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Datasheet for the decision of 18 February 2014

Case Number: T 0178/11 - 3.3.03

Application Number: 99300797.0

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Language of the proceedings: EN

Title of invention:
Flame retardant carbonate polymer composition

Patent Proprietor:
SABIC Innovative Plastics IP B.V.

Opponent:
Teijin Chemicals, Ltd.

Headword:

Relevant legal provisions:
EPC Art. 54, 56, 83

Keyword:
Inventive step - (no) (main request, auxiliary request)

Decisions cited:

Catchword:

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Case Number: T 0178/11 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 18 February 2014

Appellant: (Opponent)	Teijin Chemicals, Ltd. 2-2, Uchisaiwaicho 1-chome, Chiyoda-ku Tokyo 100-0011 (JP)
Representative:	Albrecht, Thomas Kraus & Weisert Patent- und Rechtsanwälte Thomas-Wimmer-Ring 15 80539 München (DE)
Respondent: (Patent Proprietor)	SABIC Innovative Plastics IP B.V. Plasticslaan 1 4612 PX Bergen op Zoom (NL)
Representative:	Modiano, Micaela Nadia Modiano & Partners Thierschstrasse 11 80538 München (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 23 November 2010 rejecting the opposition filed against European patent No. 936243 pursuant to Article 101(2) EPC.**

Composition of the Board:

Chairman: B. ter Laan
Members: M. C. Gordon
C. Brandt

Summary of Facts and Submissions

I. The appeal lies from the decision of the opposition division announced on 13 October 2010 and posted on 23 November 2010 rejecting the opposition against European patent number 0 936 243 (granted on European patent application number 99 300 797.0).

II. The patent was granted with a set of 8 claims, whereby claims 1 and 8 were independent and read as follows:

"1. A thermoplastic resin composition comprising

a. 75 to 98 parts by weight of a thermoplastic resin comprising at least one aromatic polycarbonate resin, and
b. 2 to 25 parts by weight of an organophosphorus flame retardant compound, each based on 100 parts by weight of the combined amount of thermoplastic resin and organophosphorus compound, wherein the organophosphorus compound (1) has an acid content that is neutralizable by titration addition of from zero to the equivalent of 1.0 mg of potassium hydroxide per gram of organophosphorus compound, (2) has a hydrolysable chloride content of zero to 100 parts per million based on the weight of the organophosphorus compound as measured by conventional gas or liquid chromatographic techniques and (3) has a magnesium content of from zero to 1000 parts per million based on the weight of the weight [*sic*] *of* the organophosphorus content.

8. A process for making a flame retardant thermoplastic resin composition, comprising combining a thermoplastic resin, said resin comprising at least on [*sic*] aromatic polycarbonate resin, and a flame-retarding amount of an

organophosphorus flame retardant compound as defined in claim 1."

Claims 2-7 were dependent, directly or indirectly on claim 1, whereby claim 2 read as follows:

"2. Thermoplastic resin composition of claim 1 wherein the organophosphorus compound has an alkenylphenyl diphenyl phosphate content of zero to 2000 parts per million based on the weight of the organophosphorus compound."

III. A notice of opposition against the patent was filed on 14 January 2008 requesting revocation of the patent on the grounds of Art. 100(a) EPC (lack of novelty, lack of inventive step) and Art. 100(b) EPC.
Inter alia the opponent relied on the following documents:

D1: EP-A-0 767 215
D3/D3a: JP 9-249768 and English translation
D4/D4a: JP 8-67685 and English translation.
D21/D21a: Ordering sheet of Quality Inspection of Daihachi dated 24 November 1994 concerning CR-741 and English translation.
D25a: English translation of a quality inspection report of Daihachi Chemical Industry Co. Ltd concerning Lot K00901 of CR-741 dated 18 January 1999.

The following document, *inter alia*, was submitted by the patent proprietor with a letter of 16 June 2008 (reply to the notice of opposition):

D10: Quality Inspection Report dated 3 September 1998 relating to Lot K00901 of Daihachi CR-741.

IV. According to the decision, the subject-matter claimed met the requirements of Art. 54 EPC because D1 did not disclose the purity of the organophosphorus flame retardant compound named CR-741. As shown by D10 and D25a, the acid value of CR-741 could lie either within or outside the claimed range even for products derived from the same production batch.

With regard to Art. 83 EPC purification techniques to reduce the acid value, chloride content and magnesium content were conventional measures known to the skilled person.

With regard to Art. 56 EPC the closest prior art was D3, which disclosed a phosphorus flame retardant but not the required acid and hydrolysable chloride contents. The objective technical problem was to provide further heat resistant flame retardant polycarbonate compositions with hydrolytic stability at high relative humidity. Table II of the patent showed that the ageing stability of polycarbonate was improved if the flame retardant had chlorine content, magnesium content and acid value within the claimed ranges.

None of the documents cited, in particular D4 - D6, provided sufficient instruction or incentive to enable the skilled person to arrive at the invention starting from the disclosure of D3 meaning that the requirements of Art. 56 EPC were satisfied.

Consequently the opposition was rejected.

V. On 21 January 2011 the opponent lodged an appeal against the decision, the prescribed fee being paid on the same date.

VI. The statement of grounds of appeal was submitted on 28 February 2011, further written submissions being made with letters dated 22 July 2011 and 28 November 2011.

VII. The patent proprietor - now the respondent - replied with a letter dated 7 July 2011.

VIII. On 6 August 2013 the Board issued a summons to attend oral proceedings.

IX. By letter of 9 December 2013 the respondent/patent proprietor withdrew the request for oral proceedings and stated that it would not attend the oral proceedings. Together with said letter the respondent/ patent proprietor filed a set of claims forming an auxiliary request, which it was submitted differed from the claims of the patent as granted in that claim 2 had been deleted and the remaining claims renumbered.

X. On 20 December 2013 the Board issued a communication setting out its provisional opinion on the case. In particular the Board indicated its opinion that the subject-matter of claim 1 did not meet the requirements of Art. 56 EPC.

XI. In a telephone consultation of 13 February 2014 the respondent confirmed that it had received the Board's communication and reiterated that it would not attend the oral proceedings.

XII. Oral proceedings were held before the Board on 18 February 2014 attended only by the appellant/opponent.

XIII. The arguments of the appellant/opponent can be summarised as follows:

a) Art. 83 EPC

The patent did not disclose how to obtain a phosphate flame retardant with the required impurity content.

With regard to claim 2 of the main request, there was no disclosure in the patent of any method of obtaining the specified compound having the required impurity content.

b) Art. 54 EPC

The subject-matter of operative claim 1 was anticipated by example 15 of D1. The impurity content of the organophosphorus flame retardant compound CR-741 used in that example was within the range specified in the operative claim, as was apparent from D25a. A further novelty destroying anticipation resulted from the disclosure of D3, with reference to D4.

c) Art. 56 EPC

The closest prior art was D3, the subject-matter of claim 1 being distinguished therefrom by the acid content and hydrolysable chloride content. No technical effect was associated with the specified limits which were thus arbitrary. D3 taught that the content of acidic substances and hydrolysable chloride should be as low as possible. Therefore the claimed subject-matter was not inventive.

XIV. The arguments of the respondent can be summarised as follows.

a) Art. 83 EPC

The appellant had advanced no arguments in support of its objections that it would have been impossible to prepare an organophosphorus flame retardant compound of the required purity at the priority date of the patent in suit.

b) Art. 54 EPC

The documents advanced did not establish the properties of the product CR-741 employed in D1, reference being made in particular to D10 and D25a.

D4 could not be relied upon to establish the properties of the product of D3 since the documents employed different processes.

c) Art. 56 EPC

The problem to be solved was to provide compositions having improved hydrolytic stability, good flame retardant properties as well as maintenance of an overall balance of physical properties under hydrolytical conditions. The examples of the patent showed that this problem had been effectively solved.

D3 was the closest prior art from which the claimed subject-matter differed in the specified acid and hydrolysable chloride contents. D3 did

not address the question of hydrolytic stability.

Lowering the acid content of one of the components of the flame retardant was not obvious, since other acid compositions would be present therein. There was no suggestion that doing so would solve the technical problem. The appellant had not shown that the claimed range was not critical.

Analogous arguments applied to the chloride content as required by the claims. Therefore, the claimed subject-matter was inventive.

XV. The appellant (opponent) requested that the decision under appeal be set aside and that the patent in suit be revoked.

XVI. The respondent (patent proprietor) requested that the appeal be dismissed. Auxiliarily it was requested to maintain the patent in amended form on the basis of the auxiliary request (claims 1-7) as submitted with the letter dated 9 December 2013.

Reasons for the Decision

1. The appeal is admissible.

Main request

2. Art. 83 EPC

The argument of the appellant that at the priority date of the patent in suit it was not known how to purify the organic phosphate compounds to the required degree is contradicted by the appellant's own evidence. D21

shows that as early as 24 November 1994, i.e. over three years before the priority date of the patent, it was possible to prepare organophosphorus compounds having the required content of impurities.

Consequently it is concluded that the appellant has failed to establish that the patent in suit does not meet the requirements of Art. 83 EPC.

3. Art. 54 EPC

3.1 D1 (claim 1) relates to an aromatic polycarbonate-styrene polymer resin composition. The composition can further contain a flame retardant (claim 4), which can be an aromatic oligo-organophosphate (claim 5). According to the examples section of D1 at page 22, one of the possible organophosphates is a compound designated as "FR-2" which is the commercial product "CR-741". This is stated to be a mixture of triphenylphosphate-bisphenol-A dimer, triphenylphosphate bisphenol-A oligomers, and triphenyl phosphate in a weight ratio of 84.7/13.0/2.3. FR-2 (CR-741) is employed in example 15 of D1 in an amount of 10 parts by weight in a polymer composition containing polycarbonate, rubber-modified styrene polymer ("HIPS") and General Purpose Polystyrene ("GPPS") in the relative amounts 50:27:2.4 parts by weight.

The impurity level of the FR-2 (CR-741) flame retardant is however not disclosed in D1.

Although a number of documents have been cited by the parties with respect to the impurity content of CR-741, none of these relate to products in the relevant period, i.e. at or before the priority date of D1 (21

June 1994). The earliest document advanced is D21, "Ordering Sheet- Quality Specification" relating to an order dated 24 November 1994. This document also bears a date at which measurements were carried out namely more than a year later on 19 December 1995. Consequently D21 cannot serve to establish the impurity content of the product CR-741 employed in the examples of D1.

Furthermore there is evidence that the designation "CR-741" does not provide direct and unequivocal information about the constitution of the product so named. According to "Quality Inspection Report" D10 a product indicated with lot number K00901 had an acid value of 1.68 KOH-mg/g, whereas according to D25a a product with the same lot number had an acid value of 0.420 KOH-mg/g. The evidence of D10 and D25a demonstrates that the impurity level of the product sold under the designation CR-741 was highly variable and that this variability even applied within a single manufacturing lot.

Consequently even if a document had been advanced dating from before the priority date of D1 relating to the properties of CR-741 the evidence of D10 and D25a indicates that such document would not have provided direct and unambiguous evidence that the product under that designation employed according to the examples of D1 necessarily had the properties, in particular acid value, as required by the operative claims.

The conclusion is that there is no direct and unambiguous evidence relating to the constitution of CR-741 manufactured or sold at any time, let alone in the critical period before the priority date of D1, so that it is not possible to draw any conclusions about

the properties of the CR-741 actually used in D1.

3.2 D3/D3a is directed to a flame retardant for resins whereby the flame retardant is composed of a mixture of phosphate oligomers based on bisphenol A and defined by a generic formula, which flame retardant contains 1 wt% or less of a diaryl phosphate and has a total metal content of 30 ppm or less by weight (claim 1). Claims 3 and 4 of D3/D3a are directed to flame retardant resin compositions containing the specified flame retardant compounds. The flame retardant is stated to have excellent flame retardancy, heat resistance and electrical insulating properties and not to give rise to problems of mould contamination (D3a abstract and paragraph [0011]).

Example 1 of D3a discloses the manufacture of the flame retardant. The impurity content of the resulting material is not disclosed in D3a.

The appellant argued that by reference to a "repetition" of the preparation procedure disclosed in synthetic example 2 and example 10 of D4, it would be possible to determine the impurity content of the product of example 1 of D3. However this argument fails because D3 and D4 disclose different processes. The process employed for the preparation of the organic phosphorus compound as disclosed in D3a differs from the process of D4 not only in respect of the amounts (total and relative) of reactants employed and the sequence of the reaction steps but also in that the final rinsing steps differ and furthermore in that D4 employs an additional purification step involving treatment with propylene oxide which step is absent from D3a. Consequently a repetition of the procedure of D4 cannot provide any information about the properties

of the properties obtained by the process of D3a.

3.3 The evidence advanced thus does not demonstrate that the subject matter of claim 1 is anticipated by the prior art.

3.4 Consequently the subject-matter of claim 1 as well its dependent claims 2 - 7 meet the requirements of Art. 54 EPC.

Since independent claim 8 relies on the flame retardant as defined in claim 1, this conclusion applies also to claim 8.

4. Art. 56 EPC

4.1 *The closest prior art*

The patent in suit aims at providing flame retardant compositions having improved hydrolytic stability, which term according to paragraph [0007] means a tendency of the composition not to undergo molecular weight changes, in particular of the polycarbonate resin, when exposed to hydrolytic conditions.

4.2 The decision under appeal held that D3 represented the closest prior art. Both parties have indicated that they share this assessment of the relevance of D3.

According to D3a, the bisphenol-A based phosphate oligomer flame retardant disclosed therein has excellent flame retardancy, heat resistance and electrical insulating properties and does not give rise to problems of mould contamination (abstract, paragraph [0011]). D3a further teaches that the defined phosphate oligomer has a higher resistance to hydrolysis than

phosphate oligomers crosslinked by monocyclic phenol groups (D3 paragraph [0006]). In paragraph [0012] it is stated that the specified bisphenol-A based phosphate oligomer is less susceptible to molecular weight reduction as a result of hydrolytic reaction at the time of heating. Consequently, D3 does relate to the question of hydrolytic stability, contrary to the statement of the respondent.

The Board is therefore satisfied that in view of the problem addressed by D3, that document does indeed represent an appropriate starting point for the assessment of inventive step.

4.3 *Defining the problem to be solved*

4.3.1 According to the examples of the patent in suit, three different mixtures of bisphenol A diphosphate oligomers were employed, all having a degree of polymerisation of 1.08 and designated BPA-DP-1, BPA-DP-2 and BPA DP-3, the properties of which were reported in Table 1 of the patent:

TABLE I

	BPA-DP-1	BPA-DP-2	BPA-DP-3
Acid level (mg KOH/g)	<0.01	< 0.01	< 0.02
Hydrolyzable chloride content (ppm)	1450	22	4
Magnesium content (ppm)	576	1296	<60
Isopropenylphenyl diphenyl phosphate content (wt%)	> 1%	>1%	<1%

Of these three compounds only BPA-DP-3 complies with the impurity requirements of the claims. BPA-DP-1 has a hydrolysable chloride content of 1450 ppm, which is more than an order of magnitude above the upper limit of 100 ppm required by operative claim 1 and BPA-DP-2 has a magnesium content of 1296 ppm which is ca 25% above the threshold of 1000ppm required by operative

claim 1.

According to the results reported in Table 2 of the patent, compositions of PC, ABS, SAN and PTFE containing BPA-DP-3 exhibit better resistance to hydrolysis ageing, as measured by the reduction in molecular weight, than otherwise identical compositions containing either BPA-DP-1 or BPA-DP-2.

4.3.2 Claim 1 permits broad ranges for the contents of the various impurities (acid value up to 1 mg KOH/g, up to 100 ppm hydrolysable chloride and up to 1000 ppm Mg). However the only example to comply with all the requirements of the claim has contents of these impurities far below the permitted upper limits, namely <0.02 mg KOH/g, 4 ppm chloride and <60ppm Mg. Furthermore, as noted above, the impurity levels of the two comparative flame retardants are significantly above the upper limits specified in claim 1. Accordingly the examples and comparative examples of the patent do not provide evidence of any technical significance or effect relating to hydrolytic stability associated specifically with the distinguishing features with respect to D3, namely the upper limits as specified in claim 1 on impurities in the organophosphorus flame retardant compound.

4.4 The absence of evidence for any technical effect over D3a associated with the specified limitations on the content of impurities means that such limitations have to be regarded as arbitrary. The consequence of that is that the problem to be solved by the claimed subject-matter can be formulated merely as the provision of further compositions containing polycarbonates and the organic phosphorus oligomeric compounds.

4.5 *The solution to the problem*

The problem was solved by specifying limits for the content of impurities in the organo-phosphorus compounds as defined in claim 1.

4.6 *Obviousness*

4.6.1 D3a itself contains clear pointers to reduce the content of all three impurities specified in operative claim 1, even if no quantitative limits are given. Thus in D3a in paragraph [0012] it is taught that residual metal can, in association with other impurities, result in reduction of the molecular weight of the resin due to ester interchange and hydrolytic reaction, i.e. hydrolysis is implicated in the reported molecular weight reduction. Page 8 (paragraph [0019], final sentence) of D3a further refers to removal of residual chlorine component after synthesis of the phosphate and in the passage bridging pages 8 and 9 discusses the desirability of removing a component - diaryl phosphate - which *inter alia* tends to release a proton leading to decomposition of the resin and corrosion at the time of moulding.

4.6.2 D4 relates to a process for purifying a phosphate ester (claim 1). According to paragraph [0002] said process provides a phosphate ester having a small acid value and excellent heat resistance. D4 also emphasises in paragraph [0004] the need to remove alkali metal content (e.g. Mg) since alkali metal remaining in the phosphate ester exerts a bad influence on heat and hydrolysis resistance of the phosphate ester.

4.6.3 In view of the teachings of either D3 alone, or in combination with D4, the step of imposing upper limits

on the content of the acid, hydrolysable chloride and magnesium is an obvious solution to the problem of providing further compositions based on those known from D3.

4.7 The main request therefore does not meet the requirements of Art. 56 EPC and is refused.

Auxiliary request

5. Claim 1 of the auxiliary request is identical to claim 1 of the main request, and does not meet the requirements of Art 56 EPC for the same reasons. The auxiliary request is refused.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. European Patent number EP 936 243 is revoked.

The Registrar: The Chairman:

E. Goergmaier B. ter Laan

Decision electronically authenticated