Internal distribution code:
(A) [ ] Publication in OJ
(B) [ ] To Chairmen and Members
(C) [X] To Chairmen

DECISION
of 18 June 1997

Case Number: T 0612/94 - 3.3.3

Application Number: 88311429.0

Publication Number: 0323047

IPC: C08K 5/52

Language of the proceedings: EN

Title of invention:
Phosphate/epoxy stabilizer for extrudable polyester blends.

Applicant:
FORD MOTOR COMPANY
FORD-WERKE AG

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:

"Inventive step - (yes); crucial effect - silence of closest state of the art - demand for further evidence not justified"

Decisions cited:

Catchword:
-

Case Number: T 0612/94 - 3.3.3

DECISION
of the Technical Board of Appeal 3.3.3
of 18 June 1997

Appellant:	FORD MOTOR COMPANY LIMITED Eagle Way Brentwood Essex CM13 3BW (GB)
	FORD-WERKE AKTIENGESELLSCHAFT Werk Köln-Niehl Henry-Ford-Strasse Postfach 60 40 02 D-5000 Köln 60 (DE)
Representative:	Messulam, Alec Moses et al A. Messulam & Co. 24 Broadway Leigh on Sea Essex SS9 1BN (GB)
Decision under appeal:	**Decision of the Examining Division of the European Patent Office dated 16 November 1992 refusing European patent application No. 88 311 429.0 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman:	C. Gérardin
Members:	R. Young
	M. K. S. Aúz Castro

Summary of Facts and Submissions

I. European patent application No. 88 311 429.0, filed on 2 December 1988, claiming a US priority of 28 December 1987 (US 138255) and published under No. 0 323 047 was refused by a decision of the Examining Division dated 16 November 1992, for lack of inventive step in the light, inter alia, of the disclosures of the documents:

D1: DE-A-2 828 552, and
D2: EP-A-0 074 337.

The decision was based on a set of Claims 1 to 14 filed on 12 September 1992 with a letter dated 7 September 1992, and on the description as originally filed with replacement pages 4 to 7, 9, 12, 13 and 15, page 14 having been deleted. Claim 1 reads as follows:

"A stabilizer useful to retard transesterification in moldable polymer blends comprising polyester, wherein the stabilizer comprises;

I. a phosphate-epoxy adduct which is the reaction product of:

(A) phosphate being selected from mono- and di-esters of orthophosphoric acid, said mono- and di ester respectively having two and one ionizable hydrogen atoms; and

(B) epoxy;

wherein said phosphate and said epoxy are reacted in amounts so as to react at least one of said ionizable hydrogen atoms of said phosphate with an epoxide group of said epoxy and

II. a component selected from

(i) a compound having (1) imide or (2) oxazoline functionality;

(ii) hindered phenol compound; and

(iii) mixtures of (i) and (ii), wherein said phosphate-epoxy adduct comprises at least 51 weight percent of said stabilizer."

Claims 2 to 4 are directed to elaborations of the stabiliser according to Claim 1.

Claim 5, an independent claim, is directed to a composition comprising a polyester and a stabiliser as claimed in any one of Claims 1 to 4.

Claim 6, also an independent claim, is worded as follows:

"A moldable composition comprising a polymer blend comprising polyester and a stabilizer as claimed in any one of Claims 1 to 4, the polymer blend comprising;

(I) aromatic compound selected from the group consisting of; polysulfone, polyarylslfone (sic), polyetherketone, polyester ether ketone and polyarylate, wherein the prolyarylate (sic) is the reaction product of at least one dihydric phenol and at least one aromatic dicarboxylic acid;

(II) polyester being the reaction product of an aliphatic or cycloaliphatic diol, or mixtures thereof and at least one aromatic dicarboxylic acid; and

(III) at least one thermoplastic polymer selected from the group consisting of an aromatic polycarbonate, a styrene resin, a vinyl chloride polymer, a poly(arylether), a copolyethester (sic) block copolymer, and a polyhydroxyether; and wherein

said phosphate-epoxy adduct is present in said composition in an amount of at least 0.1 weight percent based on the weight of said polymer blend."

Claims 7 to 14 are directed to elaborations of the composition according to Claim 6.

II. According to the decision, D2, which was considered to represent the closest state of the art, described extrudable polyester compositions stabilised against thermal degradation by a composition which comprised (i) a polyepoxide and (ii) a compound selected from capped diisocyanates, bisoxazolines or diimides. Especially preferred were such compositions which further comprised, as a costabiliser, (iii) a phosphate. Preferred phosphates were triesters. Although it had been submitted that the stabiliser compositions of D2 were not effective at temperatures above 285°C, this had not been supported by evidence, and consequently could not be taken into consideration in the formulation of the technical problem for the evaluation of inventive step. This problem could thus only be defined as being to provide **further** stabiliser compositions for polyester resins. The selection, instead of the preferred triester phosphates, of mono- or di-acid phosphates, which also fell under the broad term "phosphates" and were in any case taught in D1 as stabilising polyesters against thermal degradation, was obvious. This obvious selection would have led inevitably to the formation, under the conditions found in the extruder, of the adducts (I) defined in Claim 1 of the application in suit. The finding that these obvious

.../...

stabiliser compositions were active at temperatures over 285°C could only be regarded as a discovery of an added effect, and not as the surprising result of an inventive activity. Consequently, the subject-matter of Claims 1 and 2 did not involve an inventive step.

III. On 16 January 1993, a Notice of Appeal was filed, together with payment of the prescribed fee. In the Statement of Grounds of Appeal, filed on 13 March 1993, the Appellant referred to the US equivalent of D2 (US-A-4 499 219) and argued substantially as follows:

(i) The decision under appeal had ignored the fact that, according to Claim 1 of the application in suit, the selected phosphate was not merely mixed, but actually reacted with the epoxy compound. The use of such a reaction product rather than a mere mixture of the components was, however, necessary to achieve satisfactory stabilisation, because, at the desired high processing temperatures, the mono- or diester phosphate would either be decomposed or transformed into a non-stabiliser species. It was for this reason that the claimed composition included a high molecular weight phosphate-epoxy adduct.

(ii) The whole emphasis of D2 was to the use of a stabiliser composition comprising a mixture of (i) a polyepoxide, (ii) a compound selected from capped diisocyanates, bisoxazolines or diimides and (iii) a **triester phosphate**. There was only one reference to the inclusion of **phosphates in general** and this, in isolation, would not provide any encouragement to use a mono- or di-ester phosphate. Even more significant was the fact that there was no encouragement to react the selected phosphate with the epoxy compound in order to achieve stabilisation at high temperatures. Indeed, it would not be possible to form an adduct with an epoxy since a triester phosphate did not have any active hydrogen atoms available for reaction with the epoxy.

(iii) D1 was directed to light stabilised polyesters and was not in any way concerned with achieving heat stabilisation in polyester blends. Furthermore, considering the low concentration of the relevant components in the total bulk of material, the possibility of the ester and epoxy contacting in such circumstances would be minimal and the amount of adduct formed, according to the assumption made, would be very slight.

(iv) In any event, it could not be accepted that the person skilled in the art, even if he was aware of D1, would realise that the adduct I might be formed in the extruder and an advantage might be gained by forming such an adduct in isolation and then mixing it with component II and using the mixture as a stabiliser in polyester compositions.

IV. The Board drew attention, in a communication issued on 3 January 1997, inter alia to certain errors in independent Claim 6 and also to incorrect appendancies of certain claims.

V. The Appellant filed, on 25 January 1997, new pages 34 and 35 bearing an amended version of Claims 4 to 14, and specified, in a further submission, filed on 17 June 1997, the text on the basis of which the grant of a patent was sought.

VI. The Appellant thus requested that the decision under appeal be set aside, and that a patent be granted on the basis of the following documents:

Claims:	1 to 3 filed on 12 September 1992 with letter dated 7 September 1992; 4 to 14 filed on 25 January 1997 with letter dated 21 January 1997;
Description:	pages 1 to 3, 8, 10, 11, and 16 to 32 as originally filed; pages 4 to 7, 9, 12, 13 and 15 (page 14 as originally filed having been deleted) as filed on 12 September 1992 with letter dated 7 September 1992.

Reasons for the Decision

1. The appeal is admissible.

2. *Admissibility of amendments*

2.1 The text of the application underlying the present decision differs from that underlying the decision under appeal only in the correction, in Claim 6, of typographical errors, and in Claims 11 and 14, of the appendancy (from Claim 7 to Claim 6).

2.2 The typographically correct forms of the polymers referred to in Claim 6 are supported by the original description on page 6. The change of appendancy follows the re-numbering of original independent Claim 7 as Claim 6.

These changes do not contravene the provisions of Article 123(2) EPC.

2.3 The amendments of the text as filed leading to the text underlying the decision under appeal were found, in the decision under appeal, to comply with the requirements of Article 123(2) EPC. The Board sees no reason to differ from this finding.

2.4 Consequently, there are no objections under Article 123(2) EPC to the text underlying the present decision.

3. *The closest state of the art; the technical problem.*

The application in suit is concerned with a stabiliser useful to retard transesterification in mouldable polymer blends comprising polyester, especially during processing of the blends at elevated temperatures, wherein the stabiliser comprises:

I. an orthophosphoric acid ester compound; and

II. a component selected from

(i) a compound having (1) imide or (2) oxazoline functionality;

(ii) hindered phenol compound; and

(iii) mixtures of (1) and (ii), wherein the phosphate ester compound comprises at least 51 percent of the stabiliser (cf. Claim 1).

The stabiliser may be present in an amount of at least 0.1 percent by weight of a polymer blend comprising an aromatic compound; a polyester and at least one thermoplastic polymer (Claim 6).

.../...

Such a composition is known, however, from D2, which was considered, in the decision under appeal, to represent the closest state of the art.

3.1 According to D2, a mouldable composition based on a thermoplastic polyester is stabilised against thermal degradation during processing at elevated temperatures, by a polyepoxy compound or a polycarbodiimide together with a second thermal stabiliser (page 1, first paragraph). The second stabiliser may be a capped diisocyanate, a bisoxazoline or a dicarboxylic acid imide (page 2, line 5 to page 3, line 5). The polyester can be a homo- or co-polyester of aliphatic, cycloaliphatic or aromatic dicarboxylic acids and diols or of hydroxycarboxylic acids (page 3, paragraph 3). The first and second stabilisers can each be present in an amount of from 0.01 to 10, in particular 0.1 to 3 percent, based on the polyester (page 9, paragraph 3). The composition may additionally contain 3 to 25 wt%, based on the polyester, of a flame retardant additive (page 13, paragraph 4). Further conventional additives, such as fillers, may be present. It is particularly advantageous to add, as costabiliser, 0.1 to 5 wt%, based on the polyester, of a phosphate. This preferably has the formula $(R^3O)_3P=O$, in which R^3 is C_6 to C_{18} alkyl, C_5 to C_{15} cycloalkyl, C_6 to C_{12} aryl, C_7 to C_{18} alkaryl or aralkyl. Particularly preferred is triphenylphosphate (page 14, last paragraph to page 15, line 2). The stabilising effect is already noticeable at the stage of re-granulation (page 15, paragraph 3).

According to the examples, polybutyleneterephthalate having an intrinsic viscosity of 1.18 dl/g is mixed with certain additives and melted, by means of a laboratory extruder (20 mm screw) at a cylinder temperature of 250°C to give a stabilised granulate. This granulate is then injection moulded into small rod-shaped samples, at cylinder temperatures of 250°C and 270°C respectively. The relevant additives are :

A = Bisphenol-A epoxy resin having 0.38 epoxy equivalents/kg;

B = a diimide compound of specified formula;

C = an oxazoline compound of specified formula; and

D = triphenyl phosphate.

The tabulated results of measuring the intrinsic viscosity of the granulate before and after processing at the relevant temperatures, show that there is very little loss of intrinsic viscosity, whether at processing temperatures 250°C or 270°C, compared with an unprocessed granulate, in a composition comprising, as additive, 1 wt% A, 0.5 wt% B and 0.5 wt% D, or in a composition containing 1 wt% A, 0.5 wt% C and 1 wt% D (pages 15, 16, and Table page 16).

3.2 Compared with this state of the art, the technical problem may be seen in the provision of compositions that stabilise polyester against deterioration of properties under more extreme processing conditions.

3.3 The solution proposed according to Claim 1 of the application in suit is to provide thermal stability at processing temperatures in excess of 285°C by replacing component D (triphenyl phosphate) by a mono- or di-ester of orthophosphoric acid having respectively two or one ionisable hydrogen atoms (hereinafter component D'); pre-reacting component D' with component A (epoxy) to form a phosphate-epoxy adduct; and then combining the phosphate-epoxy adduct with component B or C to form an additive, whereby component B or C may also be a hindered phenol compound, so that the adduct forms at least 51 wt% of the additive.

3.4 It can be seen from the examples of the application in suit that polyester compositions comprising the relevant additives, including aromatic polyester, polycarbonate and polyethylene terephthalate can be processed at temperatures typically in excess of 285°C, and even above 300°C, for periods of time of the order of half an hour, without unacceptable loss of thermal properties, such as capability of crystallisation, and with retention of thermal stability as measured by differential scanning calorimetry (Examples 1 to 4).

3.5 The finding of the decision under appeal that the technical problem, starting from D2, could only be defined as being to provide **further** stabiliser compositions for polyester resins (Reasons for the decision, point 8), because no evidence had been provided that the compositions according to D2 were not equally capable of being used at temperatures in excess of 285°C (Reasons for the decision, point 7), is based on a misconception concerning the disclosure of D2. There is no indication that the stabilisers disclosed in D2 are capable of being used at processing temperatures in excess of those which would normally be used. These are stated in the application in suit itself to be **below** about 285°C (pages 30 to 33), which is in turn corroborated by the fact that, in the examples according to D2, the processing temperatures used are either 250°C or 270°C.

Consequently, D2 is entirely silent concerning the crucial effect forming the basis of the problem addressed by the application in suit, namely the extension of thermal stability performance into temperature ranges higher than those usual in the art. Even if it were assumed, for the sake of argument, that the additive compositions according to D2 would, if tested at higher processing temperatures, turn out to be inherently capable of providing stability at processing temperatures in excess of 285°C, such performance is not made directly and unambiguously available by the disclosure of D2 and therefore to this extent does not form part of the state of the art with which a comparison can be required for the establishment of the technical problem.

3.6 Nor can such performance be interpolated from the general knowledge of the skilled reader, since the temperatures adressed by the application in suit are not a selection from, but lie above the maximum disclosed in, D2. On the contrary, it belongs to such general knowledge that, other things being equal, providing thermal stability at higher temperatures will be more difficult than at lower temperatures. Furthermore, the application in suit itself specifically refers to various aspects of loss of mechanical properties in polyesters at processing temperatures in excess of about 285°C (page 3, lines 1 to 17), and cites other, independent state of the art in this connection (page 3, line 19 to page 4, line 14). Consequently, there is no reason to assume that the thermal stability problem, only disclosed as being solved at a lower temperature in D2, will automatically be solved at a significantly higher temperature.

3.7 Indeed, to adopt this position is tantamount to asserting that the entire subject-matter of the application in suit is directed to the solution of a non-existent problem. An Applicant is, however, under the greatest obligation of good faith in presenting his invention to the public. Not only this, but there is no reason to doubt what is stated by the Applicant, since all the evidence, whether from general knowledge, or from the explicit statements in the application itself, or from the silence of D2, already points in the same direction.

3.8 Whilst it is undoubtedly justified for the Examining Division to call for further evidence in appropriate cases, for instance where there is a strong, objective reason to doubt what is said in the application, the present case, for the reasons given above, is not one of them.

3.9 Consequently, it was not justified, in the absence of such further evidence, to reformulate the technical problem in the less ambitious terms adopted in the decision under appeal.

3.10 In summary, the Appellant is entitled to a statement of problem in the terms formulated by the Board (section 3.2, above). Furthermore, it is credible to the Board, in view of the thermal stability evidently achieved at the higher processing temperatures (section 3.4, above), that the stated problem is effectively solved by the claimed measures.

4. *Novelty*

Novelty of the claimed subject-matter was explicitly recognised in the decision under appeal (Reasons for the decision, point 3). The Board sees no reason to differ from this finding. Consequently, the claimed subject-matter is held to be novel.

5. *Inventive step*

It is necessary, in the assessment of inventive step, for the Board to consider whether the person skilled in the art, starting from D2 and wishing to extend the range of processing conditions under which thermal stability could be assured, would have expected that such an extension into a range of processing temperatures in excess of 285°C would be enabled by making the series of modifications forming the solution proposed by Claim 1 of the application in suit (section 3.3, above).

5.1 The disclosure of D2 does not offer any clue as to what might happen at such higher than normal processing temperatures. Indeed, it does not even specifically disclose a fully aromatic polyester of a kind which might, because of its high viscosity, have required such higher processing temperatures (application in suit, page 2, lines 9 to 33). On the contrary, it specifically warns against the addition of high viscosity polyesters because of, amongst other things, problems in processing (D2, page 1, penultimate paragraph). Thus D2 contains no hint to the possibility of processing at temperatures in excess of 285°C.

5.2 Quite apart from this, there is no suggestion in D2 to replace the triphenyl phosphate component D by a less than fully esterified phosphate, let alone to pre-react it with the epoxy component A to form a phosphate-epoxy adduct.

5.3 The finding in the decision under appeal, that the teaching of D2 extended to all phosphates, and that there was no indication that acid phosphates should not be used does not in itself demonstrate that the choice of specifically acid phosphates is obvious.

5.4 The further finding of the decision under appeal in this connection, that acid phosphates were stated in D1 to be commonly used in the art to stabilise polyetheresters against thermal degradation (page 5, lines 32 to 36), whilst correct as far as it goes, neglects what is subsequently said in D1 regarding such acid phosphates. Firstly, the use of such acid phosphates is presented, not as part of the teaching of D1, but rather as part of an acknowledgment of other art which has not been cited in the present proceedings. Secondly, such acid phosphates, according to this acknowledgment, have major disadvantages as additives due to their acid properties (D1, page 5, line 37 to page 6, line 7). Consequently, the person skilled in the art would have had no incentive to replace the preferred triphenyl phosphate of D2 by a less preferred acid phosphate according to D1.

5.5 Even if, in spite of the lack of incentive, indeed in spite of the negative remarks in D1 in this respect, such a replacement were nevertheless made in the relevant example of D2, there is no reason to suppose that any reaction would take place between the acid phosphate so chosen and the epoxy component to provide the necessary phosphate-epoxy adduct. On the contrary, according to D2, the additive components are mixed, in the extruder, with the polyester mass to be processed, and not, for instance, with each other under reaction conditions. It is noted in this connection that the method of preparation of the phosphate-epoxy adduct according to the examples of the application in suit always takes at least 24 hours, compared with an extrusion time of the order of, at most, a few minutes. Furthermore, any reaction between such components would self-evidently be seriously impeded, if not completely prevented, by the overwhelming presence of polyester. Once again, there is, in the Board's view, no justification for assuming, against the evidence, that such a reaction would be inevitable.

5.6 Even if it were assumed, against the interests of the Appellant, that some degree of reaction would nevertheless take place and some phosphate-epoxy adduct would thus be formed, it is, in the Board's view, inconceivable that such a reaction should take place to a degree sufficient to provide the adduct in a substantial amount, let alone at least 51 wt% of the additive, as required by the solution of the technical problem (section 3.3, above).

5.7 In summary, there is no suggestion in D2 either of the specific result to be obtained according to the solution of the stated problem, or of the means of obtaining it, or even of means capable of obtaining it, as defined in the solution.

5.8 According to the teaching of D1 (page 2, line 1 to page 5, line 1; Claim 1), there is disclosed a process for preparing a lustreless (matt), linear, light-stabilised polyetherester from terephthalic acid and glycols with up to 15 wt%, based on the polyetherester, of a polyethyleneglycol, whereby an oxygen-containing compound of phosphorus is added to the reaction mixture, the compound being added after the esterification or transesterification step or during the polycondensation reaction. The compound is a triester of phosphoric acid having 1 or 2 alkyl groups of 1 to 4 carbon atoms as well as, respectively, 2 or 1 alkyleneglycol or polyalkyleneglycol groups with 2 to 4 carbon atoms in the alkylene group. The delustering additive is TiO_2.

.../...

The triester may be obtained by reaction of an acid alkylphosphate having 1 to 2 alkyl groups of 1 to 4 carbon atoms with an alkylene oxide having 2 to 4 carbon atoms until the resulting product has an acid number less than 1 (Claim 2).

The acid phosphate is preferably a methyl phosphate with 1 or 2 methyl groups per phosphorus atom, and the alkylene oxide is preferably ethylene oxide (Claims 3, 4).

The amount of triester may be such as to provide a phosphorus content in the polyester of 20 to 800 ppm, preferably 30 to 300 ppm, based on the final polyester (Claim 5).

The product may be utilised in textiles, e.g. in the form of fibres or filaments (page 11, lines 11 to 15).

5.9 Thus, the teaching of D1 is not concerned with the problem of elevated temperature polymer processing, but rather with providing light stability in a specific class of polyetheresters, the internal polyether groups of which have been rendered light sensitive by the presence of TiO_2. In particular, there is no teaching that the phosphate compounds taught according to D1 would have a relevant utility in addition to that of light stabilisation.

5.10 Consequently, the skilled person faced with the stated problem would have had no incentive to consult D1 because of its lack of relevance to the task in hand.

5.11 Even if an attempt were nevertheless made to utilise the teaching of D1 in some way in relation to the stated problem, the remarks in the document concerning the disadvantages of acid phosphate esters as additives compared with neutral esters would discourage any attempt to replace the triphenyl phosphate exemplified in D2 by such an acid phosphate (sections 5.5, 5.6, above). As to the neutral phosphate esters taught according to D1, to the extent to which these correspond to the phosphate-epoxy adducts of the application in suit, whilst the skilled person might conceivably consider adding them to other polyester compositions, he would not do so in the expectation of solving the stated problem.

5.12 In other words, the subject-matter of Claim 1 does not arise in an obvious way from the state of the art. Hence, the subject-matter of Claim 1 and, by the same token, that of dependent Claims 2 to 4, involves an inventive step. Furthermore, the subject-matter of independent Claims 5 and 6, as well as that of dependent Claims 7 to 14 also involves an inventive step, because all these claims require, as a limitation, the presence of a stabiliser according to 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 the following documents:

Claims: 1 to 3 filed on 12 September 1992 with letter dated 7 September 1992;
4 to 14 filed on 25 January 1997 with letter dated 21 January 1997;

Description: pages 1 to 3, 8, 10, 11, and 16 to 32 as originally filed;
pages 4 to 7, 9, 12, 13 and 15 (page 14 as originally filed having been deleted) as filed on 12 September 1992 with letter dated 7 September 1992.

The Registrar: The Chairman:

C. Gérardin

E. Görgmaier C. Gérardin