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**D E C I S I O N**  
**of 7 April 2003**

**Case Number:** T 0975/00 - 3.3.3

**Application Number:** 95911758.1

**Publication Number:** 0745106

**IPC:** C08K 13/00

**Language of the proceedings:** EN

**Title of invention:**

Polyamides having improved color and processibility and process for manufacturing

**Patentee:**

E.I. DU PONT DE NEMOURS AND COMPANY

**Opponent:**

DSM N.V. Patent Department

**Headword:**

-

**Relevant legal provisions:**

EPC Art. 54, 56

**Keyword:**

"Novelty (no) - product-by-process claims (main request)"  
"Inventive step (yes) - separate stabiliser addition not obvious (first auxiliary request)"

**Decisions cited:**

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**Catchword:**

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Case Number: T 0975/00 - 3.3.3

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.3  
of 7 April 2003

**Appellant:** E.I. DU PONT DE NEMOURS AND COMPANY  
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**Representative:** Abitz, Walter, Dr.-Ing.  
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**Respondent:** DSM N.V.  
(Opponent) Patent Department  
P.O. Box 605  
NL-6160 AP Geleen (NL)

**Representative:** -

**Decision under appeal:** Decision of the Opposition Division of the  
European Patent Office posted 2 August 2000  
revoking European patent No. 0 745 106 pursuant  
to Article 102(1) EPC.

**Composition of the Board:**

**Chairman:** R. J. Young  
**Members:** P. Kitzmantel  
U. J. Tronser

## Summary of Facts and Submissions

I. Mention of the grant of European patent No. 0 745 106 in respect of European patent application No. 95 911 758.1 in the name of E.I. Du Pont de Nemours and Company, which had been filed on 15 February 1995 as PCT/US95/01950 claiming a US priority of 16 February 1994, was announced on 1 October 1997 on the basis of 14 claims, independent Claims 1, 2, 9 and 11 to 14 reading as follows:

"1. A process for preparing a polyamide composition comprising polymerizing at least one polyamide-forming reactant in the presence of, or introducing into a polyamide melt:

(a) a phosphorus compound selected from the group consisting of

(1) phosphorous acids;

(2) phosphorous acid salts selected from the group consisting of phosphorous acid salts of Groups IA and IIA, manganese, zinc, aluminum, ammonia, and alkyl and cycloalkyl amines and diamines; and

(3) phosphorous organic esters that do not have direct carbon phosphorous bonds and which undergo hydrolysis in the presence of water to form inorganic phosphorous acids or salts; and

(b) a multivalent metal compound selected from the group consisting of carboxylate and water soluble compounds of Group IIA, zinc and aluminum;

wherein the phosphorus compound is added in an amount sufficient to yield a phosphorus concentration ranging from 0.097 mol to 2.091 mols phosphorus per million grams polyamide, and the multivalent metal compound is added in an amount sufficient to yield a multivalent

metal compound concentration ranging from 0.097 mol to 50 mols per million grams polyamide."

"2. A process for preparing a polyamide composition comprising

(a) polymerizing at least one polyamide-forming reactant in the presence of a phosphorus compound selected from the group consisting of

(1) phosphorous acids;

(2) phosphorous acid salts selected from the group consisting of phosphorous acid salts of Groups IA and IIA, manganese, zinc, aluminum, ammonia, and alkyl and cycloalkyl amines and diamines; and

(3) phosphorous organic esters that do not have direct carbon phosphorous bonds which undergo hydrolysis in the presence of water to form inorganic phosphorous acids or salts, to form a polyamide melt; and

(b) introducing into said polyamide melt a multivalent metal compound selected from the group consisting of carboxylate and water soluble compounds of Group IIA, zinc and aluminum;

wherein the phosphorus compound is added in an amount sufficient to yield a phosphorus concentration ranging from about 0.097 mol to about 2.091 mols phosphorus per million grams polyamide, and the multivalent metal compound is added in an amount sufficient to yield a multivalent metal compound concentration ranging from about 0.097 mol to about 50 mols per million grams polyamide."

"9. A polyamide composition prepared in accordance with the process of Claim 1 or Claim 2."

"11. A polyamide composition prepared in accordance with the process of Claim 3."

"12. A polyamide composition prepared in accordance with the process of Claim 4."

"13. A polyamide composition prepared in accordance with the process of Claim 7."

"14. A polyamide composition prepared in accordance with the process of Claim 8."

Claims 2 to 8 were dependent on Claims 1 and/or 2, Claim 10 was dependent on Claim 9.

II. Notice of Opposition requesting revocation of the patent in its entirety on the grounds of Article 100(a) EPC was filed by DSM N.V. on 1 July 1998.

The opposition was based on documents

D1: US-A-3 173 898,

D2: US-A-3 384 615,

D3: DE-A-1 142 696,

D4: US-A-3 352 821,

D5: US-A-3 640 948, and

D6: US-A-4 471 081.

III. By its decision orally announced on 28 June 2000 and

issued in writing on 2 August 2000 the Opposition Division revoked the patent.

This decision was based a set of nine claims of a main request submitted on 29 April 1999 and on sets of five claims each of a first and a second auxiliary request, both submitted at the oral proceedings before the Opposition Division held on 28 June 2000.

(i) Independent Claims 1, 6, 8 and 9 of the main request read as follows:

"1. A process for preparing a polyamide composition comprising

(a) polymerizing at least one polyamide-forming reactant in the presence of a phosphorous compound selected from the group consisting of

- (1) phosphorous acids;
- (2) phosphorous acid salts selected from the group consisting of phosphorous acid salts of Groups IA and IIA, manganese, zinc, aluminum, ammonia, and alkyl and cycloalkyl amines and diamines; and
- (3) phosphorous organic esters that do not have direct carbon phosphorous bonds and which undergo hydrolysis in the presence of water to form inorganic phosphorous acids or salts, to form a polyamide melt; and

(b) introducing into said polyamide melt a multivalent metal compound selected from the group consisting of carboxylate and water soluble compounds of Group IIA, zinc and aluminum; wherein the phosphorous compound is added in an amount sufficient to yield a phosphorous concentration ranging from 0.097 mol to 2.091

mols phosphorous per million grams polyamide, and the multivalent metal compound is added in an amount sufficient to yield a multivalent metal compound concentration ranging from about 0.097 mol to about 50 mols per million grams polyamide."

"6. A polyamide composition prepared in accordance with the process of Claim 1."

"8. A polyamide composition prepared in accordance with the process of Claim 4."

"9. A polyamide composition prepared in accordance with the process of Claim 5."

Claims 2 to 5 are dependent on Claim 1, Claim 7 is dependent on Claim 6.

- (ii) The first auxiliary request comprises only the process Claims 1 to 5 of the main request.
- (iii) The second auxiliary request is identical to the first auxiliary request, but for the following "proviso" appended to Claim 1:  
"... to yield a multivalent metal compound concentration ranging from about 0.097 mol to about 50 mols per million grams polyamide with the proviso that the molar ratio of metal to phosphorous is at least 1.4." (emphasis by the Board)

IV. That decision arrived *inter alia* at the following conclusions:

- (i) New experimental evidence submitted by the Patentee three weeks only before the oral proceedings was disregarded by the Opposition Division because it had not been filed in accordance with the time limit set under Rule 71a EPC and because it was not considered to be *prima facie* relevant. The latter conclusion was taken because, in the Opposition Division's view, the different times of exposure to elevated temperatures of the "inventive" Example and the "comparative" Example (according to D3) made it impossible to attribute the lower YI (yellowness index) of the "inventive" Example to the presence of the P-compound (phosphorus compound) already during the polyamide polymerization.
  
- (ii) The main request was not allowable because product-by-process Claim 6 lacked novelty over the products disclosed in D1, Example III and D3, Examples 1 and 3.
  
- (iii) The first auxiliary request was not allowable because the subject-matter of its claims (identical to Claims 1 to 5 of the main request) was not inventive over the closest prior art represented by D3, a document already teaching the combined use as polyamide melt stabilizers of P-compounds with alkaline or alkaline earth metal or zinc salts of alkane (di)carboxylic acids in amounts matching those of the patent in suit. Since evidence was lacking for any unexpected technical effect caused by the presence of the P-compound already during the polymerization stage of the polyamide, the only technical problem that could objectively be recognized was the provision



of a further process for preparing polyamide compositions. The solution of this problem was, however, obvious in the light of the disclosures of D1 and D2 which taught the addition of P-compounds during the polymerization stage.

- (iv) The second auxiliary request was not allowable because the feature of the metal to phosphorus molar ratio of at least 1.4 lacked a basis in the original disclosure; the teaching of the three examples in Table 1 from which the ratio of 1.4 was taken could not be generalised.

V. On 22 September 2000 the Patentee (Appellant) lodged an appeal against this decision and paid the appeal fee on the same day. The Statement of Grounds of Appeal was submitted on 12 December 2000 and supplemented by a further written submission dated 26 April 2001.

VI. The arguments of the Appellant in its written submissions may be summarised as follows:

- (i) Contrary to the Opposition Division's view, it was not obvious to split the common addition of P-compound and metal compound. Indeed, neither would such a measure make sense when a combination of these compounds was used as polyamide polymerization catalyst (as according to D1) nor when it was used as stabilizer (as according to D2 and D3) because also in this event the desired effect was dependent on the combined action of these compounds (cf. D3 column 3, lines 5 to 21).

- (ii) The inventive separation of the point in time of

the addition of the two compounds (i.e. presence of P-compound at polymerization stage, addition of metal compound to polyamide melt) surprisingly led to a better heat stabilisation of the polyamide composition, as demonstrated by the Appellant's test report of 7 June 2000 which was not considered by the Opposition Division. This report showed that the "inventive" resins initially appeared less yellow and had also a lesser degree of colour build-up on storage as compared with resins according to D3:

Polymer	rel.Visc.	YI, Hunter	Acid ends m/ppm	Amine ends m/ppm
Polymer 1*): no catalyst at all	51.3	+ 3.8	78.9	51.4
+ 100 ppm SHP= Na- hypophosphite monohydrate + 1100 ppm Ca-ac = Ca-acetate monohydrate		+ 34.0		
Polymer 2**): 100 ppm SHP	56.0	- 1.2	84.2	45.2
+ 1100 ppm Ca-Ac		+ 25.3		

\*) Nylon 66 preparation without any additive; SHP and Ca-ac added to melt

\*\*) Nylon 66 preparation with SHP; Ca-ac added to melt

These results were meaningful in spite of the different heat exposure times of polymers 1 and 2 because these different exposure times were required in order to get comparable molecular weights which was in turn a prerequisite for a fair comparison.

(iii) Moreover, the further experimental evidence submitted by the Appellant on 26 April 2001 which comprised the Nylon 66 compositions Lot ML08S23 ("inventive"), Lot # ML08S20 ("comparative" according to D3) and Lot # ML08S21 ("comparative" according to D1) showed that under essentially identical polymerisation and extrusion conditions the "inventive" composition exhibited a YI superior by about 31% to the YI of the composition according to D1 and by about 18% to the YI of the composition according to D3.

sample according to	addition of P-compound	addition of Ca compound	RV of as made polyamide	initial YI of polyamide	mol ratio Ca/P	YI extruded
# ML08S23: inv.	to polym.	to melt	60.9	-0.2	1/1	25.7
# ML08S21: D1	to polym.	to polym.	57.0	+0.4	1/2	37.4
# ML08S20: D3	to melt	to melt	50.3	+3.6	1/6	31.3

VII. The arguments of the Respondent (Opponent) contained in its submission dated 19 July 2001 may be summarised as follows:

(i) Contrary to the Appellant's assertion, D3 was "completely indifferent with regard to the way or moment of adding the compounds" (page 1, second

paragraph of the Respondent's submission).

- (ii) The Appellant's evidence of 7 June 2000 was inadequate to show an advantage of the claimed invention. The desired preparation of high molecular weight products did not free the Appellant from "making adequate comparisons with the prior art, introducing differences only on the features presented as essential for the alleged invention" (page 1, third paragraph of the Respondent's submission).
- (iii) A comparison of the initial YI values of the three lots comprised by the further experimental evidence submitted by the Appellant on 26 April 2001 only showed the positive influence, known from D3 (column 1, lines 5 to 8), of the presence of a P-compound on the yellowing of the polymer during polymerization.
- (iv) Similarly, the different YI values of the subsequent extrusion experiments could be explained by the absence of a metal compound in sample 1 (Lot # 08S21 (D1)) as compared to the other samples; and the higher YI value of sample 3 (Lot # 08S20 (D3)) as compared to sample 2 (Lot # 08S23 ("invention")) only was a consequence of the higher initial YI.
- (v) Consequently, the Appellant's experimental evidence only proved the advantage of the combined presence of a P-compound and a metal compound, a fact known from the prior art, and could not establish any advantage of their separate addition as according to the alleged

invention.

VIII. On 26 September 2002 the Rapporteur communicated the following provisional comments to the parties and invited them to file observations thereon within a period of two months:

"Provisional comments of the Rapporteur

1. The Appellant requests that the decision under appeal be set aside and that the patent be maintained on the basis of the main request (Claims 1 to 9 of 29 April 1999), first auxiliary request (Claims 1 to 5 of 29 April 1999) or second auxiliary request (Claims 1 to 5 of 28 June 2000), respectively.

2. Second auxiliary request:

The Rapporteur concurs with the opinion of the Opposition Division, namely that Claim 1 of this request contravenes Article 123(2) EPC. In addition to the arguments in the decision under appeal (Reasons 4), it has to be kept in mind that the very specific composition of Example 1-4 does not lend itself to a generalisation of its metal/P ratio to the much broader compositional definition of Claim 1.

This issue was not commented upon by the Appellant.

3. Main request:

Novelty of product-by-process Claims 6 to 9:

Also in this respect, the Rapporteur agrees with the conclusion drawn by the decision under appeal (Reasons 5.2), namely that the subject-matter of these claims lacks novelty vis-à-vis D1 and D3.

This finding is not invalidated by the Appellant's argument that the initial yellowness of a polyamide resulted from the specific mixture of molecular entities having different chain lengths including low molecular weight and chain degradation components.

On the one hand low molecular weight and/or chain degradation components which are responsible for the polyamide's yellowness are to be considered as impurities, and on the other hand such compositional peculiarities do not constitute a "fingerprint" by which the process of manufacture can unambiguously be identified because the same compositional features may result from polycondensation conditions which are not covered by present Claim 1.

4. Main request;

Novelty of process Claims 1 to 5:

The available citations do not disclose the process conditions of Claim 1, i.e. the polycondensation in the presence of a P-compound and the introduction of a metal compound into the polyamide melt.

The conclusion of novelty of the Opposition Division is therefore confirmed.

5. Main request;

Inventive step of process Claims 1 to 5:

5.1 Document D3 (DE-B-1 142 696) relates to a process of stabilising the melt viscosity of polyamides by incorporating therein 0.001% to 1% by weight of a phosphorus acid and 0.1% to 2% by weight of alkane(di)carboxylic acids or their (earth)alkali metal or zinc salts. The skilled person is aware that stabilising the melt viscosity is equivalent to

preventing an increase of the molecular weight.

Example 1 of D3 uses disodium hydrogenphophite pentahydrate (P-compound) and Ca-stearate in amounts falling within the ranges of present Claim 1.

5.2 The claimed "invention" differs from the method of D3 by the presence of the P-compound already during the polycondensation reaction.

5.3 Starting from D3 the "objective" problem underlying the present subject-matter can thus be seen in the development of a process for the preparation of polyamide compositions which have a low initial yellowness (YI) and a low degree of color build-up on storage (page 2, lines 9 to 14 of the patent specification).

Since Document D3 already teaches that too big an increase of the molecular weight during melt processing can be prevented by the conjoint presence of certain P- and metal compounds (which are within the scope of present Claim 1) and since a further improvement of this property by the claimed process has not been demonstrated, this aspect of the patent's "subjective" problem cannot be recognised as part of the "objective" problem existing vis-à-vis D3.

Table III of the patent specification shows however that the considerable RV (molecular weight) increase occurring during solid phase polymerisation in the presence of the P-compound SHP can be prevented by the incorporation of Ca- or Al-acetate into the melt (Example 1-1 vs. Examples 1-2, 1-3 and 1-4). This benefit of the subject-matter of D3 is thus preserved

by the "invention".

5.4 From the evidence submitted with the Statement of Grounds of Appeal it appears that the partial problem of achieving a low initial YI is solved by the process according to present Claim 1 ("inventive" polymer 2 vs. "comparative" polymer 3).

5.5 The same holds true for the partial problem of preventing a color build-up on storage (Table II of patent specification).

In the absence of evidence to the contrary (the burden of proof being on the Opponent/Respondent), this conclusion appears justified in spite of the fact that these experiments do not comprise a comparative sample which was prepared strictly in accordance with D3 because this effect is dependent on the simultaneous presence of the two stabilizers and not on the time of their incorporation.

5.6 The issue of inventive step thus turns on the question whether it is obvious to the skilled person starting from D3 and aiming at a solution of the aforementioned "objective" problem to separate the addition of P-compound from that of the metal compound by adding the P-compound already to the polyamide forming reactants, i.e. to the polycondensation mixture.

5.7 There is no information in D3 which could justify the assumption that such a change would be beneficial in any respect:

The disclosure concerning D3's "invention" does not mention any light or heat stabilising properties of the



P-compound and from the fact that D3 acknowledges in its prior art portion that phosphoric acid and its salt had been known as polyamide stabilisers against the action of light, heat and oxygen (column 1, lines 5 to 8) it cannot be concluded that by changing the time of addition of the P-compound from "to the polymer melt" to "to the polycondensation mixture" any advantage could be gained.

5.8 Nor is there any information in any of the further citations that P-compounds which are present during the polyamide polycondensation reaction, when used alone, provide improved heat/light/oxygen resistance. D2 (US-A-3 384 615) and D5 (US-A- 3 640 948) employ stabiliser combinations of certain P-compounds with other compounds.

5.9 It furthermore appears that the known use of certain P-compounds as catalysts for the polyamide polycondensation (D1: US-A-3 173 898; D6: US-A-4 471 081) does not, in the face of the available prior art, suggest their use under the process conditions as claimed by the patent in suit. Any speculation that the improved yellowing properties of the polyamides manufactured according to the patent in suit should be considered as a mere "bonus" effect is therefore not sustainable."

- IX. Neither of the parties submitted any comment on the Rapporteur's above-quoted communication within the time limit set therein.
  
- X. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request, first auxiliary request or

second auxiliary request, respectively.

As a secondary alternative, the Appellant requested that an oral hearing be scheduled.

The Respondent requested that the appeal be dismissed.

### **Reasons for the Decision**

1. The appeal is admissible.

2. *Procedural*

The Appellant, in its submission dated 13 March 2003, confirmed the Board's interpretation of the passage in the Notice of Appeal: "As a secondary alternative, we request that an oral hearing be scheduled", namely that this passage was to be understood as a request for oral proceedings only in the case that, contrary to the opinion expressed in the Rapporteur's communication of 26 September 2002, the Board intended not to allow any of the Appellant's three substantial requests (main, first and second auxiliary request). Since this decision results in the admission of the first auxiliary request there was thus no obligation under Article 116 EPC to hold oral proceedings. Neither was there a need.

3. *Article 113 EPC*

Since the Board concurs with the conclusions drawn by the Rapporteur in his afore-mentioned communication of 26 September 2002 and since the parties did not avail themselves of the opportunity to present their comments

thereupon during the two months period of time set therein, issue of a decision confirming the Rapporteur's conclusions is possible.

4. The relevant conclusions referred to in the preceding paragraph are

(i) that the main request is not allowable because the subject-matter of Claims 6 to 9 lacks novelty over the disclosures of documents D1 and D3, and

(ii) that the first auxiliary request (identical to Claims 1 to 5 of main request) meets the requirements of the EPC, particularly those of Article 54 and 56 EPC, and is thus allowable.

5. The grounds of opposition raised by the Respondent/Opponent do not therefore prejudice the maintenance of the patent on the basis of the set of claims of the first auxiliary request.

## **Order**

### **For these reasons it is decided that:**

1. The decision under appeal is set aside.
2. The case is remitted to the Opposition Division with the order to maintain the patent on the basis of the claims of the first auxiliary request (i.e. Claims 1 to 5 filed on 29 April 1999) after any necessary consequential amendment of the description.

The Registrar:

The Chairman:

E. Görgmaier

R. Young