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**Datasheet for the decision
of 23 July 2015**

Case Number: T 1557/12 - 3.3.03

Application Number: 05818676.8

Publication Number: 1824899

IPC: C08G64/30

Language of the proceedings: EN

Title of invention:

METHOD FOR MAKING POLYCARBONATE

Patent Proprietor:

SABIC Innovative Plastics IP B.V.

Opponent:

Bayer MaterialScience AG

Headword:

Relevant legal provisions:

EPC Art. 56

Keyword:

Inventive step - (no)

Decisions cited:

Catchword:



**Beschwerdekammern
Boards of Appeal
Chambres de recours**

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Case Number: T 1557/12 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 23 July 2015

Appellant: SABIC Innovative Plastics IP B.V.
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Decision under appeal: **Interlocutory decision of the Opposition
Division of the European Patent Office posted on
11 May 2012 concerning maintenance of the
European Patent No. 1824899 in amended form.**

Composition of the Board:

Chairman B. ter Laan
Members: O. Dury
R. Cramer

Summary of Facts and Submissions

- I. The appeal by the patent proprietor lies against the interlocutory decision of the opposition division maintaining European patent No. 1 824 899 (application No. 05 818 676.8, which is based on international application PCT/US2005/038376, published as WO 2006/049955) in amended form according to auxiliary request 1E (10 claims) comprising claims 1-5 as filed with telefax on 2 March 2014 and claims 6-10 filed during the oral proceedings of 4 April 2012.
- II. The granted patent was based on ten claims of which claim 1 read as follows:
- "1. A method of making polycarbonate, comprising:
- melt-polymerizing an aromatic dihydroxy compound and a carbonic acid diester in a presence of a polymerization catalyst in a reactor system producing a byproduct stream (26, 28, 30), wherein the polymerization catalyst comprises a quaternary phosphonium compound; and
- purifying the byproduct stream (26, 28, 30) to separate the carbonic acid diester, wherein the separated carbonic acid diester has a phosphorous concentration of less than or equal to 30 parts per million."
- Claims 2-10 were directed to embodiments of claim 1.
- III. An opposition against the patent was filed in which the revocation of the patent was requested on the grounds of Art. 100(a) EPC (lack of novelty and lack of an inventive step).

IV. During the opposition procedure the following documents, *inter alia*, were cited:

D1: EP 1 221 454 A1

D5: WO 02/077101 A2

D6: Patent proprietor's submission dated 30 March
2012

V. In its decision the opposition division held in particular that D6 was not admitted to the proceedings and that the subject-matter of claim 1 as granted was novel but did not meet the requirements of Art. 56 EPC in view of D1.

VI. The patent proprietor (appellant) lodged an appeal against the above decision. In its statement of grounds of appeal the appellant requested that the decision of the opposition division be set aside and the patent be maintained as granted, or, alternatively, that the patent be maintained in amended form according to auxiliary request 1 filed with the statement of grounds of appeal, which corresponded to the set of claims maintained by the opposition division. Also, the following document was filed:

D7: Experimental report by J. van den Bogerd dated
15 August 2012 (4 pages)

The appellant filed further arguments with letter of 4 May 2015.

VII. In its rejoinder to the statement of grounds of appeal, the opponent (respondent) requested that the appeal be dismissed. With letter dated 19 June 2015, the respondent filed further arguments.

- VIII. In a communication following the summons to oral proceedings, the Board set out its preliminary view of the case, *inter alia* regarding inventive step. In particular, it was noted that the patent in suit failed to provide any data on hydrolytic stability. Moreover, there was no comparison on file between a process according to the patent in suit and that according to the closest prior art D1.
- IX. During the oral proceedings, which were held on 23 July 2015 in the presence of both parties, the appellant withdrew the auxiliary request filed with the statement of grounds of appeal.
- X. The appellant's arguments relevant for the present decision may be summarised as follows:

Inventive Step

- a) D1 was the closest prior art document, and in particular example 1 thereof, in which diphenyl carbonate was recovered in a method for producing polycarbonate using sodium hydroxide as catalyst.
- b) The problem to be solved was to provide a process for producing polycarbonates having improved hydrolytic stability.
- c) The solution to that problem resided in the purification of the carbonic acid diester to a phosphorus concentration of less than or equal to 30 ppm while working with a quaternary phosphonium catalyst.
- d) The technical effect and the demonstration of the inventive character of the invention were based on

paragraph [0049] of the contested patent, where it was stated that organic phosphorus in polycarbonate could "be oxidized/hydrolysed to phosphoric acid derivatives", which "could cause hydrolytic stability issues in the polycarbonate resin".

A comparison with a method according to D1 was not possible since D1 focused its teaching on alkali/alkaline metal earth catalysts such as sodium hydroxide, and on problems arising from using those catalysts. D1 did not deal with quaternary phosphonium catalysts and the problem of hydrolytic instability caused by their decomposition products as did the patent in suit.

As indicated in the patent in suit, hydrolytic instability was not always an issue when using quaternary phosphonium catalysts, especially when no phosphorus was transferred to the byproduct streams. However, D7 showed that the statement of paragraph [0049] of the patent in suit as to the possible impairment of the hydrolytic stability by the presence of some phosphorus compounds was correct, which also gave a possible explanation for the differences between the appellant's findings and those of D5.

The subject-matter of claim 1 did not encompass methods in which less than 30 ppm of quaternary phosphonium was used as catalyst, as argued by the respondent; that did not make any technical sense. The skilled reader would understand that the byproduct stream mentioned in claim 1 had to contain more than 30 ppm phosphorus derived from

the catalyst, otherwise there was no need to purify it.

For those reasons, the problem defined above was credibly solved over the whole breadth of claim 1.

- e) As explained in D6, the value of 30 ppm specified in claim 1 was not arbitrary but corresponded to the maximum concentration of phosphorus in order to allow the addition of further phosphorus compounds to the polycarbonate, such as heat stabilizers or anti-discolouring agents, without jeopardizing the hydrolytic stability of the polycarbonate.

In that respect D6, which had been submitted during the opposition proceedings in response to the late-filed document D5, had not been admitted to the proceedings, contrary to D5. This was unfair to the appellant. Therefore, D6 should be admitted to the appeal proceedings.

- f) There was no teaching in D1 as to the importance of controlling the phosphorus content in the polycarbonate. The calculation regarding the phosphorus content of the separated carbonic acid diester when using 10^{-8} mol quaternary phosphonium catalyst provided by the respondent in its notice of opposition was based on the lowest concentration disclosed in D1, which in practice was not feasible when quaternary phosphonium catalysts were involved.
- g) For those reasons, the subject-matter of claim 1 of the main request was inventive.

XI. The respondent's arguments relevant for the present decision may be summarised as follows:

Inventive step

- a) The teaching of D1, which was the closest prior art document, was not limited to its examples. In that respect, various quaternary phosphonium compounds were disclosed as suitable polymerisation catalysts in D1 and tetraphenylphosphonium phenolate was explicitly disclosed as one of the particularly preferred catalysts. Therefore, claim 1 of D1 read in combination with that specific catalyst, constituted the closest prior art and not example 1 of D1, as argued by the appellant.

- b) The statement made in paragraph [0049] of the patent in suit was purely speculative. Besides, the patent in suit aimed at solving a problem that occurred only for a limited group of phosphorus compounds, as confirmed by the appellant. Indeed, from paragraph [0026] it could be derived that those compounds that were not sufficiently volatile would not be distilled off, in particular not with diphenyl carbonate, and would not cause the problems of hydrolytic instability mentioned in paragraph [0049] of the patent in suit. Those quaternary phosphonium compounds that did not decompose into the products identified in those passages of the patent in suit would also not cause hydrolytic instability.

D7, which was first filed with the statement of grounds of appeal, was not directed to hydrolytic instability but to thermal stability. Also, no

effect was shown for all phosphorus compounds studied therein. Since it was not relevant, D7 should not be admitted to the proceedings.

Moreover, the subject-matter of claim 1 encompassed processes in which very low amounts of quaternary phosphonium compounds were used, in particular also less than 30 ppm. In such situations, purification of the carbonic acid diester to a phosphorus concentration of less than or equal 30 ppm was meaningless. Therefore, an improvement in terms of hydrolytic stability, as relied upon by the appellant, was not achieved over the whole breadth of claim 1 and the problem effectively solved could only be formulated as an alternative.

- c) The solution to that problem resided in the purification of the carbonic acid diester to a phosphorus concentration of less than or equal to 30 ppm.
- d) D6 had not been admitted to the proceedings by the opposition division and there was no reason to admit it at the appeal stage.

The value of 30 ppm as defined in claim 1 of the main request was arbitrary and was, at most, relevant when tetrabutylphosphonium acetate was used as a catalyst.

According to D1, it was desirable to separate secondary products including catalyst residues and secondary products thereof from the polymer melt and to recover unreacted diphenyl carbonate in a form as pure as possible to be recycled to the

polycarbonate synthesis. Hence, the skilled person starting from D1 and wishing to solve the problem defined above, would purify the diphenyl carbonate as much as possible, in particular until the phosphorus reached amounts of 30 ppm or less.

In that respect, it had been shown on page 3 of the notice of opposition that using the lowest amount of tetraphenylphosphonium phenolate catalyst in a process according to D1 led to a phosphorus concentration in the separated diphenyl carbonate within the range specified in claim 1. Therefore, the problem defined above was already solved when working according to D1.

- e) For those reasons, the subject-matter of claim 1 of the main request was not inventive.

XII. The appellant (patent proprietor) requested that the decision under appeal be set aside and that the patent be maintained as granted.

The respondent (opponent) requested that the appeal be dismissed.

XIII. The Board announced its decision at the end of the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

Inventive step

2. Closest prior art

- 2.1 The patent in suit relates to a method of making polycarbonate by reacting an aromatic dihydroxy compound with a carbonic acid diester in the presence of a polymerization catalyst (paragraph [3]). It concerns in particular a method of making polycarbonate comprising melt-polymerizing an aromatic dihydroxy compound and a carbonic acid diester with a polymerization catalyst comprising a quaternary phosphonium compound which is carried out in a reactor system producing a byproduct stream and wherein the byproduct stream is purified to separate the carbonic acid diester (paragraph [6]).

Such a process is known from D1 which both parties as well as the opposition division considered to be the closest prior art document. The Board sees no reason to depart from that view.

3. Problem to be solved

According to paragraphs [0004], [0006] and [0049] of the patent in suit, the problem to be solved is to provide a method for producing polycarbonates by transesterification in the melt, in which the carbonic acid diester may be recycled efficiently and be reused in the same process.

The appellant further formulated the problem to be solved in view of D1 as providing a process for producing polycarbonates having improved hydrolytic stability. In view of paragraph [0049] of the patent specification, that formulation is acceptable.

4. Solution

D1 deals with the melt transesterification reaction of diaryl carbonate (carbonic acid diester) with dihydroxy aryl compounds using a catalyst, which is characterised in that, besides the monohydroxyaryl compound, also the excess diaryl carbonate is isolated from the vapour streams and is recycled directly to the transesterification process (claim 1; figure on page 4). The specific separation process of the diaryl carbonate described in D1 is illustrated in Fig. 1 and further detailed in paragraph [0022] of D1.

The process of D1 allows the separation of diphenyl carbonate (carbonic acid diester) with a purity of e.g. more than 99.9% (paragraph [0023]), which implies that the separated carbonic acid diester can comprise more than 30 ppm of impurities. No information is present on file that allows to relate said impurity level to the phosphorus concentration of the separated carbonic acid diester of "less than or equal to 30 ppm" specified in granted claim 1.

Various suitable polymerisation catalysts are disclosed in paragraph [0040] of D1, including phosphorus compounds such as quaternary phosphonium compounds (page 7, lines 4-6). Although the examples of D1 are performed using NaOH as catalyst (paragraph [0056]), tetraphenylphosphonium phenolate by itself, i.e. not in combination with any other catalysts (see paragraph [0042] of D1), is indicated as one of the most preferred catalysts in a list of four alternatives, including NaOH (D1: page 7, lines 13-14). Further, D1 teaches that the catalyst(s) is/are used in amounts of

10^{-2} to 10^{-8} mol based on 1 mol of dihydroxy aryl compound (paragraph [0041]).

In view of the above, the method defined in claim 1 of the patent in suit differs from the method disclosed in D1, in particular in claim 1, in that the catalyst used mandatorily comprises a quaternary phosphonium compound and in that the separated carbonic acid diester has a phosphorus concentration of less than or equal to 30 ppm.

5. Success of the solution - problem effectively solved

5.1 In examples 1 and 2 of the patent in suit a polycarbonate is prepared by melt polymerisation of bisphenol A and diphenyl carbonate in the presence of tetrabutylphosphonium acetate as catalyst and the byproduct stream is purified so that the separated diphenyl carbonate has a phosphorus concentration of less than 30 ppm, as required by claim 1 (Tables 1 and 2).

However, no data on hydrolytic stability are provided in the patent in suit. Therefore those examples are not related to the improvement relied upon by the appellant in formulating the problem to be solved.

5.2 There is also no comparison on file between the method now being claimed and the one according to D1. In that respect, the appellant's argument according to which no comparison with D1 was possible because D1 did not deal with quaternary phosphonium catalysts cannot be accepted because, as explained above, D1 specifically discloses tetraphenylphosphonium phenolate, which is a quaternary phosphonium compound, as one of the most preferred catalysts.

5.3 Besides, it is derivable from paragraphs [0049] and [0026] of the patent in suit that the problem of hydrolytic stability does not always occur for all quaternary phosphonium catalysts. This was further confirmed by the appellant, both in writing (page 5, fourth paragraph of the statement of grounds of appeal) and during the oral proceedings before the Board.

In that respect, although according to the wording of granted claim 1, a purifying step is required and the separated carbonic acid diester has a phosphorus content of less than or equal to 30 ppm, nothing is said in claim 1 about the phosphorus content of the byproduct stream before the purification step, which may have any value, hence also below 30 ppm, as long as the separated carbonic acid diester has a phosphorus content of less than or equal to 30 ppm.

In addition, claim 1 encompasses methods in which the quaternary phosphonium compounds or the decomposition products thereof either have such vapour pressures that they will not be distilled off with the carbonic acid diester or will not necessarily lead to hydrolytic instability. Since it cannot be excluded that impurities other than phosphorus are present in the byproduct stream, there is no reason to consider that such a reading of granted claim 1 does not make sense. Therefore, the appellant's argument in that sense cannot be followed.

In that respect, there is in particular no evidence on file that the quaternary phosphonium compounds disclosed in D1, in particular the preferred catalyst tetraphenylphosphonium phenolate, have hydrolytic stability issues at all. Also, it has not been shown

that separating the carbonic acid diester in the methods according to D1 so as to result in a phosphorus concentration of less than or equal to 30 ppm, as required by granted claim 1, would lead to the improvement in hydrolytic stability relied upon by the appellant. Therefore, the appellant has not shown that that problem (see second paragraph of section 3 above) in fact occurred when using the quaternary phosphonium catalyst taught in D1. Nor was any improvement over that embodiment of D1 demonstrated.

- 5.4 The appellant relied on D7, the admission to the proceedings of which was contested by the respondent.
- 5.4.1 D7 was filed by the appellant together with its statement of grounds of appeal in support of the argumentation regarding inventive step to reinforce the line of argumentation already brought forward before the opposition division. There is no reason not to admit D7 to the proceedings (Art. 12(4) RPBA).
- 5.4.2 D7 deals with the addition of various phosphorus compounds to a melt polycarbonate in order to study its hydrolytic stability in an autoclave.

In examples 2 and 3 of D7 (see Table on page 4), various amounts of Irgaphos 168 and triphenyl phosphine (usual stabilisers, e.g. described in the examples of D5) were used. Although example 2 shows that Irgaphos 168 may lead to hydrolytic instability, the use of Irgaphos 168 is not in accordance with the teaching of D1, nor can it support any effect of the features identified above as distinguishing features of granted claim 1 over D1 (quaternary phosphonium catalyst; phosphorus content in the separated carbonic acid diester). Regarding example 3 of D7, triphenylphosphine

is not shown to lead to hydrolytic instability, as acknowledged by the appellant (paragraph "conclusion" on the last page of D7).

In examples 4 and 5 of D7, various amounts of dibutyl butylphosphonate and tributylphosphine oxide, which are considered by the appellant to be "possible decomposition products" of a quaternary phosphonium polymerisation catalyst according to granted claim 1 (statement of grounds of appeal: page 6, last paragraph).

Those experiments can at most show that an improvement is present for catalysts that do indeed lead to such decomposition products. However, it was neither shown nor argued that such phosphorus compounds would be obtained when using the quaternary phosphonium compound described in D1. Therefore, the experiments carried out in D7 constitute no evidence to support an improvement in hydrolytic stability over D1, in particular as compared to the preferred quaternary phosphonium catalyst disclosed therein. Even less do they support an improvement over the whole breadth of granted claim 1 i.e. for any quaternary phosphonium catalyst.

5.5 Finally, the method of claim 1 is defined *inter alia* in that the "polymerization catalyst comprises a quaternary phosphonium compound" (emphasis by the Board).

5.6 As elucidated above (section 5.3, second full paragraph), the subject-matter of claim 1 encompasses methods in which low concentrations of quaternary phosphonium compounds are used (either alone or in admixture with other, non quaternary phosphonium catalysts as described in paragraphs [0040]-[0042] of

- D1), which would lead to a phosphorus content of less than 30 ppm in the carbonic acid diester in the byproduct stream before purification. There is, also in that respect, no evidence on file to support any improvement in hydrolytic stability.
- 5.7 For those reasons, the appellant has not shown that the problem of providing a method for producing polycarbonates having improved hydrolytic stability compared to D1 is credibly solved over the whole breadth of claim 1.
- 5.8 As a consequence, the technical problem effectively solved can only be formulated as to provide a method for producing polycarbonates by transesterification in the melt, in which the carbonic acid diester may be recovered and recycled efficiently, further to that of the closest prior art D1.
- 5.9 As explained in section 5.1 above, examples 1 and 2 of the patent in suit show that that problem is solved, which was not contested by the respondent.
6. Obviousness
- 6.1 It remains to be decided whether it was obvious to solve the above-defined problem by modifying the teaching of D1 so as to arrive at the subject-matter of granted claim 1.
- 6.2 D1 teaches in paragraph [13] that the purity of the diarylcarbonate recovered should be as high as possible in order to allow its direct use in the polycarbonate synthesis without lowering its quality. Catalyst decomposition products are in particular mentioned as potential impurities (paragraph [7]).

D1 further discloses concentrations of catalysts as low as 10^{-8} mol with respect to 1 mol of the dihydroxy aryl compound (paragraph [41]). The respondent's findings (see page 3 of the notice of opposition) according to which using concentrations of one of the preferred catalysts in D1 - tetraphenylphosphonium phenolate - in such low amounts would lead to a phosphorus concentration in the separated carbonic acid diester in the range specified in granted claim 1 was not contested by the appellant.

- 6.2.1 The appellant's argument according to which it was not possible to perform a process according to D1 using said (preferred) quaternary phosphonium catalyst at such a low concentration, is not supported by any evidence. Since the teaching of D1 is clear about such use, the appellant's argument can not be followed.
- 6.2.2 In view of the above, the subject-matter defined in granted claim 1 may be arrived at by working within the framework of the teaching of D1, selecting tetraphenylphosphonium phenolate alone among the preferred alternatives disclosed in D1 as the polymerisation catalyst and using it in a concentration at the lower end of the range indicated in D1.
- 6.3 The appellant argued that D6 showed that the value of 30 ppm mentioned in claim 1 was not arbitrary. D6 had not been admitted to the proceedings by the opposition division because of its late filing and its lack of *prima facie* relevance (section 2.2 of the contested decision). In the present case, it was not shown that the opposition division had thereby exercised its discretionary power in a unreasonable way or not according to the correct principles. The fact that D5,

which was also late filed, was admitted to the opposition proceedings, is of no relevance.

Moreover, D6 and in particular the part thereof relating to the criticality of the 30 ppm value is based on conclusions drawn by the appellant on the basis of an internal report dated 2005, which had not been made public, by the appellant's decision (D6: page 2, last full paragraph). Therefore, neither the respondent nor the Board are in a position to evaluate the content of said report.

For those reasons, the appellant's request to admit D6 to the proceedings is rejected and the arguments relying thereon cannot be considered.

7. Therefore, granted claim 1 does not fulfil the requirements of Art. 56 EPC.
8. For those reasons, the appellant/patent proprietor's only request is not allowable and the appeal has to be dismissed.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:

The Chairman:



L. Malécot-Grob

B. ter Laan

Decision electronically authenticated