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D E C I S I O N
of 21 June 1994

Case Number: T 0565/91 - 3.3.3

Application Number: 86902181.6

Publication Number: 0217863

IPC: C08K 5/09

Language of the proceedings: EN

Title of invention:
Polyurethane plasticizers

Applicant:
CasChem, Inc.

Opponent:
-

Headword:
-

Relevant legal norms:
EPC Art. 56

Keyword:
"Inventive step (yes) - claimed subject-matter omitting feature
deemed essential in the prior art"

Decisions cited:
-

Catchword:
-



Case Number: T 0565/91 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 21 June 1994

Appellant: CasChem, Inc.
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Decision under appeal: Decision of the Examining Division of the
European Patent Office dated 13 March 1991
refusing European patent application
No. 86 902 181.6 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: C. Gérardin
Members: H.H. Fessel
W.M. Schar

Summary of Facts and Submissions

I. The European patent application No. 86 902 181.6, filed as International Application No. PCT/US86/00536 on 19 March 1986 (claiming 4 US priorities dated 19 March 1985 and 3 March 1986) and published under the International Publication No. WO 86/05502, was refused by a decision of the Examining Division of the European Patent Office dated 13 March 1991.

That decision was based on a set of 16 claims as filed on 14 February 1990, of which Claim 1 reads as follows:

"1. A polyurethane gel composition comprising from 10 to 90 parts by weight of the liquid reaction product of an organic polyisocyanate and a polyol and from 90 to 10 parts by weight of an ester plasticizer compound characterized in that the ester plasticizer compound is selected from ditridecyl adipate and esters having a total solubility parameter of from above 9.1 to 10.1 and in that the composition is substantially free of mineral oil."

The further independent Claims 13 and 16 read as follows:

"13. A process for providing a fluid impervious protective seal around an insulated electrical device which comprises introducing the components of the composition of any one of Claims 1 to 12 into a confined space surrounding the section of a device to be protected and allowing said composition to cure."

"16. An insulated electrical device comprising a plurality of insulated wire conductors and the cured polyurethane gel of any one of Claims 1 to 12."

Claims 2 to 12, 14 and 15 are dependent thereon.

II. The reason for that decision was that the subject-matter of Claim 1 did not involve any inventive step vis-à-vis the teaching of

(1) US-A-4 168 258, equivalent to GB-A-2 014 595.

More specifically, it was stated that the solution of the problem to improve the "grease compatibility" of polyurethane gels in contact with grease by omitting the mineral oil was self-evident since mineral oil was known to migrate towards grease, resulting in weight loss and wet contact surface. In the absence of any evidence to support the alleged properties, the Applicant's statement that the claimed mineral oil-free gels were surprisingly just as good as mineral oil-containing gels in terms of hydrophobic character and viscosity, was not accepted. Moreover, the absorption of grease by the gel, as such, was not regarded as a solution to any technical problem and an absorption due to the absence of mineral oil was not demonstrated or supported by any evidence.

It had been noted before that, after the values of the total solubility parameter of the individual plasticizers had been corrected in the description on the basis of the method of calculation mentioned in the application as originally filed, the range of from above 9.1 to 10.1 for that parameter did not contravene Article 123(2) EPC.

III. On 2 May 1991 a Notice of Appeal was lodged against that decision together with payment of the prescribed fee. The Statement of Grounds of Appeal was submitted on 12 July 1991.

In that statement the Appellant (Applicant) disputed the arguments given in the decision under appeal and put forward that, starting from the teaching given in document (1), a skilled person would have no reason to forego the advantageous properties resulting from the incorporation of mineral oil; in other words, said person would have no incentive to exclude mineral oil from such polyurethane compositions.

The experimental data provided with the letter dated 9 November 1990 demonstrated that the difference between the results obtained for the mineral oil-free formulations and the mineral oil-containing formulations was outstanding and not at all to be expected. As further evidence of inventive step, a graph was provided as annex to the Statement of Grounds of Appeal showing the improved long-term performance of polyurethane formulations according to the application in suit with respect to the compositions known from document (1).

- IV. The Appellant requested that the decision under appeal be set aside and that the case be remitted to the Examining Division for further prosecution.

Reasons for the Decision

1. The appeal is admissible.
2. The wording of the claims does not give rise to any objections having regard to Article 123(2) EPC.
 - 2.1 Claim 1 differs from the original version of Claim 1 by (i) the change of the expression "in the presence of" into "and" between the two components of the composition, (ii) the deletion of the alternative

"ricinoleate" as a plasticizer, (iii) the addition of "ditricresyl adipate" as a plasticizer, (iv) the requirement that the plasticizer should have a total solubility parameter "of from above 9.1 to 10.1" instead of the two original alternative sub-ranges between 8.3 and 8.9 and between 9.1 and 9.7, and (v) the indication that the composition is substantially free of mineral oil.

Amendments (ii) and (v) are not objectionable, since they do not result in a claimed subject-matter extending beyond the content of the application as originally filed. Amendment (iii) is supported by Table I, page 11 of the original application, wherein ditricresyl adipate is mentioned as a suitable plasticizer. Amendment (i), which can be regarded as a clarification, is supported by page 13, lines 12 to 15 of the original application.

In its reply to the first communication the Appellant pointed out that some total solubility parameter values mentioned in Table I, page 11 of the original application were erroneous; in particular, the figure 9.7 corresponding to dibutyl phthalate should be amended into 10.1, the latter being used as the upper limit of the new range of that parameter. The fact that this range has been built from a non-amended value for the lower limit and from an amended value for the upper limit is not inconsistent, thus objectionable since, unlike the upper limit which corresponds to a specific plasticizer whose total solubility parameter was erroneous in the original application, the lower limit is not related to any particular plasticizer and, therefore, did not have to be amended. This is supported by the Appellant's arguments in its submission of 9 August 1989 (page 1, paragraph 2 to page 2, paragraph 1), from which it appears that only some of

the total solubility parameter values mentioned in the original application had to be amended. It follows that amendment (iv) does not contravene Article 123(2) EPC, which confirms the finding of the Examining Division.

- 2.2 The preferred range of total solubility parameter of from 9.3 to 10.1 according to Claim 2 has been built from the non-amended value corresponding to glyceryl tri(acetyl ricinoleate) for the lower limit (original application, page 11, Table I) and from the figure 10.1 discussed above. As far as Claims 3 to 16 are concerned, they correspond to Claims 2 to 15 as originally filed with their numbers and appendancies adjusted; the deletion of ditricresyl adipate in Claim 9 is consistent with the incorporation of that compound into Claim 1.
3. Novelty has not been disputed and the Board cannot see any reason to dispute it.
4. Polyurethane compositions extended with mineral oils used in the reclamation or rehabilitation of insulated electrical devices can be regarded as the starting point of the technical developments which led to the present application. As reported in the introduction of the application in suit (starting from page 1, line 15), these extended polyurethane compositions were liquid castable elastomers which had the advantage that they involved the use of cheap oil components; in addition to that substantial cost advantage, these extenders usually imparted improved pot life and hydrolytic stability properties. However, these extended compositions suffered the major disadvantage that the extender oil tended to "bleed" or "exudate".

The compositions disclosed in document (1) must be regarded as an attempt to overcome that shortcoming. They can be defined as ternary polyurethane/mineral

oil/coupling agent formulations, wherein each of these components has to satisfy specific criteria.

- 4.1 Since the compositions according to the application in suit are concerned with the same general objective, document (1) qualifies as relevant prior art. More specifically, Example IV of that citation, which discloses a polyurethane gel composition comprising 35% by weight of a polyurethane, 35% of a mineral oil and 30% of a coupling agent, which can be in particular diundecyl phthalate (Table F), represents the closest state of the art in that such a composition only differs from the claimed compositions by the additional presence of a mineral oil. Like the general compositions according to document (1), this particular composition possesses excellent chemical and electrical properties, which makes them useful in the reclamation or encapsulation of underground cables. However, when these compositions are brought into contact with grease, which is often present in newer underground cables, the mineral oil tends to migrate towards the grease.
- 4.2 In the light of that prior art, the technical problem underlying the present patent application can be seen in providing a formulation with improved grease compatibility.
- 4.3 According to the application in suit this problem is to be solved by a polyurethane composition comprising from 10 to 90 parts by weight of polyurethane and 90 to 10 parts by weight of an ester plasticizer selected from ditridecyl adipate and an ester having a solubility parameter of 9.4 to 10.1 (determined according to the method disclosed on page 11, line 22 to page 12, line 16 of the present application), but being essentially free of any mineral oil.

4.4 In view of (i) the experimental data in the application in suit, which provide evidence that a polyurethane composition containing a mixture of mineral oil and diisodecyl phthalate, the latter being a plasticizer within the terms of the application in suit, in the weight ratio 1:2 gives poor results (Example 3, formulation P), (ii) the Comparative Example filed as annex on 14 November 1990, which demonstrates that compositions based on plasticizers alone (samples A and C) give better results in terms of weight changes, thus in terms of grease compatibility, after a period of three weeks than compositions containing a plasticizer and a mineral oil (samples B and D), and (iii) the graph (Flex Gel Migration Study) filed as annex to the Statement of Grounds of Appeal, which shows a linear drop-off in grease adsorption for prior art compositions, thus indicating a weight loss in the formulation due to mineral oil separation from the polyurethane gel over time, whereas plasticized formulations within the scope of the application in suit retain or even increase grease absorbance over at least one year, the Board is satisfied that the above-defined technical problem is effectively solved.

5. It remains thus to be decided whether the claimed subject-matter involves an inventive step with regard to the teaching of document (1), in particular whether the omission of mineral oil is obvious in view of the above-defined technical problem.

5.1 The grease compatible, mineral oil extended polyurethane compositions are described in document (1) as ternary mixtures, each of the three components contributing to the general properties of these compositions. This is quite clear from both the general and preferred definitions thereof, which require that each component be present in about the same weight proportions

(Claims 1 and 8; column 4, line 53 to column 5, line 4). The mineral oil itself is defined so as to reduce the tendency to migrate to the grease interface; optimal results are obtained with aromatic carbon contents of about 20% (column 8, lines 54 to 68). Neither the description of the citation, which does not suggest any compositional variations outside the framework of the above ternary mixtures, nor Example IV, which illustrates the effect of various esters as coupling agents on oil migration, would provide an incentive to operate without such mineral oil.

In that respect, it is essential to observe that the main role of the coupling agent is to compatibilise the mineral oil and the polyurethane (column 9, lines 1 to 4). There is thus a clear teaching that the coupling agent is added to a system comprising a polyurethane and a mineral oil and that, consequently, in the absence of mineral oil there would be no reason to incorporate a coupling agent. This shows that the role of the coupling agent in the citation cannot be equated with the function of the plasticizer in the application in suit.

- 5.2 This difference is reflected in the respective definitions of the coupling agent in document (1) and the plasticizer in the application in suit.
- 5.2.1 Although both document (1) and the application in suit refer to the total solubility parameter to define the coupling agent and the plasticizer respectively, the method of calculation is not the same, so that a direct comparison of the suitable ranges of that parameter would not be conclusive. As pointed out by the Appellant (statement filed on 14 November 1990, page 1, paragraph 3 to page 2, paragraph 2), there are various

experimental methods for measuring and theoretical models for calculating the total solubility parameter of chemical compounds.

In the application in suit the plasticizer is defined as an ester having a total solubility parameter within a specific range, as determined from parameters directly related to the structure of the compound, i.e. the additive atomic and group contribution for the energy of vaporisation and the molar volume; compounds explicitly envisaged are phthalates, adipates and ricinoleates (page 10, line 7 to page 12, line 16).

In document (1), by contrast, the total solubility parameter must not only be within a first range calculated from the energy of vaporisation to a gas at zero pressure and the molar volume of the compound (column 9, lines 11 to 32), but also within a second range determined from the values of three components, namely the polar solubility parameter, the hydrogen bonding solubility parameter and the non-polar solubility parameter (column 11, lines 16 to 29). Additionally, besides the obvious requirement of miscibility in the mineral oil in all proportions (column 9, lines 4 to 10), the coupling agent should have a hydrogen bonding index number between 6 and 12 (column 9, lines 39 to 47). Chemical compounds satisfying these criteria are liquid esters, ketones as well as compounds in which a polar group is attached to an alkyl structure (column 10, lines 39 to 42).

5.2.2 A comparison between the coupling agents mentioned in document (1) (column 10/11, Table B; Example IV, Table F) and the plasticizers envisaged in the application in suit (page 10, lines 16 to 27) reveals that only few ester compounds would be suitable in both cases. In particular, it appears that dioctyl adipate,

which is described in the citation as a coupling agent particularly effective in reducing migration (column 12, lines 18 to 21; Examples I, III and IV), is not a suitable plasticizer in the framework of the application in suit (page 18, lines 28/29 in conjunction with the statement filed on 9 August 1989, page 2, paragraph 1; and the statements filed on 14 February 1990, page 2, paragraph 3 and on 14 November 1990, page 4, paragraph 1).

The overlap thus does not extend beyond some ester compounds, which means that in most cases the difference between the claimed subject-matter and the prior art compositions is not only the absence of mineral oil, but a different low molecular weight compound as well.

5.3 The possibilities offered within the teaching of document (1) to the skilled person faced with the above-defined technical problem have to be examined in the light of that conclusion.

First, since spewing is caused by insufficient compatibility between mineral oil and polyurethane (column 9, lines 1 to 5), the most obvious solution would be to improve the effectiveness of the coupling agent by considering the parameters which are said to be essential. This would lead the skilled person to manipulate in particular the total solubility parameter and the hydrogen bonding index number. Even if, for the sake of argument, a class of compounds corresponding to the ester plasticizers as defined in the application in suit proved to be particularly effective, such a solution would still involve the presence of a mineral oil.

Secondly, there is no incentive to depart from the literal teaching of document (1), since it is specified that migration can be eliminated only by defining particularly the polyurethane, the mineral oil and the coupling agent (column 4, lines 46 to 52), thus when the three components are present simultaneously. Even if an acceptable reduction of migration may still be achieved by defining only two components particularly, i.e. by defining one of the three components in a less restrictive manner (column 3, lines 39 to 54), thus possibly by modifying the definition of the mineral oil, this cannot be equated with an incentive to omit this component entirely.

- 5.4 It is thus evident that document (1) does not suggest any connection between omission of mineral oil and long term performance in terms of grease absorption. It follows that the claimed subject-matter cannot be derived in an obvious manner from this citation and that, therefore, it involves an inventive step.
6. Claim 1 being allowable, the same applies to independent Claims 13 and 16, which concern applications of such polyurethane compositions substantially free of mineral oil and are thus based on the same inventive concept as Claim 1, as well as to dependent Claims 2 to 11, 14 and 15, which are directed to preferred compositions according to Claim 1 and to particular embodiments of the process according to Claim 13, and whose inventiveness is supported by that of Claim 1.

Order

For these reasons it is decided that:

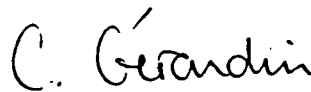
1. The decision under appeal is set aside.
2. The case is remitted to the Examining Division with the order to grant a patent on the basis of Claims 1 to 16 filed on 14 February 1990 and the following description:
 - pages 1 to 3 and 14 to 17 as originally filed;
 - pages 6, 9, 11 to 13 and 18 filed on 9 August 1989;
 - pages 4, 5, 7, 8, 10, 19 and 20 filed on 14 February 1990.

The Registrar:



E. Görgmaier

The Chairman.



C. Gérardin

27/07/89