was to employ at least 10% by weight of 4G7 as a plasticizer. This compound only differs from 3G7 by an ethylene group. Whilst 4G8 is only larger than 4G7 by a methylene group, the disclosure of the former (cf. (1) page 614) is silent about any uses with PVB and need not therefore be treated as the closest state of the art. None of the citations in the search report or raised in opposition mentions 4G7 specifically, and the claimed composition must therefore be considered as novel.

4. The compatability of 4G7 with PVB at various hydroxy contents is shown in Table 1(page 5 of the specification), in Example 16 (Table II) and in the Figure. The maximum tolerance of 4G7 is about 49 or 32%, respectively, at a 20 or 25% hydroxy level, whilst the same was only about 46 and 20% respectively, with 3G7 at the same levels (cf. also page 3, lines 15-19). The best tolerance was still shown by 3GH. As regards mechanical properties, Example 17 and Table II indicated that a lower tensile creep and higher secat modulus was found for PVB with 4G7 (23.3% and 6.1 MPa), in spite of the lower concentration of the plasticizer (35.6% against 38%) at a higher hydroxy level in the former

instance. This should mean improved dimensional stability for the layer without additional difficulty in manufacture (the tensile strength remained virtually the same).

5. Further comparisons were made with the same commercial sample of 3G7 after both products had been laminated between two layers of glass (Example 18, Table IV). Again the results with 4G7 were achieved with less plasticizer (35%) than in the case with 3G7 (38%) in circumstances of higher hydroxy content (23% and 20.8% respectively). Nevertheless, edge cloud resistance and Pummel adhesion (4.4mm and 3 respectively) were significantly better with 4G7 than with 3G7 (7.9 mm and 2) at very similar penetration resistances. This means the product with the composition according to the patent is less likely to be unsatisfactory at the edges when manufactured and retains more of the broken glass when breaking. The point that the comparison might have been unfair since the thickness of the plasticized interlayer (0.80mm) was greater than with 3G7 (0.76mm) cannot be accepted. This is only a 5% fluctuation, which occurred in spite of trying to apply the same amount (cf. page 7, line 6). The results make it credible that the material according to the patent solves the problem underlying the invention, which is to have improved limits of compatability and mechanical properties whilst possibly reducing the concentration of the agent.

6. As to the question of inventive step, it is relevant that the results in these respects are better than with the closest plasticizer in the art, 3G7. It would have been à priori expected that 4G7 is not significantly different from 3G7, being only a small step away from the latter.
It was, however, also known in the state of the art that the increase of the triethylene glycol chain to the tetraethylene glycol chain could mean a further loss of compatibility and even of utility with regard to PVB. 3G0 was

shown to be only partially incompatible with PVB (cf. (1), page 615, (2), page 607 and (4), page 696). Document (2) suggested that the "lengthening of the carbon chain 6, by two methylene groups had, as a consequence, total incompatibility with PVC, and the reduction of the property to less than 100% with polyacrylate. The good result with PVB required that the dosage stays below 25%" (p. 607). 4G0 was, on the other hand, declared as totally unworkable with PVB (p. 608). Such loss of compatability through increasing the ethylene glycole chain was confirmed by evidence submitted on behalf of the patentee which comprised a pamphlet by the manufacturers of "Flexol Plasticizer 4G0" dated July 1965. The document states that there is already incompatibility with PVB at the 10% level (Table 1, first column). Thus this relevant property seems to be lost in consequence of the increase of the polyethyleneglycol component alone whilst the other part of the molecule remains unchanged.

8. It appears that the closest state of the art, using 3G7, was not free from compatability problems either. The specification relies on comparative examples of J to R suggesting (cf. Figure) a limit ranging from 46 to 20% (with the hydroxy content increasing from 20 to 25%). The known worsening of the limits of compatability, i.e. when moving from 3G0 to 4G0, cannot be considered as having been encouraging to the idea of modifying 3G7 in an identical manner. The actual advantageous reversal of performance through an improvement of compatability could not have been predicted and the same applies to some advantages in the mechanical properties. There was no good reason to arrive at 4G7 from 3G7 in the expectation of a significant improvement and even less to embark upon the threefold modification of the most popular plasticizer 3GH in the hope that another agent with high compatibility would be found. In the absence

of any other reason or expectation of a different kind, if there was no incentive to prepare the compound whatsoever, the skilled man could have instead contemplated different kinds of modifications in order to render the plasticizer more compatible with hydroxy groups or to impart some change of mechanical properties of combination in view of the problem to be solved and there was therefore no compelling "one-way-street" situation leading inevitably to 4G7.

9. The argument that the claimed use of 4G7 was obvious because the compound stands in between 4GH and 4G0 cannot be accepted either. It is true that the suitability of, inter alia, 4GH for PVC was mentioned in FR-823 470 (Example 6, and page 5, line 7) but the content of the document was an undifferentiated disclosure of many polyethyleneglycol diacylates, without additional factual details. Even if the expert had decided to take 4GH in particular, as his starting point from the 47 year old document, the idea of two modification (increasing and straightening the chain) was not encouraged by the knowledge concerning 4G0 and 4G8. The first is incompatible with PVB, and the second is listed in (1), pages 614-615, without any usefulness for PVB being mentioned at all, although the same capability is expressly stated for the other homologues included in the survey. Thus the suggested investigation of 4G7, lying in-between these compounds, would not have been a reasonable choice, since the general move from a shorter C_6-acid to a longer C_8-acid seemed to be associated with a less compatibility. The step in this direction, however small, must have therefore been inventive from any starting point in the neighbouring area.

10. To summarize, the teaching according to Claim 1 of the contested patent is new and involves and inventive step. The subsidiary claims are dependent on this claim and relate to special embodiments thereof. The patentability of these is derived from that of the main claim.