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Datasheet for the decision of 19 October 2011

Case Number: T 2347/08 - 3.3.03

Application Number: 01979099.7

Publication Number: 1322702

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Language of the proceedings: EN

Title of invention:
Halogen-free flame retarder composition and flame retardant polyamide composition

Patentee:
BASF SE

Opponent:
LANXESS Deutschland GmbH

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56, 114(2), 123(2)

Keyword:
"Main request - inventive step - (no)"
"First auxiliary request - inventive step - (yes)"

Decisions cited:
-

Catchword:
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Case Number: T 2347/08 - 3.3.03

D E C I S I O N
of the Technical Board of Appeal 3.3.03
of 19 October 2011

Appellant: (Patent Proprietor)	BASF SE 67056 Ludwigshafen Deutschland
Representative:	Richter, Helmut Kirchbündtenstrasse 19 CH-4107 Ettingen (CH)
Respondent: (Opponent 1)	LANXESS Deutschland GmbH LXS-LIP-IPR Building Q 18 D-51369 Leverkusen (DE)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office dated 4 September 2008 and posted 20 October 2008 concerning maintenance of European patent No. 1322702 in amended form.**

Composition of the Board:

Chairman: B. ter Laan
Members: M. C. Gordon
R. Cramer

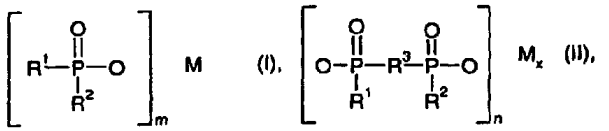
Summary of Facts and Submissions

I. The appeal by the patent proprietor lies against the decision of the opposition division announced on 4 September 2008 and posted 20 October 2008 according to which it was held that European patent EP-B1 1 322 702 (application number 01 979 099.7) could be maintained in amended form on the basis of the second auxiliary request consisting of 15 claims and filed at the oral proceedings held before the opposition division on 4 September 2008.

II. Independent claims 1, 2 and 9 of the patent as granted read as follows:

"1. Halogen-free flame retardant composition for use in a thermoplastic composition, which flame retardant composition contains at least

a) 10-90 Mass% phosphinate compound according to formula (I) and/or formula (II) and/or polymers thereof;



in which

R^1, R^2 is hydrogen, a linear or branched C_1-C_6alkyl radical, or a phenyl radical;

R^3 is a linear or branched C_1-C_{10}alkylene, arylene, alkylarylene, or arylalkylene radical;

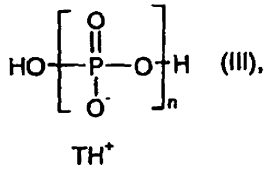
M is an alkaline earth metal or alkali metal, Al, Zn, Fe, or a 1,3,5-triazine compound;

m is 1, 2, or 3;

n is 1 or 3;

x is 1 or 2; and

b) 90-10 Mass% polyphosphate salt of a 1,3,5-triazine compound according to formula (III)



(III),

in which

T represents a 1,3,5-triazine compound,

n is a measure of the number average degree of condensation and is higher than 3;

and

c) 1-30 Mass% olefin copolymer;

the sum of components a)-c) being 100%.

2. Halogen-free flame retardant composition for use in a thermoplastic composition, which flame retardant composition contains at least

a) 10-90 Mass% phosphinate compound according to formula (I), wherein R^1, R^2, M and m are as defined in claim 1, and or formula (II), wherein R^1, R^2, R^3, M, n and x are as defined in claim 1; and

b) 90-10 Mass% polyphosphate salt of a 1,3,5-triazine compound according to formula (III), wherein T and n are as defined in claim 1 and the 1,3,5-triazine content is higher than 1.1 mol 1,3,5-triazine per mol of phosphorus atom; and

c) 0-30 Mass% olefin copolymer;

the sum of components a)-c) being 100%.

9. Flame retardant polyamide composition that contains the following components:

a) 95-10 Mass% polyamide;

b) 0-50 Mass% glass fiber;

c) 5-40 Mass% flame retardant composition according to any one of claims 1-8;

d) 0-50 Mass% other additives;
the sum of components a)-d) being 100 mass%."

III. Notices of opposition against the patent were filed by opponent 1 on 4 September 2006 and by opponent 2 on 5 September 2006. Both opponents requested revocation of the patent in its entirety on the grounds of Article 100 (a) EPC (lack of novelty as well as lack of inventive step). Opponent 2 withdrew its opposition by letter of 15 April 2008. By letter of 16 June 2008 Opponent 1 invoked an additional ground for the opposition based on Article 100 (b) EPC. That as well as the novelty objection against claims 1 and 2 in view of the document designated D1 were abandoned during the oral proceedings before the opposition division (see the decision under appeal, Facts and Submissions, point 10).

The oppositions were *inter alia* based on

D1 WO-A-99/67326
D2 WO-A-97/39053
D10 Experimental Report filed by the Patent Proprietor during the proceedings before the examining division with letter of 2 June 2005
D11 WO-A-00/02869

IV. The decision of the opposition division was based on three sets of claims. The patent was maintained on the basis of the second auxiliary request which corresponded to the claims as granted but from which claim 2 had been omitted.

(a) The opposition division held that the claims as granted fulfilled the requirements of Article 83 EPC and also that the subject-matter of all granted claims was novel and that the subject-matter of claim 1 was inventive.

(b) For claim 2, D2 was considered to be the closest prior art document. The subject-matter of claim 2 was distinguished from the disclosure of D2 by the specified triazine/phosphorus (hereinafter "T/P") ratio of the melamine phosphate. However, as no effect had been shown for that difference, the technical problem to be solved by the subject-matter of claim 2 was merely the provision of a further halogen-free flame retardant composition. D2 addressed, *inter alia,* the problem of improving the thermal stability of nitrogen containing flame retardants in polymers such as polyester and polyamides. The excellent thermal stability of a melamine polyphosphate as specified in the opposed patent was known from D11 which document also suggested the presence of a second flame retardant component such as a phosphorus containing compound. Hence it was obvious to combine the teachings of D2 and D11, i.e. to use the melamine polyphosphate of D11 instead of the melamine phosphate of D2 and thus to arrive at the subject-matter of claim 2.

(c) The argumentation regarding the main request also applied to the first auxiliary request, which differed from the main request only in the lower limit of n in formula III, which was disclosed in D11.

(d) Since the second auxiliary request did not contain claim 2, the opposition division had no objection to this request.

V. The patent proprietor lodged an appeal against this decision on 19 December 2008, the prescribed fee being paid on the same day. Together with the statement of grounds of appeal, filed on 25 February 2009, the appellant filed a further experimental report.

VI. The opponent - now the respondent - filed a reply with a letter of 14 April 2009, citing a new document:

D17 WO-A-99/57187

VII. On 1 June 2011 the Board issued a summons to attend oral proceedings on 19 October 2011. In an accompanying communication the Board noted, *inter alia* that the appellant had not as yet advanced any arguments as to why the Board should not take D17 into account during the appeal proceedings.

VIII. By a letter of 13 September 2011 the appellant made further written submissions including a further experimental report and submitted 9 sets of claims forming first to ninth auxiliary requests.

IX. By letter dated 20 September 2011 the respondent stated that since the appellant had not provided any arguments or comparative examples in response to the summons it was intended not to attend the oral proceedings, which intention was affirmed in a telephone consultation with the Board's Registrar on 10 October 2011 in which the respondent confirmed that it had received the

submission of 13 September 2011, including the experimental report.

X. Oral proceedings were held before the Board on 19 October 2011 attended only by the appellant. During the course of the oral proceedings the appellant withdrew the first-seventh auxiliary requests as filed with the letter of 13 September 2011.

A new first auxiliary request, based on the sixth auxiliary request as filed on 13 September 2011 and designated "6. Hilfsantrag (geändert)" was submitted.

Claim 1 of this request was as granted; claim 2 read as follows:

"2. Use of a halogen-free flame retardant composition which contains at least

a) 10-90 Mass% phosphinate compound according to formula (I), wherein R^1, R^2, M and m are as defined in claim 1, and or formula (II), wherein R^1, R^2, R^3, M, n and x are as defined in claim 1; and

b) 90-10 Mass% polyphosphate salt of a 1,3,5-triazine compound according to formula (III), wherein T and n are as defined in claim 1 and the 1,3,5-triazine content is higher than 1.1 mol 1,3,5-triazine per mol of phosphorus atom; and

c) 0-30 Mass% olefin copolymer;

the sum of components a)-c) being 100%, in glassfiber-reinforced polyamide."

Dependent Claims 3-7 and 13 were amended to be dependent only from claim 1. Newly introduced dependent claims 8-12 were dependent only from claim 2.

Claim 14 read as follows:

"14. Flame retardant polyamide composition that contains the following components:

a) 95-10 Mass% polyamide;

b) 10-40 Mass% glass fiber;

c) 5-40 Mass% flame retardant composition as defined in any one of claims 1 to 12;

d) 0-50 Mass% other additives;

the sum of components a)-d) being 100 mass%."

Claims 15-20, corresponding to granted claims 11-16, were amended so as to depend on claim 14.

The second and third auxiliary request corresponded to the eighth and ninth auxiliary requests, respectively, as filed with the letter of 13 September 2011, the latter request corresponding to the set of claims on which the patent had been maintained by the opposition division.

XI. The arguments of the appellant only referred to claim 2 and can be summarised as follows:

Main request

(a) Starting from D2, as the opposition division as well as the respondent had done, the problem to be solved was to provide polyamide compositions with good flame retardant properties and improved processability, viscosity and stability. The evidence submitted together with the statement of grounds of appeal demonstrated that that problem

had effectively been solved by the use of melamine polyphosphate instead of the melamine phosphate used in the compositions according to D2.

(b) No prior art document provided any comparison of melamine phosphate and melamine polyphosphate with respect to processing properties. It was however generally known in the prior art that addition of melamine phosphate containing flame retardant resulted in substantial worsening of the noted properties since it promoted polymer degradation. Surprisingly, replacement of melamine phosphate by melamine polyphosphate either did not impair such properties or even resulted in an improvement.

(c) D2 disclosed many compounds which could be employed as "synergists" - meaning compounds that merely augmented or "boosted" the flame retardant effect, but for which no true synergism had been shown - with phosphinates, but melamine polyphosphate was not among them. Hence there was no incentive to select melamine polyphosphate to employ in the composition of D2.

(d) D11 disclosed the use of melamine polyphosphate as flame retardant in polymer compositions. The teaching in D11 (page 2, lines 22 to 34) that the use of conventional melamine polyphosphate led to polymer degradation related to processing at elevated temperatures at which the polyphosphates were not stable. Thus this teaching related in fact to the properties of the melamine polyphosphate, not of the polymer. According to D11 it had been found that this problem could, in

the case of melamine polyphosphate, be addressed by controlling the T/P ratio. This observation however had nothing to do with the behaviour of the polymer itself under processing conditions but merely taught that an agent within the polymer matrix might lead to degradation of the polymer. D11 provided no comparison with polymers containing melamine phosphate. Further D11 contained no information with respect to phosphinates. Therefore, D11 did not render the claimed solution obvious.

(e) Regarding the alternative approach, starting from D11 as closest prior art, the appellant submitted that D11 focused on melamine polyphosphate and taught that this could be combined with a wide range of other flame retardants. The problem to be solved with respect to D11 was to provide further melamine polyphosphate based flame retardants. The evidence showed that this problem had been solved by the claimed subject-matter. The combination was not obvious as D11 contained a vast range of possible additional flame retardants in a long list, in which phosphinates were not mentioned. The combination of the teachings of D11 with D2 would not have been considered, in particular because each employed different principal components. The disclosure in D2 of melamine phosphate as a possible component to be combined with phosphinates would not render it obvious to replace this by melamine polyphosphate and consequently would not establish a link between D2 and D11.

(f) Even accepting that D11 and D2 were linked by the technical problem, neither document provided an indication of the route to take in solving the problem of providing further melamine polyphosphate based flame retardants which would lead the skilled person to the other document. In particular D2 disclosed in effect any nitrogen compound, including melamine phosphate, in combination with phosphinate. Similarly, the list of second components in D11 was very large, encompassing practically all known flame retardants.

First auxiliary request

(g) Also with regard to the first auxiliary request, D2 was the closest state of the art. This disclosed a combination of a phosphinate with a monophosphate but did not disclose glass fibre reinforced polyamide. In the experimental report of 25 February 2009 it had been shown that the combination of phosphinate with melamine polyphosphate as claimed improved the processability of glass fibre containing polyamide. There was no suggestion of this in the state of the art. D11 disclosed a large number of different flame retardants and it would not have been obvious to consider D11 in combination with D2. Hence D11 did not render the combination of melamine polyphosphate with phosphinates obvious.

(h) Starting from D11 the problem was to provide synergistic flame retardants, which as shown in the experimental report of 13 September 2011 had

been solved. D11 did not mention synergy and D2 did not in fact demonstrate any synergy. Also there was no disclosure in D2 of a mixture with melamine polyphosphate, let alone with the ratio as specified. In particular, D2 did not give any hint to a synergistic effect arising in the case of glass fibre reinforced polyamide. Hence, the subject-matter of the first auxiliary request was inventive.

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

Main request

(a) The problem to be solved by the patent in suit was, according to paragraph [0003] thereof, to obtain a good combination of flame retardancy and mechanical properties. However according to examples 2 and 3 as submitted with the statement of grounds of appeal the flame retardancy had not been improved since both compositions - whether containing melamine phosphate or melamine polyphosphate had the same flame retardancy rating (UL-94 rating of V-2).

(b) D2, the closest document, disclosed in Table 6 a polyamide composition containing methylethyl phosphinic acid (i.e. phosphinate) and 8% melamine phosphate and achieved a better flame retardancy rating (V-0).
Starting from D2 the objective technical problem to be solved by the subject matter of claim 2 was to improve flame resistance and processability of

polyamide compositions. The solution to that problem lay, according to claim 2, only in the combination of a phosphinate with 1,3,5-triazine polyphosphate. That it was possible to employ melamine polyphosphate instead of melamine phosphate in combination with phosphinate for polyesters was, according to page 3 of the patent in suit, known from D17. As the combination of phosphinate and melamine polyphosphate was known for polyesters, the application of this teaching to polyamides was not inventive.

(c) Claim 2 contained a restriction relating to the ratio of melamine to phosphorus, which was however the preferred range - leading to better mechanical properties - of D11, which document was also cited in the patent in suit.

(d) Therefore, the skilled person in knowledge of this state of the art, would have been motivated to provide a composition having the features of operative claim 2.

XIII. The appellant requested that the decision under appeal be set aside and the patent be maintained as granted, or alternatively on the basis of auxiliary request 1 entitled "6. Hilfsantrag (geändert)" or on the basis of auxiliary request 8 filed with letter of 13 September 2011 (auxiliary request 2).

The respondent requested in writing that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. The decision under appeal held that the subject matter of claim 1 of the patent as granted fulfilled the requirements of the EPC, but that claim 2 did not meet the requirements of Art. 56 EPC. That decision had not been challenged on appeal by the respondent. The appellant had only argued regarding claim 2. Therefore, the only matter to be decided with respect to the main request is whether the subject-matter of claim 2 meets the requirements of Article 56 EPC.

Main request

3. Inventive step

3.1 The patent in suit relates to a halogen-free flame retarder composition and flame retardant polyamide. Halogen-free flame retarder compositions were known from D2, which, by common consent, is regarded as the closest prior art document.

3.1.1 D2 discloses a synergistic flame protection agent combination for thermoplastic polymers, in particular for polyesters, comprising as component A a phosphinate or a diphosphinate of specified formulae I and/or II or their polymers, of calcium, aluminium or zinc, and as component B a nitrogen containing compound according to a number of specified formulae III to VIII (claim 1). Component B can be melamine phosphate (formula VII on page 4 of D2). On page 1, last paragraph, of D2 it is explained that calcium and aluminium phosphinates are

known to be particularly effective as flame retardants in polyesters. The polymer compositions may also contain fillers such as glass fibres (page 12, fourth paragraph).

One aspect explicitly considered by D2 is the need for the flame retarded polymers to be processable and to this extent discusses in the second paragraph of page 2 that a number of known nitrogen compounds used in combination with phosphinates can lead to poor polymer properties, including lack of processability. In D2 that problem is solved by the above indicated combination of certain specific phosphinate or diphosphinate compounds with certain specific nitrogen compounds such as melamine phosphate.

The subject matter of operative claim 2 is hence distinguished from the teaching of the closest prior art by the feature that melamine polyphosphate of the defined properties rather than melamine phosphate is employed in the flame retardant, whereby melamine phosphate is the closest analogue thereto disclosed in D2.

3.2 *The technical problem*

3.2.1 The examples in the patent in suit contain no comparison with compositions according to D2.

3.2.2 Experimental report D10 relates to unreinforced polyamide compositions containing a proportion of an olefin polymer. In all the compositions aluminium diethylphosphinate ("DEPAL") was employed. Comparisons were presented between compositions containing the

aforementioned components and additionally either melamine polyphosphate or melamine phosphate, the latter being compositions according to D2. However D10 does not report the T/P ratio of the melamine polyphosphate employed meaning that it is not established whether the material employed in these data corresponds to that required by the operative claim. Therefore, these experiments cannot be taken into account.

3.2.3 Together with the statement of grounds of appeal a further experimental report was provided showing a comparison between compositions containing a phosphinate and either melamine polyphosphate or melamine phosphate in glass fibre reinforced polyamide, without any olefin component. A further example was provided containing none of these additives, i.e. only the glass fibre reinforced polyamide. The results show that the composition without any additives had good processability - as measured by extrusion properties (extrusion temperature, torque and melt pressure at nozzle) or injection moulding properties (melt temperature injection pressure and demoulding properties) as well as viscosity number and melt volume rate but poor combustion properties ("Not Classified" according to the UL94 test). The composition containing melamine phosphate and the phosphinate, a composition according to Table 6 of D2, had poor processing properties, whereas the example containing melamine polyphosphate and the phosphinate exhibited good combustion properties, the processing properties being in some respects superior to those of the polyamide without any additives.

3.2.4 With the letter of 13 September 2011 further evidence was advanced showing four compositions based on a glass fibre reinforced polyamide, aluminium diethyl phosphinate and each of four melamine polyphosphates with triazine/phosphorus ratios of 0.96, 1.00, 1.12 and 1.16, the first two thus being outside the scope of claim 2, the latter two being within the scope of claim 2. From the results it can be concluded that in the case of glass fibre filled polyamides, the combination of the two flame retardants as claimed gives rise to an improvement in flame retardant properties which goes beyond that which would arise from a simple addition of the effects of each flame retardant individually, i.e. appears to be synergistic. The combination also gives good processing properties. The ratio triazine/phosphorus in the melamine polyphosphate component however exerts no influence over the flame retardant properties of the combination.

3.3 The evidence provided therefore shows that, in the case of glass fibre reinforced polyamide compositions, the use of melamine polyphosphate instead of melamine phosphate leads to an improvement in the processing properties. The evidence also demonstrates that there is a synergistic improvement in flame retardancy as a result of the claimed combination of additives.

The subject matter of claim 2 however is not restricted to glass fibre filled compositions but encompasses also non-filled compositions. The available evidence does not permit any technical effect to be identified in the case of non-filled compositions.

Consequently the technical problem effectively solved over D2 by the subject matter of claim 2 of the main request has to be formulated as providing further flame retardant polyamide compositions.

3.4 Obviousness

3.4.1 D2 does not suggest to use polyphosphates as now claimed in flame retardant thermoplastic compositions. Therefore, D2 by itself does not render claim 2 of the main request obvious.

3.4.2 D11 discloses a polyphosphate salt of a 1,3,5-triazine compound having a number average degree of condensation of higher than 20 and with the melamine content amounting to more than 1.1 mole of melamine per mole of phosphorus atom (claim 1), such as melamine polyphosphate (D11, page 5, lines 27 to 33), i.e. a compound corresponding to component (b) of claim 2 of the patent as granted. D11 also relates to the use of this compound as a flame retardant in polymer compositions (page 1, first paragraph). On page 8 starting at line 7 it is stated that the flame retardant action can be enhanced by the presence of a compound with a synergistic effect for the flame retardant, in particular a carbon forming compound, possibly in combination with a catalyst promoting carbon formation. Among the compounds mentioned are melamine resins (page 8, line 24). Page 9 starting at line 16 teaches that the flame retardant action of the salt can be further enhanced by addition of a second flame retardant composition. Phosphorus compounds are mentioned, including melamine phosphate.

D11 contains two examples, both based on glass filled polyamide. In the inventive example a salt having an T/P ratio of 1.26 is employed; in the comparative example the salt has a T/P ratio of 0.94. The results show that the composition containing the former salt has the better flame retardancy as well as tensile strength, elongation at break, Charpy notch impact and modulus of elasticity.

3.4.3 D11 therefore teaches that melamine polyphosphate having the ratio melamine/phosphorus required by present claim 2 is suitable for conferring good flame retardant as well as mechanical properties to e.g. polyamide compositions. Therefore the skilled person looking for a further flame retardant polyamide composition would consider the use of melamine polyphosphate instead of melamine phosphate. This renders obvious the use of melamine polyphosphate of the specified constitution as the nitrogen compound instead of one of those mentioned in D2 with a view to finding a further flame retardant composition to the one of D2.

3.5 Consequently the subject matter of claim 2 of the main request is obvious and does not meet the requirements of Art 56 EPC. The main request is therefore refused.

4. *First auxiliary request.*

4.1 Amendments

The features of claim 2 as amended compared to the main request, are based on those of originally filed claims 1, 9-11 and page 3 lines 19, 20.

The subject matter of claims 3-7 and claims 8-12 is based on that of originally filed claims 2-6. Claim 13 corresponds to originally filed claim 9, however restricted in its dependency to claim 1. Claim 14 is a combination of original claims 10 and 11. Claims 15-20 correspond to originally filed claims 12-17. Accordingly the requirements of Art. 123(2) EPC are satisfied.

The amendment of claim 2 to the use of the defined flame retardant composition in a glass fibre reinforced polyamide results in a restriction of the scope of protection compared to claim 2 as granted. Consequently the requirements of Art. 123(3) EPC are also met.

4.2 Inventive step

4.2.1 Closest prior art

As claim 2 of the first auxiliary request is restricted to glass fibre filled polyamides the closest prior art in this case is D11, as it, contrary to D2, specifically discloses glass filled polyamide.

4.2.2 In view of the evidence, particularly that advanced with the letter of September 2011 (see sections VIII, XI.(g) and 3.2.4, above) the technical problem to be solved vis-à-vis D11 can be formulated as being to provide compositions with enhanced flame retardancy. This problem was solved according to the claims by incorporating metal phosphinates.

4.2.3 Obviousness

Metal phosphinates are known from D2 where they are stated to be useful in "synergistic" flame retardant compositions also containing as the second component a nitrogen compound selected from a number of defined formulae (see section 3.1.1, above). The examples of D2 however provide no data for compositions containing only the nitrogen compound without phosphinate. Accordingly D2 does not provide any evidence of the existence of a true synergistic effect arising from specific combinations of flame retardants in general, let alone specifically for glass fibre reinforced polyamide compositions. Therefore, the improvement in flame retardancy due to the replacement of the phosphates of D11 by the phosphinates of D2 as demonstrated in the evidence of September 2011 could not be foreseen.

Consequently it was not obvious for the skilled person seeking to solve the above mentioned technical problem to do this by incorporation of metal phosphinates into the compositions of D11.

4.3 D17 relates to flame retardant polyester moulding compositions. It has no particular relationship with the above-defined problem nor would it render the subject-matter now being claimed obvious.

4.4 Therefore the Board is satisfied that the subject matter of claim 2 of the first auxiliary request is not obvious in the light of the prior art and that the claims of this request meet the requirements of Art. 56 EPC.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the department of first instance with the order to maintain the patent with claims 1 to 20 according to the first auxiliary request, entitled "6. Hilfsantrag (geändert)" filed during oral proceedings, and a description yet to be adapted thereto.

The Registrar: The Chairman:

E. Goergmaier B. ter Laan