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Datasheet for the decision of 1 April 2009

Case Number: T 0673/06 - 3.3.01

Application Number: 99910871.5

Publication Number: 1062203

IPC: C07D 201/16

Language of the proceedings: EN

Title of invention:
Process for the continuous purification of crude epsilon-caprolactam

Patentee:
DSM IP Assets B.V., et al

Opponent:
BASF SE

Headword:
Purification of caprolactam/DSM

Relevant legal provisions:
EPC Art. 113(1)

Keyword:
"Amending claims during oral proceedings in the absence of the duly summoned opponent - admissible"
"Novelty (yes)"
"Inventive step (yes) - closest prior art leads away from the solution"

Decisions cited:
G 0004/92, T 0771/92, T 0133/92

Catchword:
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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0673/06 **-** 3.3.01

D E C I S I O N
of the Technical Board of Appeal 3.3.01
of 1 April 2009

Appellant: (Opponent)
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Decision under appeal:
Interlocutory decision of the Opposition Division of the European Patent Office posted 1 March 2006 concerning maintenance of European patent No. 1062203 in amended form.

Composition of the Board:

Chairman: P. Ranguis
Members: C. M. Radke
R. Menapace

Summary of Facts and Submissions

I. The opponent (Appellant) and the patent proprietors appealed against the interlocutory decision of the opposition division posted on 01 March 2006 that the European patent no. 1 062 203 amended during oral proceedings according to the auxiliary request meets the requirements of the EPC. The patent proprietors (Respondents) withdrew their appeal during the oral proceedings before the Board.

II. The opposition was based on grounds under Article 100 (a) EPC; the opponent contested that the subject-matter of the claims was novel and involved an inventive step.

III. The following documents were *inter alia* cited during the opposition proceedings:

(D1) EP-A-0 826 665
(D2) DE-A-1 944 910
(D4) EP-A-0 337 323.

IV. The opposition division found that the subject-matter of claim 1 of the main request was not novel in view of the disclosure of document (D1). The subject-matter of the claims of the auxiliary request was deemed to meet the requirements of the EPC, in particular it was novel in view of (D1), (D2) and (D4) and not obvious in view of the combined teachings of documents (D1) and (D2).

V. The auxiliary request comprised claims 1 to 6 submitted during the oral proceedings before the opposition division dated 15 February 2006, the only independent claim reading as follows:

"1. A process for the purification of crude ε-caprolactam, characterised in that crude ε-caprolactam prepared by cyclization of alkyl 6-aminocaproate, 6-aminocapronitrile, 6-aminocaproic acid, 6-aminocaproic amide and/or oligomers thereof, is subjected to a crystallization process comprising the following steps:

(1) liquid crude ε-caprolactam is fed into a crystallizer
(2) in the crystallizer conditions are set such that ε-caprolactam crystals and a mother liquid are formed
(3) a stream from the crystallizer is fed to a separator where the ε-caprolactam crystals are separated from the mother liquid
(4) the mother liquid is recycled,

and the liquid crude ε-caprolactam is obtained from a previous process step in a caprolactam synthesis process after removal of heavy and light compounds by distillation, wherein the light compounds include light organics having a lower boiling point than ε-caprolactam and the heavy compounds include ε-caprolactam cyclic oligomers, having a higher boiling point than ε-caprolactam."

VI. During appeal proceedings, ***inter alia*** the following document was additionally cited:

(D5) US-A-5 496 941.

VII. The Respondents argued that document (D1) did not disclose directly and unambiguously in a single embodiment a process in which the heavy and light

compounds are removed from crude ε-caprolactam by distillation and then the ε-caprolactam is crystallised.

The problem to be solved in view of document (D1) was to improve the purity of crude ε-caprolactam obtained by cyclisation of alkyl 6-aminocaproate, 6-aminocapronitrile, 6-aminocaproic acid, 6-aminocaproic amide and/or oligomers thereof, with respect to typical impurities, notably N- or C-substituted lactams and/or amides. The Respondents filed experimental evidence to show that N- or C-substituted lactams were also formed when 6-aminocapronitrile was cyclised. Example III and comparative tests A to C of the patent in suit showed, so they argued, that this problem was solved. There was no suggestion in document (D1) that the combination of features of present claim 1 would yield such pure ε-caprolactam.

VIII. The Appellant considered the subject-matter of claim 1 not to be novel in view of any of the documents (D1), (D2) and (D4).

Moreover, it deemed the subject-matter of claim 1 not to be based on an inventive step in view of

- the disclosure of document (D1) if combined with that of document (D4) (the latter disclosing the trivial features (1) to (4) of present claim 1):
- the disclosure of document (D2) if combined with that of document (D4); or
- the combinations of the teachings of documents (D1), (D2) and (D4);

where the teaching of document (D4) was not limited to the treatment of ε-caprolactam obtained by Beckmann rearrangement.

In its view the problem of removing N- and C-substituted lactams did not occur for the cyclisation of 6-aminocapronitrile which gave rise to other side products, as was disclosed in document (D5). It considered comparative test C of the patent in suit not to be relevant as the crude caprolactam of example I was used and not one made by cyclisation of 6-aminocapronitrile.

IX. The parties were duly summoned to the oral proceedings before the Board. The Appellant was absent at the oral proceedings as announced in its letter dated 27 March 2009. The proceedings were thus continued in the absence of the Appellant in accordance with Rule 115(2) EPC.

X. The Appellant requested in writing that the decision under appeal be set aside and the patent be revoked.

The Respondents requested that the patent be maintained on the basis of claims 1 to 6 of the Auxiliary Request submitted during the oral proceedings before the opposition division dated 15 February 2006 (see point V above). During the oral proceedings before the Board, the Respondents withdrew their request to maintain the patent on the basis of the main request rejected in the decision under appeal.

XI. At the end of the oral proceedings the decision of the Board was announced.

Reasons for the Decision

1. The appeal is admissible.

2. *Article 113(1) EPC*

In accordance with the consistent jurisprudence of the boards of appeal, a decision may be based on claims amended during oral proceedings held in the absence of the duly summoned appealing opponent. This is not in conflict with decision G 04/92 (OJ EPO 1994, 149), because the appellant had reasonably to expect that the respondent would try to overcome the objections raised by amending the claims (see the decisions T 0771/92 of 19 July 1995, point 7 of the reasons, and T 0133/92 of 18 October 1994, point 7 of the reasons).

This applies in particular to the present case as the amended claims were held to be allowable in the decision under appeal and thus formed the most obvious fall-back position of the Respondents. Moreover, the Appellant submitted his observations regarding these claims in the statement setting out the grounds for appeal (see, e.g., pages 1 and 2 of Appellant's letter dated 11 July 2006 where the features of present claim 1 are listed).

Hence the present decision which is based on these amended claims complies with Article 113(1) EPC.

3. *Article 123 EPC*

Neither was an objection under Article 123 EPC raised against the present claims (see the fourth paragraph of page 2 of the minutes of the oral proceedings before the opposition division), nor was the opposition based on grounds under Article 100(c) EPC (see under point II above). Present claim 1 is based on claims 1, 2 and 7 and on page 7, lines 15-20 of the application as originally filed. Claims 2 to 6 have their basis in original claims 3-6 and 8, respectively.

The extent of protection of the claims as granted was restricted by the additional mandatory features disclosed in claims 2 and 7 and on page 7, lines 15-20 of the application as originally filed.

4. *Novelty*

4.1 Document (D1)

4.1.1 This document discloses

(a) to prepare ε-caprolactam starting from 6-aminocapronitrile, 6-aminocaproic acid or mixtures of 6-amonocaproamide with 6-aminocaproic acid (see claims 9 and 10, and column 2, lines 29-38);

(b) to distil off the ammonia and any unconverted 6-aminocapronitrile, if present, and part of the water (see column 2, lines 39-51);

(c) to extract the aqueous solution, preferably with an alkyl phenol having a higher boiling point than ε-caprolactam (see claim 5 and column 3, lines 41-43);

(d) to recover the ε-caprolactam by distilling it off (see column 4, lines 32-36);

(e) to purify the distilled liquid ε-caprolactam, e.g. by crystallisation in a process of concentration (see column 5, lines 11-19);

(f) to recycle the mother liquid to the aqueous solution before the extraction with the organic solvent (see column 5, lines 15-18).

4.1.2 The present claims require that "... the liquid crude ε-caprolactam is obtained from a previous process step in a caprolactam synthesis process after removal of heavy ... compounds by distillation, wherein ... the heavy compounds include ε-caprolactam cyclic oligomers, having a higher boiling point than ε-caprolactam." (see claim 1).

4.1.3 It has, thus, to be examined whether or not document (D1) discloses a process meeting this requirement in combination with the other features of present claim 1.

In document (D1), the distillation step (b) described under point 4.1.1 above only removes the low boiling components of the reaction mixture and thus does not separate ε-caprolactam from any higher boiling component. The distillation step (d) is designed to separate ε-caprolactam from any compound having a higher boiling point, such as the alkyl phenol extracting agent. Therefore, it will also separate ε-caprolactam from its cyclic oligomers, if present. However, said distillation step (d) is preceded by the extraction step (c) which removes the cyclic oligomers from the reaction mixture (see (D1), column 1, lines 31-33). The examples of document (D1) show that this

extraction is so efficient that no detectable amount of oligomer is found in the organic phase (see, e. g., column 6, lines 40-41). This means that no detectable amount of cyclic oligomers is present in the extract to be distilled in step (d). Hence, document (D1) does not teach to remove the cyclic oligomers having a higher boiling point than ε-caprolactam by distillation, contrary to the requirements of present claim 1.

4.2 Document (D2)

According to the decision under appeal, document (D2) does not teach step (4) according to present claim 1, namely that the mother liquid separated from the ε-caprolactam crystals is recycled (see the third sentence under point 4.3 of the reasons; compare point V above). Neither did the Appellant argue that document (D2) disclosed such a step, nor is the Board aware of such a disclosure in this document (see pages 3 to 6 of Appellant's letter dated 11 July 2006). Hence, this document does not disclose the subject-matter of present claim 1.

4.3 Document (D4)

Present claim 1 requires that the crude ε-caprolactam to be purified was "prepared by cyclization of alkyl 6-aminocaproate, 6-aminocapronitrile, 6-aminocaproic acid, 6-aminocaproic amide and/or oligomers thereof". In contrast to this, document (D4) does not disclose directly and unambiguously crude ε-caprolactam prepared by such a process; it only mentions the preparation of crude ε-caprolactam by Beckmann rearrangement of cyclohexanone oxime (see page 2, lines 41-47).

The crude ε-caprolactam prepared by Beckmann rearrangement of cyclohexanone oxime differs from the one to be purified in the process according to present claim 1 in that it does not contain cyclic oligomers (see document (D1), column 1, lines 36-43). Hence, document (D4) does not disclose a process according to present claim 1 which requires that ε-caprolactam is separated from its cyclic oligomers by distillation.

4.4 For these reasons, none of the documents (D1), (D2) and (D4) discloses the subject-matter of present claim 1; nor is the Board aware of any other document which does. Therefore, the subject-matter of claim 1 and that of dependent claims 2-6 is considered to be novel.

5. *Inventive step*

5.1 The closest prior art

The closest state of the art is normally a prior art document disclosing subject-matter with the same objectives as the claimed invention and having the most relevant technical features in common.

As the patent in suit, document (D1) is directed to the purification of crude ε-caprolactam prepared by cyclisation of 6-aminocapronitrile, 6-aminocaproic acid and/or 6-aminocaproamide (see (D1), column 1, lines 25-28 and claims 9 and 10; see paragraph [0006] of the patent in suit). Whereas document (D1) discloses to recycle the mother liquid separated from the ε-caprolactam crystals as required in present claim 1, document (D2) does not (see step (f) under point 4.1.1

and point 4.2 above). Hence, document (D1) has more features in common with the subject-matter of the present claims, and, as a consequence, represents the closest prior art.

5.2 The problem to be solved

According to the patent in suit, the problem to be solved was "... to provide a purification process for crude ε-caprolactam prepared by cyclization of alkyl 6-aminocaproate, 6-aminocapronitrile, 6-amino caproic acid, 6-aminocaproic amide and/or oligomers thereof." (see paragraph [0006]).

As such a purification process is already disclosed in document (D1), the objective problem to be solved may be considered as to provide a further effective purification process for the type of crude ε-caprolactam mentioned above.

A comparison between example I (now comparative) and example III of the patent in suit shows that this problem is solved.

Whether or not a more ambitious problem is solved, such as the removal of N-substituted or C-substituted lactams, need not be discussed as it has no effect on the outcome of the present decision (see the second paragraph under point VII and the third paragraph under point VIII above).

5.3 Document (D1) teaches to separate ε-caprolactam from its oligomers by extraction. This extraction step is to replace the distillation of the prior art process which

requires high reboiler temperatures, thus converting more ε-caprolactam to its oligomers which in turn may foul the process equipment as they tend to solidify (see column 1, lines 6-24). In the light of this teaching, the person skilled in the art looking for a further effective purification process for crude ε-caprolactam would **avoid** to distill off the caprolactam from its oligomers, even if such a distillation step is disclosed in document (D2) or (D4). Hence, the person skilled in the art would not envisage a process as defined in present claim 1.

5.4 It follows, that the subject-matter claim 1 and that of dependent claims 2-6 involves an inventive step.

Order

For these reasons it is decided that:

The appeal of the opponent is dismissed.

The Registrar: The Chairman:

M. Schalow P. Ranguis