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DECISION of 11 January 2002

Case Number:

T 0682/98 - 3.3.5

Application Number:

92300183.8

Publication Number:

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IPC:

C01B 25/40

Language of the proceedings: EN

Title of invention:

A process for producing finely divided particles of II type ammonium polyphosphate

Patentee:

Budenheim Iberica, S.L. Sociedad en Comandita

Chemische Fabrik Budenheim R.A. Oetker Clariant GmbH Werk Knapsack

Headword:

Ammonium polyphosphates/BUDENHEIM

Relevant legal provisions:

EPC Art. 56, 113(1)

Keyword:

"Amended claims submitted during oral proceedings - decision taken in the absence of appellant - right to be heard (yes) " "Inventive step (yes, after amendment)"

Decisions cited:

G 0004/92

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 0682/98 - 3.3.5

DECISION of the Technical Board of Appeal 3.3.5 of 11 January 2002

Appellant 02:

(Opponent 02)

Clariant GmbH

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Representative:

Other party: (Opponent 01) Chemische Fabrik Budenheim R.A. Oetker

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Respondent:

(Proprietor of the patent)

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Representative:

Weber, Dieter, Dr. Weber, Dieter, Dr.,

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Decision under appeal:

Interlocutory decision of the Opposition Division

of the European Patent Office posted 22 May 1998

concerning maintenance of European patent

No. 0 494 778 in amended form.

Composition of the Board:

Chairman:

R. K. Spangenberg

Members:

B. P. Czech J. H. Van Moer

Summary of Facts and Submissions

The appeal is from a decision of the opposition division maintaining the patent in amended form.

Claim 1 as amended during the opposition proceedings read as follows (amendment highlighted):

"1. A process for producing finely divided particles of II type ammonium polyphosphate by reacting ammonium phosphate with phosphorous anhydride, each in equimolar quantity, in an ammonia gas atmosphere and at an elevated temperature, which comprises:

a first step of reacting the raw materials as a melt at a temperature of at least 250°C in a nitrogen atmosphere without feeding ammonia gas;

a second step of feeding ammonia gas or an ammonia gasgenerating substance when hot (an ammoniating compound) in an amount of 30 to 90% of the stoichiometric quantity relative to polyammonium hydrogen phosphate or polyammonium phosphate; and

a third step of adding ammonia gas corresponding to in a quantity the remainder of the stoichiometric quantity or more and ageing therewith, to crystallize the resulting II type ammonium polyphosphate."

II. In the contested decision, the opposition division considered the following documents:

D1 = DE-B-23 30 174 D2 = US-A-3 978 195

D3 = DE-A-32 08 202 D4 = US-A-3 653 821

 $D5 = DE-A-39 \ 25 \ 675$ $D6 = EP-A-0 \ 049 \ 763$

D7 = US-A-3 397 035 D8 = Leaflet about AP-B mill

D9 = Hoechst Product Sheet "Exolit 700", October 1989

The opposition division came to the conclusions that the subject-matter of the amended claims was novel and inventive in view of the cited prior art.

III. Upon appeal, both appellants (opponents) maintained their objections concerning the lack of inventive step.

Appellant 01 (opponent 01) essentially based his objections on the disclosure of D1.

Appellant 02 (opponent 02, Clariant GmbH) submitted that the subject-matter of the amended process claims lacked an inventive step in view of D1, D3, D4, D5, D6, D7, D8 and/or D9.

- IV. With his reply, the respondent (patent proprietor) contested the submissions of the appellants, pointing out differences between the claimed invention and the disclosures of the cited documents, both in terms of process features and in terms of the products obtained.
- V. With his letter dated 7 June 1999 appellant 01 filed an experimental report allegedly reproducing and comparing the method according to Example 1 of D1 and according to the claimed invention, respectively, together with a corresponding video tape and an offer of witnesses.
- VI. With his letter dated 15 May 2000, the respondent submitted that the experiments carried out by appellant 01 were not accurate reproductions of Example 1 of D1 and the examples of the contested patent.
- VII. The parties were summoned for oral proceedings, as requested by the respondent and appellant 02.
- VIII. With his letter dated 17 September 2001 appellant 01 withdrew his opposition, the experimental report, the video tape and the offer of witnesses.

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- IX. With his letter dated 17 September 2001, the respondent filed an auxiliary request comprising amended description pages and again discussed the contents of documents D1 and D3 to D9.
- X. With his letter dated 03.01.2002, appellant 02 withdrew his request for oral proceedings and requested continuation of the proceedings in writing.
- XI. Oral proceedings took place on 11 January 2002 in the presence of the respondent. During the oral proceedings, the respondent presented a further amended set of claims 1 and 2 as new and sole request. The claims according to this request read as follows (amendments to granted version highlighted):
 - "1. A process for producing finely divided particles of II type ammonium polyphosphate 80% by weight or more of which have a particle diameter of 10 µm or less and the surface of the crystals being smooth by reacting ammonium phosphate with phosphorous anhydride, each in equimolar quantity, in an ammonia gas atmosphere and at an elevated temperature, which comprises:
 - a first step of reacting the raw materials as a melt for 10 minutes or longer at a temperature of at least 250°C in a nitrogen atmosphere without feeding ammonia gas;
 - a second step of feeding ammonia gas or an ammonia gas-generating substance when hot (an ammoniating compound) in an amount of 30 to 90% of the stoichiometric quantity relative to polyammonium hydrogen phosphate or polyammonium phosphate for a period of from 5 to 30 minutes

a third step of adding ammonia gas corresponding to in a quantity the remainder of the stoichiometric quantity or more and ageing therewith, to crystallize the resulting II type ammonium polyphosphate.

- 2. A process according to claim 1, wherein in the third reaction step, an atmospheric temperature of 240°C or higher is applied for 30 minutes or longer."
- XII. The submissions of the parties, as far as they remain relevant to the claims as amended during the oral proceedings, can be summarised as follows:

Appellant 01 submitted that D1 did not disclose the reaction of the materials as a melt in a nitrogen atmosphere. However, Examples 2 and 3 of the patent would show that this feature did not contribute to the solution of the technical problem stated and was therefore irrelevant for the assessment of inventive step, since the properties of the ammonium polyphosphates (designated as "APPs" in the following) obtained according to Example 1 on the one hand (nitrogen atmosphere), and Examples 2 and 3 on the other hand (ammonia atmosphere), were the same. Concerning the information comprised in Example 5 of D6, he pointed out that D1 was not limited to the method and temperature disclosed in its Example 1, and that present claim 1 did not exclude the presence of APPs other than type II.

Appellant 02 argued that D1 implicitly disclosed the melting of the raw materials before the feeding of ammonia gas, and that it was irrelevant whether nitrogen or air was present at this stage. D1 also lead to very fine particles requiring no further milling

before use. Moreover D3 suggested to carry out some reaction steps under a nitrogen atmosphere. The method as claimed lacked an inventive step in view of documents D1 and D3 to D9, taken alone and/or in combination.

The respondent argued that D1 was to be considered as the closest prior art since it disclosed a process for the preparation of ammonium polyphosphate which was similar to the one claimed, except for the missing "first step of reacting the raw material as a melt for 10 minutes or longer at a temperature of at least 250°C in a nitrogen atmosphere without feeding ammonia gas". D1 did not explicitly disclose an initial melting of the raw materials before the starting of the ammonia feeding. This first step of the claimed method was however essential for avoiding the formation of other crystalline types than type II and for obtaining type II APPs having all the properties referred to in claim 1 as amended. According to D6, the APPs obtained by the method disclosed in Example 1 of D1 would mainly be of the V type. The other documents cited related to different methods and/or APP products. Hence, the skilled person could not derive from D1 or the other documents cited that such a first step would lead to particles having the properties stated in claim 1. Concerning Examples 2 and 3 of the patent, he pointed out that the desired APP particles could only be obtained under very specific conditions when the melting was carried out in the presence of ammonia gas.

XIII. Appellant 02 requested in writing that the decision under appeal be set aside and that the patent be revoked. The respondent requested that the decision under appeal be set aside and that the patent be maintained on the basis of the claims submitted during the oral proceedings and a description to be adapted.

Reasons for the Decision

1. Amendments

- 1.1 No objections under Article 100(c) EPC had been raised against the claims submitted before the first instance. Claim 1 according to the new main request is based on claim 1 as granted and comprises additional restricting features taken from the description and the dependent claims.
- 1.2 The additional properties of the products to be obtained by the process as now claimed, i.e. the particles size distribution and the smooth crystal surface are referred to on page 1, first paragraph and page 8, lines 17 to 23 of the description as filed (page 2, lines 3 to 5 and page 3, lines 48 to 51 of the contested patent).
- 1.3 The feature "as a melt for 10 minutes or longer" is based on page 5, lines 1 to 4, page 6, lines 15 to 20, and claim 6 of the application as filed (page 2, lines 44 to 45, and page 3, lines 10 to 13 and claim 2 of the granted patent). It emanates from the cited passages that the upper temperature limit of 320°C referred to in granted claim 2 is merely a preferred feature.

- 1.4 The added feature concerning the second process step, i.e. "for a period of from 5 to 30 minutes", is based on page 5, lines 5 to 9 and claim 7 of the application as filed (page 2, lines 45 to 47 and claim 3 of the granted patent).
- 1.5 Moreover, the examples of the patent (see Tables 1, 4 and 5) also form a basis for the amendments since they show that type II APPs with all the specified properties may be obtained by working according to the process of claim 1 and balancing the "retention time of the melt" and the amount and addition time of the ammonia gas-generating substance within the indicated ranges. The examples thus further support the amendments to claim 1.
- 1.6 Hence the board is satisfied that the requirements of Articles 123(2) and (3) are met by the amended claims.
- 2. Appellant 02 had been duly summoned to the oral proceedings that he had requested initially. He withdrew his request for oral proceedings shortly before the date scheduled for the oral proceedings without indicating any reasons for doing so. Since oral proceedings had also been requested by the proprietor, the board felt that under these circumstances it was not appropriate to cancel the oral proceedings and to continue the proceedings in writing.

The new request presented at the oral proceedings comprised a fresh set of claims restricted by the incorporation of various features. The process features added were taken from the dependent claims of the patent as granted (see items 1.3 and 1.4 here above). The features relating to the properties of the final product obtained by the claimed process were taken from the description. However, during the entire procedure

up to the oral proceedings, the respondent had repeatedly emphasised the importance and distinctiveness of the physical properties of the APP particles to be obtained. The amendments were carried out to overcome the inventive step objections raised by appellant 02. Hence, the board holds that all of these amendments could have been reasonably expected by appellant 02 and could not have taken him by surprise. Appellant 02 has had sufficient opportunity to comment on such foreseeable amendments during the written proceedings. The amended claims according to the new request cannot, therefore, be considered as surprising facts or evidence within the meaning of opinion G 4/92 (OJ EPO, 1994, 149). Hence, the board holds that the requirements of Article 113(1) EPC are met (see also "Case Law of the Boards of Appeal of the EPO", 3rd. edition 1998, English version, VI-B.3, page 258, last paragraph to page 259, second paragraph).

3. Interpretation of claim 1

Present claim 1 is restricted to a process leading to APP particles with all the indicated properties.

Moreover, it is plausible in view of the results in Table 1, that the APPs obtained implicitly do have relatively high viscosity values. Moreover, the board holds that since the wording of claim 1 is directed to the preparation of type II APP, methods leading to products mainly comprising APPs of other crystalline types than type II are not encompassed by the claim.

Finally, the board holds that in view of the wording of claim 1, the second step referred to in claim 1 merely specifies that 30 to 90% of the required stoichiometric quantity of ammonia must be fed to the reactor within a period of from 5 to 30 minutes. The claim does not

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require the third step to be distinct from the second step, e.g. by means of a different rate of ammonia addition or a different temperature.

4. Novelty

Novelty has not been contested during the opposition and appeal procedures. The board is also satisfied that none of the documents cited discloses a process with all the features recited in claim 1. The differences between the claimed process and the ones disclosed by the prior art emanate in more detail from the following discussion of inventive step.

- 5. Inventive step
- 5.1 Closest prior art document D1
- 5.1.1 Document D1 is a patent family member of document JP-B-53-011280, which was already cited as prior art in the application as originally filed (page 2, line 17). D1 discloses a method for the preparation of various APPs having low solubilities, comprising heating an equimolar mixture of ammonium phosphate and phosphorous pentoxide (phosphorous anhydride in the language of the contested patent) to relatively high temperatures of from 170°C to 320°C in the presence of gaseous ammonia under continuous mixing, kneading and size reduction of the components of the mixture. According to D1, the degree of insolubility of the end product can be controlled by selecting an appropriately high temperature (column 3, lines 31 to 56 and column 4, lines 40 to 49). Moreover, the reaction products obtained according to D1 are so finely divided that a milling step is not required (see column 4, lines 50 to 59). According to Examples 1 to 4 and 6 of D1, the ammonia gas is fed to the reactor in two distinct steps, first at a relatively higher rate and then at a

relatively lower rate. The respondent has repeatedly acknowledged that 89% of the required ammonia amount was fed in the first step, see e.g. his letter dated 17 September 2001, page 3, second paragraph. Since the duration of this first step is one hour, it follows that according to this example an amount of more than 30% and less than 89% must implicitly be fed to the reactor in the first 30 minutes, in the same way as according to the second step of claim 1 of the contested patent (see item 2. here above).

- Hence, the sole method feature not disclosed in D1 is 5.1.2 the "first step of reacting the raw materials as a melt for 10 minutes or longer at a temperature of at least 250°C in a nitrogen atmosphere without feeding of ammonia gas". None of the examples of D1 actually refers to a melt, let alone to keeping the melt at a high temperature for a certain time before starting the addition of ammonia. The board holds that a skilled person would take from the examples of D1 that the feeding of the ammonia is to be started immediately after filling the raw materials into the reactor, without first waiting for the formation of a melt. Whether the pasty mixture of materials generally referred to in the description of D1 (column 3, line 40 and column 5, line 47) can be considered as a melt or not is irrelevant, since according to the cited passages this pasty state is only reached after the beginning of the ammonia addition.
- 5.1.3 D1 does not explicitly mention the crystal type of the APPs obtained. However, as the respondent rightly observed, D6, Example 5, discloses that a reproduction of Example 1 of D1 leads to APPs predominantly of the type V, although comprising noticeable amounts of type II APP. The board in agreement with the opposition division (see contested decision, page 5, third

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paragraph, last sentence) - has no reason to doubt these results. The other reproduction of this example of D1 as carried out according to D5 (comparative Example 1) is clearly flawed, since the starting temperature applied is much lower (150°C) than according to D1 (265°C). The corresponding spectrum given in Figure 2 of D5 can thus not establish the crystal type of the products obtained according to Example 1 of D1. Finally, the (withdrawn) evidence provided by appellant 01 cannot establish either that the products according to Example 1 of D1 would be of the type II. As pointed out by the respondent, the experiments 2 and 3 of appellant 01 are not true reproductions of Example 1 of D1. In particular, the feeding of ammonia is only started after the formation of a melt. As already indicated under 5.1.2 here above, this information is not comprised in D1. Hence, the board accepts that Example 1 of D1 does not lead to a type II product, and has no reason to believe that the other examples of D1, carried out under similar conditions, would lead to different results.

5.1.4 Finally, D1 does not disclose the particle size distribution referred to in claim 1. Only Example 5 of D1 comprises an explicit indication of the particle size distribution obtained. The board holds that neither the general mention of "fine" particles (column 4, line 51), nor the particle size distributions referred to in Example 5, according to which up to 98% of the APP particles obtained have a size of less than 63 µm, are anticipations of the particle size distribution indicated in claim 1. A possible overlap of the size distributions, in contrast with particle size ranges, as claimed and as shown in Example 5 of D1, respectively, cannot take away the novelty of this feature. Moreover, it was not submitted and the board has no reason to believe that the

particles obtained according to the other examples of D1 would show the particle size distribution as claimed.

- 5.1.5 However, the board can assume in the favour of appellant 02 that equimolar amounts of ammonium phosphate and phosphorous pentoxide would form a melt at temperatures near the upper limit (350°C) of the range indicated in claim 1 of D1, and that the process of D1 could also lead to type II products under specific conditions. This view is confirmed by comparative Example 2 according to the contested patent, which is carried out under conditions similar to those according to the examples of D1, except for the higher temperature (290°C to 300°C) used, and which leads to a type II APP having a high viscosity but relatively large particle sizes (see Table I of the patent).
- 5.1.6 Summarising, the board came to the conclusion, that although it may be possible to obtain type II APPs having a low solubility by working under the conditions specified in claim 1 of D1, neither Example 1, nor any other example of D1 actually leads to APPs mainly composed of the II type. In any case, D1 does not disclose all of the process steps recited in claim 1 and does not, implicitly or explicitly, disclose the preparation of APP particles mainly of the II type having the particle size distribution and crystal smoothness specified in claim 1.
- 5.2 The technical problem
- 5.2.1 According to the contested patent, the technical problem to be solved by the invention consists in providing a method for the preparation of finely divided particles of APP overcoming some of the disadvantages associated with the use of the known

APPs, including the ones known from D1, in particular when incorporated into polyolefin resins, moulded products, fibres and the like (see page 2, lines 14 to 31). During the oral proceedings, in view of amended claim 1, the respondent suggested a more specific formulation of the technical problem with reference to page 2, lines 3 to 5 and page 3, lines 48 to 56 of the contested patent (see also page 2, line 58 to page 3, line 2), according to which the problem consisted in the provision of a process leading to type II APP with all of the specific properties referred to in claim 1. More particularly, the APP particles obtained must

- (i) be difficult to extract when incorporated in polyolefin materials, due to their low solubility and relatively high suspension viscosity values,
- (ii) have a particle size distribution not requiring further grinding of the individual particles, 80% or more by weight of the particles having a diameter of 10 µm or less,
- (iii) and, therefore, smooth crystal surfaces.

For the sake of argument, the board assumes in the favour of appellant 02 that D1 discloses a process leading - under certain conditions embraced by the general teaching of the document - to type II APPs having viscosity and solubility values comparable to the ones of the products obtained according to the claimed process. Taking D1 as the closest prior art, the technical problem can thus at least be seen in the provision of a further process leading to type II APPs with low solubility and high viscosity values, but with a particle size distribution shifted towards smaller

sized particles, which does not require any intensive grinding leading to deterioration of the smoothness of the crystal surface.

- 5.2.2 As already noted under 1.5 here above, the examples of the contested patent (see Tables 1,4 and 5) show that the method according to amended claim 1 can lead to particles having all of these desired properties, whereas variations not covered by present claim 1 lead to products with different properties (see e.g. the comparative examples represented in Table 5, and in particular comparative Example 11). The properties of the particles obtained according to the claimed process make them particularly suitable for being incorporated into polyolefin resin products. See page 2, lines 6 to 10 and page 3, line 58 to page 4, line 5. This has never been contested by the appellants.
- 5.3 Non-obviousness of the solution
- 5.3.1 Example 5 of D1 and the corresponding D2, respectively, show that the particle size distribution of the APP obtained is shifted to smaller particle sizes with an increased duration of the mixing, kneading and size reduction operation described. They do not, however, comprise any further hint concerning measures to be taken in order to obtain an even finer product having the required crystal type, solubility and viscosity, but not requiring strong grinding, and therefore comprised of crystals with smooth surfaces.
- 5.3.2 D3 discloses a different process for the preparation of APPs having very low solubilities by mixing and reacting equimolar amounts of ammonium phosphate and phosphorous pentoxide in the presence of ammonia at varying pressures and at relatively low temperatures of from 50°C to 150°C, followed by annealing the product

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obtained at higher temperatures in the presence of ammonia (see claim 1 and Example 1). According to D3, APPs having the desired properties can only be obtained by interrupting the ammonia addition during the low temperature phase of the reaction for a intermediate period of from 10 to 60 minutes, during which the material in the reactor is held under a nitrogen atmosphere (see page 6, line 18 to page 7, line 3). However, in contrast to the method according to the contested patent, D3 aims at preparing APPs with viscosity values as low as possible (page 3, lines 18 to 20), and in particular much lower than the ones according to D1 (see table on page 10). Moreover, D3 does not mention any melting of the raw materials and does not address the crystal types and the particle size distributions of the APPs obtained, or their incorporation into polyolefin resins.

Hence, the board holds that the skilled person cannot, without the knowledge of the invention, derive from D3 that "a first step of reacting the raw materials as a melt for 10 minutes or longer at a temperature of at least 250°C in a nitrogen atmosphere without feeding of ammonia gas" would be useful for obtaining type II APPs with all the properties i), ii) and iii) as referred to under 5.2.1 above.

5.3.3 D4 describes the continuous preparation of APPs with low solubilities by reacting equimolar amounts of ammonium phosphate and P₄O₁₀ (phosphorous anhydride) in the presence of ammonia gas (see e.g. Example 4). During the first stage of the reaction, i.e. before and after a first addition of ammonia gas (zones I and II in Figures 1 and 2), the temperature is not higher than approximately 200°C (see also column 3, lines 61 to 64). The mixture is then annealed at high temperatures under an ammonia atmosphere (zone III in Figures 1 and

- 2). The solubilities of the APPs obtained are stated to decrease with an increase of the temperatures of from 200°C to 340°C used in the subsequent annealing zone (see column 3, line 64 to column 4, line 12). According to Examples 2 and 4, crushing and grinding is required in order to obtain fine particles. D4 is silent about an initial melting of the starting materials and does not mention the crystal types and particle size distributions of the products obtained and their incorporation into polyolefin resins.
- D5 undisputedly belongs to the prior art according to 5.3.4 Article 54(2) EPC, since the patent in suit cannot validly claim the first of the two priority dates. D5 is directed to a process for the preparation of powdery APPs of low solubility, which are suitable for being incorporated into thermoplasts (see page 2, lines 7 to 21 and lines 44 to 45). The method comprises mixing and reacting equimolar amounts (see e.g. Example 3) of ammonium phosphate and phosphorous pentoxide in the presence of ammonia at temperatures of from 100°C to 300°C, preferably 150°C to 280°C, followed by annealing at high temperature under an ammonia atmosphere (see claim 1). However, it appears from the examples of D5 that the mixture of the starting materials is filled in a reactor heated-up to 150°C only (see also claim 7), and that higher reaction temperatures of 240°C or more are only reached upon feeding of and reaction with the ammonia. It is to be noted that, in contrast to the method according to the contested patent, D5 aims at preparing APPs having lower viscosity values than the ones according to D2 (see page 2, lines 7 to 21). Apart from the qualifier "powdery", the sole explicit mention of particle sizes is to be found in Example 3, which mentions average particle sizes of 50 to 200 µm obtained by milling. Moreover, D5 is silent about an initial melting of the starting materials and does not

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explicitly mention the crystal types and particle size distributions of the products obtained. The crystal type of the APPs obtained is only indicated by means of the spectrum represented in Figure 1. The respondent has repeatedly asserted that the spectrum shown in Figure 1 of D5 does not correspond to the type II spectrum (see letter dated 9 September 1997, page 6, second paragraph and letter dated 8 April 1999, page 7, first paragraph). Since this remained undisputed, the board accepts that the method of D5 does not lead to type II APPs.

- D6 relates to a method for preparing type II APPs for 5.3.5 use in - inter alia - thermoplasts (page 5, lines 1 to 4). The method comprises mixing and reacting equimolar amounts (see Examples 1, 2, 3, 7 and 8 and claim 1) of ammonium phosphate and phosphorous pentoxide in the presence of excess ammonia. In a first step, the reaction is carried out at a relatively low temperature of from 50 to 150°C, followed by annealing at a higher temperature. The method of D6 requires the presence of a melamine compound in the initial reaction mixture (see claim 1) and does not disclose the melting of the raw materials. Moreover, D6 is silent about the particle sizes of the APPs obtained. Their solubilities are lower than the ones of the APPs obtainable according to D1, but the viscosity values are drastically reduced in comparison to the ones of the products according to D1 (see page 4, lines 5 to 31).
- 5.3.6 D7 relates to the preparation of substantially water-insoluble APPs of four different crystalline forms labelled "Form 1" to "Form 4". The respondent has accepted that "Form 2", as obtained according to Examples III, IV, VI, VII and VIII can be equated to type II (see letter dated 9 September 1997, page 10, second paragraph). The "Form 2" APPs have low solubility and high viscosity values which make them

suitable for incorporation into plastics (see column 10, lines 16 to 28). D7 generally mentions ammonium phosphates, phosphorous pentoxide and ammonia (see column 2, lines 43 to 49 and column 3, line 58 to column 4, line 10) as possible reagents for obtaining APPs. In order to obtain the desired type of APP, seeding crystals of the same type need to be added to the reaction mixture. However, none of the processes specifically disclosed in the examples of D7 refers to a reaction of ammonium phosphates and anhydrous phosphorous pentoxide with ammonia or an ammoniareleasing compound, let alone to an initial reaction step at a temperature above 250°C. Moreover, D7 is silent about the particle size distributions obtained.

5.3.7 It emanates from the above analysis of D4 to D7 that none of these documents addresses the desire to obtain a particle size distribution and crystal smoothness as referred to in claim 1. Moreover, the processes disclosed do not disclose the use of the claimed reactants (document D7), the use of an initial reaction step at a temperature of 250°C or more (documents D4 to D6), the preparation of type II APP (documents D4 and D5), and/or the preparation of APPs having low solubility and relatively high viscosity values (documents D4, D5 and D6). It is thus arguable whether the skilled person would consider these documents at all when trying to solve the stated technical problem. Assuming for the sake of argument that he/she would, there is nothing in these documents that suggests without knowledge of the invention - that the incorporation of the claimed "first step of reacting the raw materials as a melt for 10 minutes or longer at a temperature of at least 250°C in a nitrogen atmosphere without feeding of ammonia gas" into a

process for manufacturing APPs would be a conceivable measure contributing to obtaining type II APPs with all the properties (i), (ii) and (iii) as referred under 5.2.1 above.

- 5.3.8 D8 merely illustrates the grinder used according to Examples 1 to 6 and 10, and comparative Examples 1, 2 and 11 to 15 of the patent to separate the aggregates obtained into single crystals. Since D8 does not relate to the preparation of APPs, it cannot suggest any particular modification of the known processes for the preparation of APPs.
- 5.3.9 D9 refers to low viscosity, low solubility APP particles of relatively fine particle size ("max.2% > 24 µm"). Their crystal type is not specified, and the incorporation thereof into polyolefin resins is not addressed. No information is given in D9 concerning the method used for the preparation of this product.

 Therefore the skilled person could not derive from this document that the method as claimed would lead to the desired APPs.
- 5.3.10 Since the cited documents, taken alone or in combination, do not suggest the claimed process, the presence of an inventive step is acknowledged.
- 5.4 Whether or not Examples 2 and 3 of the contested patent, operating under process conditions differing from the ones claimed, actually lead to the desired APPs is an issue which cannot reverse the above finding. Their teaching does not belong to the prior art and differs from the method disclosed in D1 in that they do not refer to a substantial feeding of ammonia gas during the initial melting step.

- 5.5 The board is convinced that the (withdrawn) comparative experiments carried out by appellant 01 are not accurate reproductions of Example 1 of D1 and of the examples of the contested patent. The conclusions drawn from these experimental results by appellant 01 were thus disregarded.
- 6. Description to be adapted

The board notes that due to the amendments carried out in the claims, the description is not in line with the latter. It may also be arguable whether examples referring to process conditions different from the ones according to claim 1 should be clearly identified as "not according to the invention".

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the first instance with the order to maintain the patent with the following documents:
 - claims 1 and 2 as submitted during oral proceedings,
 - a description to be adapted,
 - Figure 1 as granted.

The Registrar:

The Chairman:

U. Bultmann

R Spangerberg

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