

Internal distribution code:

- (A) [] Publication in OJ
(B) [] To Chairmen and Members
(C) [X] To Chairmen
(D) [] No distribution

D E C I S I O N
of 18 January 2001

Case Number: T 0684/98 - 3.3.1

Application Number: 89106200.2

Publication Number: 0337323

IPC: C07D 201/16

Language of the proceedings: EN

Title of invention:

Method for producing high-purity caprolactam

Patentee:

MITSUBISHI CHEMICAL CORPORATION

Opponent:

Allied Signal Inc.

Headword:

High-purity caprolactam/MITSUBISHI

Relevant legal provisions:

EPC Art. 54, 56, 100(b), 123(2) and (3)

Keyword:

"Insufficiency of disclosure (no) - exceptional failure"
"Novelty (yes) - claimed subject-matter not directly and
unambiguously derivable from prior art"
"Inventive step (no) - obvious choice of process parameters"

Decisions cited:

T 0020/81, T 0435/91

Catchword:

-



Case Number: T 0684/98 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 18 January 2001

Appellant: MITSUBISHI CHEMICAL CORPORATION
(Proprietor of the patent) 5-2, Marunouchi 2-chome
Chiyoda-ku
Tokyo (JP)

Representative: Wächtershäuser, Günter, Prof. Dr.
Patentanwalt
Tal 29
D-80331 München (DE)

Respondent: Allied Signal Inc.
(Opponent) Columbia Road and Park Avenue
P O Box 2245R
Morristown
New Jersey 07962-2245 (US)

Representative: Lawrence, Peter Robin Broughton
Gill Jennings & Every
Boardgate House
7 Eldon Street
London EC2M 7LH (GB)

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 11 May 1998
revoking European patent No. 0 337 323 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: A. J. Nuss
Members: R. Freimuth
S. C. Perryman

Summary of Facts and Submissions

- I. The Appellant (Proprietor of the Patent) lodged an appeal on 6 July 1998 against the decision of the Opposition Division posted on 11 May 1998 revoking European patent No. 337 323 and filed on 11 September 1998 a written statement setting out the grounds of appeal.
- II. Notice of Opposition had been filed by the Respondent (Opponent), requesting revocation of the patent in its entirety for lack of novelty and inventive step (Article 100(a) EPC) and for lack of sufficient disclosure (Article 100(b) EPC). The following documents were submitted *inter alia* in opposition proceedings:
- (3) US-A-2 813 858
 - (4) DE-A-1 022 591
 - (6) Chemical Engineering, 22 February 1982, pages 91 and 92, and Figures 1 to 3 annexed thereto.
- III. The decision under appeal was based on two alternative single claims, i.e. as main request on the claim as amended during opposition proceedings and as auxiliary request on the claim as granted.

The Opposition Division decided that the amendment made to the claim according to the then pending main request lacked original disclosure, thus, contravening Article 123(2) EPC. While the subject-matter claimed according to the then pending auxiliary request was novel, it did not involve an inventive step.

The Opposition Division held that the fresh upper limit of the water concentration of 2% in the claim of the main request represented an undue generalisation of the examples. The continuous process claimed according to the auxiliary request was novel over document (3) which disclosed a batch process and did not comprise any explicit disclosure of a continuous process. Starting from document (3) as closest state of the art in the assessment of inventive step the problem underlying the patent in suit was considered to be the provision of a process for the preparation of caprolactam having a decreased water content. That document directed the person skilled in the art to consider a water concentration in the melt, a temperature and a pressure falling within the scope of the then pending claim. Furthermore, going from a batch to a continuous process was conventional in the art, in particular in view of document (4) already describing a continuous process.

IV. At the oral proceedings before the Board, held on 18 January 2001, the Appellant defended the maintenance of the patent in suit in amended form on the basis of a single claim submitted during those oral proceedings superseding any previously submitted request. That claim read as follows:

"1. A method for continuously producing high-purity caprolactam from a mixture comprising a melt of crude caprolactam, which comprises supplying and cooling the mixture in a crystallizer under a reduced pressure by means of latent heat of evaporation, so that the cooling surface is not the wall surface of the crystallizer, but is the liquid surface constituting the mixture, while maintaining the water concentration in the mixture at a predetermined level, to crystallize

high-purity caprolactam, and then separating the resulting crystals, characterized in that the mixture is continuously supplied, the water concentration in the mixture is maintained at a level of from 1 to 8 %, the reduced pressure is at a level of from 6.6 to 26.6 mbar (5 to 20 Torr), the temperature for crystallization is within a range of from 30 to 65 °C and the crystal is large in size."

- V. The Appellant argued with respect to novelty in particular that the claimed process was novel over document (3) since that prior art disclosed a discontinuous, not a continuous process. The numerical indication of specific amounts of caprolactam to be purified revealed the batchwise operation of the process of that state of the art.

Having regard to inventive step, the Appellant submitted that document (4), not document (3), represented the closest state of the art since the former referred to a continuous process, the latter, however, to a batch process. The claimed process provided high purity caprolactam with large crystal size, decreased water content and avoided sticking of the crystals to surfaces coming in contact therewith. The solution to these problems proposed by the patent in suit was neither disclosed nor suggested by the state of the art cited in the proceedings, in particular documents (3) and (4). Moreover, it was not to be expected that the water content in the crystals varied with the water content in the starting solution, hence rendering the claimed subject-matter inventive. Furthermore, the crystals obtained by the claimed process contained water occluded in the crystals as shown in the fresh document

(8) Journal of Crystal Growth, Vol. 177, pages 119 to 124 (1997).

In respect of the insufficiency of disclosure objected to by the Respondent for the reason that the claim embraced areas that could not operate, the Appellant argued that according to the continuous process of the present invention the cooling was carried out by reduced pressure in a non-equilibrium state. Therefore the Respondent's theory based on an equilibrium state of a batch process was not applicable in the present case. Furthermore the onus of proof for non-operability of the claimed process rested on the Respondent.

VI. The Respondent argued having regard to novelty that document (3) disclosed a continuous process since this was the only sensible way of conducting the multistage cyclic process specified by the flow diagram and of interpreting the indication to commercial scale installations.

In respect of inventive step, document (3) represented the closest prior art since it was the closest as regards its specific teaching and as regards its objectives, namely optimizing crystal size and purity and reducing the amount of entrained liquor. The evaporation cooling avoided sticking of the crystals at contact surfaces. The water content in the crystals prepared by the claimed process was roughly proportional to the amount of water present in the system during processing which was suggested to be small following the teaching of the table at column 4 of document (3).

With respect to the insufficiency of the disclosure,

the Respondent argued that the claim embraced process conditions that could not operate successfully.

Furthermore, the patent in suit gave no guidance on how to choose the water concentration, the temperature and the pressure in order to arrive at crystals being large in size.

VII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the claim submitted at the oral proceedings on 18 January 2001.

The Respondent requested that the appeal be dismissed.

VIII. At the end of the oral proceedings the decision of the Board was given orally.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments (Article 123 EPC)*

In the claim the fresh feature of cooling "by means of latent heat of evaporation, so that the cooling surface is not the wall surface of the crystallizer, but is the liquid surface constituting the mixture" finds support on page 4, lines 18, 19 and 22 to 24 of the application as filed. Therefore the amendment made to the claim as granted complies with the requirements of Article 123(2) EPC.

That amendment of the claim as granted brings about a restriction of the scope of that claim, and therefore

of the protection conferred thereby, which is in keeping with the requirements of Article 123(3) EPC.

3. *Insufficiency of disclosure of the invention*
(Article 100(b) EPC)

3.1 The Respondent argued that the claim embraced process conditions that could not operate successfully. Thus, the claim covered a water concentration down to the lower limit of 1% and a pressure up to the upper limit of 26.6 mbar (20 Torr). However, according to Figure 3 of document (6), which is a phase diagram of the vapour pressure of saturated lactam solutions *versus* the weight percentage of water therein, the claimed process was not feasible when operating at a water concentration of 1% and a pressure of 20 Torr. The Appellant challenged the validity of that diagram in the oral proceedings before the Board and indicated that the Respondent's objection referred to the borderline of the present invention which did not remove the feasibility of the whole process claimed.

The phase diagram of Figure 3 may be interpreted indeed to indicate that the process of the patent in suit is inoperational exclusively at the particular water concentration of 1% and the particular pressure of about 15 to 20 Torr. Even assuming the validity thereof for the present case, the Respondent's objection is not convincing. It is established jurisprudence of the Boards of Appeal that the disclosure of an invention is sufficient if the skilled person can reasonably expect that substantially all embodiments of the claimed invention can be put into practice. Exceptional failures can be tolerated (see decision T 435/91, OJ EPO 1995, 188, point 2.2.3 of the reasons). In the present

case, the Respondent did not challenge that substantially all embodiments of the claimed invention could be carried out by the skilled person. The inability to operate the claimed process at the particular combination of a water concentration of 1% and a pressure of 20 Torr objected to by the Respondent is in fact an isolated failure just at the combined limits of respective ranges claimed which, hence, does not impair the sufficiency of the disclosure of the present invention.

- 3.2 Furthermore the Respondent objected that the patent in suit gave no guidance on how to choose the three parameters water concentration, temperature and pressure in the claimed process in order to arrive at crystals being large in size.

However, the claimed invention specifies for each of those three parameters a particular range wherein the water concentration, the pressure and the temperature is to be selected. Moreover the patent in suit comprises several examples giving the skilled person detailed guidance on how to operate the invention. Additionally, the common general knowledge found *inter alia* in the phase diagram of Figure 3 in document (6) addressed by the Respondent gives the skilled person a clear indication about the interrelationship of pressure and water concentration. Furthermore, a particular selection of the values for those three parameters is a matter of routine trial and error experiments for the skilled person thereby arriving at successfully reproducing the claimed invention. The Respondent neither submitted nor provided any evidence that the skilled person would encounter serious difficulties when doing so, let alone that an undue

burden was associated therewith. For these reasons, the Respondent's argument cannot convince the Board.

3.3 Consequently, the Respondent's challenge of the sufficiency of the disclosure of the patent in suit under Article 100(b) EPC is rejected.

4. *Novelty*

4.1 The Respondent challenged the novelty of the claimed invention exclusively with regard to document (3), not relying on any further document cited so far in the proceedings. Therefore, the Board limits its detailed considerations with respect to novelty to that document.

4.2 Document (3) is directed to a process for purifying caprolactam which comprises partially freezing molten caprolactam by evaporating water therefrom (claims 1 and 5). That process is exemplified in example 4 at column 8, lines 62 and following. At column 8, line 63, column 9, lines 50 and 75 and column 10, lines 7 and 19 it specifies numerically the quantity in parts by weight of caprolactam to be purified in that process, hence indicating the discontinuous operation of that process. The claimed process, however, is operated continuously.

4.3 While conceding the above finding, the Respondent argued that document (3) disclosed also the continuous operation of that process since this was the only sensible way of conducting the multistage cyclic process specified by the flow diagram and of interpreting the indication to commercial scale installations.

The flow diagram referred to is silent about whether to operate the process continuously or discontinuously; from a technical point of view it fits both operating possibilities. However, that flow diagram is explained in more detail at column 3, lines 6 to 9 indicating that it reflects a preferred multistage cyclic method described in detail in example 4. That example 4, however, discloses a discontinuous operation of the process as set out in point 4.2 above in detail. The term "cyclic" has the sole technical meaning of recycling product(s) in that process whether operated continuously or discontinuously. The reference to commercial scale installations at column 2, line 59 does not give any information about how to operate the process since both discontinuous and continuous operations are conventional in the art on a commercial scale.

According to established jurisprudence of the Boards of Appeal a document does not disclose a specific technical feature if it does not, for the skilled person, emerge clearly and unambiguously from that document. Applying that principle in the present case results in the conclusion that document (3) does not disclose clearly and unambiguously the continuous operation of the purification process with the consequence that it is not detrimental to the novelty of the process claimed.

- 4.4 To summarize, in the Board's judgement, document (3) does not anticipate the claimed invention. Therefore the Board concludes that the subject-matter of the claim is novel within the meaning of Articles 52(1) and 54 EPC.

5. *Inventive step*

5.1 According to the established jurisprudence of the Boards of Appeal it is necessary, in order to assess inventive step, to establish the closest state of the art, to determine in the light thereof the technical problem which the invention addresses and successfully solves, and to examine the obviousness of the claimed solution to this problem in view of the state of the art. This "problem-solution approach" ensures assessing inventive step on an objective basis and avoids an *ex post facto* analysis.

5.2 The patent in suit is directed to a method for continuously producing high-purity caprolactam from a melt of crude caprolactam which comprises cooling under reduced pressure by means of latent heat of evaporation. In relation to that particular process, a selection among the documents cited in the proceedings must be made as to which one is to be considered as the closest prior art. The Appellant and the Respondent concurred that this selection was to be made among either document (4) or document (3), since those documents referred to a purification process for caprolactam. However, the parties had divergent views on the matter which of those documents should be treated as the closest prior art.

While document (4) is directed to a continuous process, as is the patent in suit, document (3) refers to a batch, i.e. discontinuous, process. Since continuous and batch processes are two different types of operation requiring engineering distinct from one another, the discontinuous process described in document (3) is further away from the claimed invention

than the continuous process of document (4).

Thus, the Board considers, in agreement with the Appellant, that in the present case document (4) represents the closest state of the art and, hence, takes it as the starting point when assessing inventive step.

5.3 Document (4) describes a continuous process (column 3, line 68) for the purification of a melt of crude aqueous caprolactam by cooling, which may be achieved directly or indirectly. The direct cooling is achieved by means of latent heat of evaporation by applying a vacuum to the crude caprolactam to evaporate water contained therein and to dissipate the heat of crystallisation (column 3, lines 10 to 13; claim 2). A reduced pressure of 18 Torr for that vacuum is exemplified (column 4, line 37). The temperature of below 50°C is suitable for the process (column 3, line 18). The lactam concentration is preferably between 75 to 95%, which means *vice versa* a water concentration of about 5 to 25%. A water concentration of 7,5% is exemplified (Column 4, line 10). That process results in caprolactams of high purity, of large crystal size and of low water content (column 3, lines 22 to 26 and 53 to 60; example 3, column 4, line 48).

5.4 In view of this closest state of the art, the Appellant submitted at the oral proceedings before the Board that the problem underlying the patent in suit consists in providing a purification process resulting in a caprolactam of high purity having a large crystal size, in lowering the water content thereof and in avoiding the sticking of the caprolactam crystals on surfaces

coming in contact therewith.

The Respondent never disputed that the claimed process successfully achieves a caprolactam of high purity and of large crystal size, and avoids the sticking thereof on surfaces coming in contact therewith; and the Board is not aware of any reason for challenging this finding. However, the Appellant and the Respondent were divided on the matter whether or not the purported improvement of decreasing the water content in the resulting caprolactam crystals is successfully achieved by the claimed process vis-a-vis document (4). To this end the Appellant compared the water content of the caprolactams prepared in examples 1 to 3 of the patent in suit on the one hand with that of the caprolactam prepared in example 3 of document (4) on the other, in both cases the water content resulting after centrifugation. However, the way how the centrifugation step is operated is decisive for the aqueous mother liquor retained on the surface of and in between the crystals and, thus, has a strong impact on the water content thereof. Due to the complete lack of information in the patent in suit as well as in document (4) about the operation characteristics of the centrifugation step, the comparison made by the Appellant is unfair and cannot support the alleged fact that an improved, i.e. lowered, water content is achieved by the claimed invention.

According to the jurisprudence of the Boards of Appeal, alleged but unsupported advantages cannot be taken into consideration in respect of the determination of the problem underlying the claimed invention (see e.g. decision T 20/81, OJ EPO 1982, 217, point 3, last paragraph of the reasons). Since in the present case

the alleged advantage, i.e. lowering the water content, lacks the required adequate support, the technical problem as defined above needs reformulation.

Thus, the objective problem underlying the patent in suit can only be seen in providing a purification process resulting in a caprolactam of high purity having a large crystal size and **low** water content and in avoiding the sticking of the caprolactam crystals on surfaces coming in contact therewith.

5.5 As the solution to this problem, the patent in suit proposes a continuous process for purifying crude caprolactam which is characterised in that the water concentration is maintained at a level of 1 to 8% and the cooling is achieved under a reduced pressure of 5 to 20 Torr by means of latent heat of evaporation at a temperature of 30 to 65°C.

5.6 Finally, it remains to decide whether or not the proposed solution to the objective problem underlying the patent in suit is obvious in view of the state of the art.

5.6.1 The closest prior art document (4) used as starting point describes a purification process offering both direct and indirect cooling. Document (3) referring also to a process for purifying crude caprolactam addresses the aspect of the problem underlying the patent in suit of avoiding the sticking of the caprolactam crystals on surfaces coming in contact therewith (column 5, lines 65 to 68). As the solution to this problem that document teaches at column 5, line 65 to apply "evaporative freezing methods", i.e. the direct cooling of document (4) by means of latent

heat of evaporation. Thus, document (3) gives a clear incentive to choose the direct cooling method described in the closest prior art document (4) in order to avoid the sticking of the caprolactam crystals on surfaces coming in contact therewith which is the solution proposed by the claimed process.

5.6.2 With respect to the aspect of the problem underlying the patent in suit of providing a purification process resulting in a caprolactam having low water content, the Appellant and the Respondent concurred on the matter that the person skilled in the art is well aware of the fact that the water content consists of two components, namely the aqueous mother liquor retained on the surface of and in between the crystals, and that occluded within the crystals. The Appellant, referring to document (8), and the Respondent, however, were divided on the matter which of the two components had the most impact on the overall water content of the resulting caprolactam. However, a decision on this point is unnecessary since it is irrelevant in the present case as shown below.

Document (3) gives a hint on how to keep low the water content which results from the aqueous mother liquor occluded in the purified caprolactam crystals. At column 4, lines 35, 42 and 43 it teaches that the "DF/DM ratio" should be preferably 4 or above in order to "avoid solvent occlusion". According to the table at that column 4 of document (3) the "DF/DM ratio" is reciprocally proportional to the water concentration in the melt of the crude caprolactam, e.g. the "DF/DM ratio" of 5.3 or 5.75 corresponds to a water concentration of 4.8 or 2.4%, respectively, thus hinting at maintaining a low water concentration

therein. Having regard to the water content of the purified caprolactam crystals resulting from the aqueous mother liquor retained on the surface of and in between the crystals, the person skilled in the art is well aware that it is proportional to the amount of water present in the system during processing, i.e. that a low water concentration in the melt of the crude caprolactam necessarily results in a low water content of the caprolactam crystals, thus, also hinting at maintaining a low water concentration.

Thus, to keep low the water content of the purified caprolactam crystals, document (3) gives a clear incentive to choose a low water concentration of the melt of crude caprolactam within the range described in the closest prior art document (4) which is generally at least 5%, 7,5% being exemplified, which is within the water concentration range of 1 to 8% specified in the claimed process, i.e. the solution suggested by the patent in suit.

5.6.3 The problem underlying the patent in suit of providing a purification process resulting in a caprolactam of high purity having a large crystal size has already been solved by the process described in document (4) operating generally at a temperature of below 50°C which lies within the claimed range of 30 to 65°C and at a reduced pressure of e.g. 18 Torr, which is within the claimed range of 5 to 20 Torr. Thus, these features cannot provide the claimed process with any inventive ingenuity, which was not disputed by the Appellant.

5.6.4 The Board concludes from the above that the state of the art, in particular documents (4) and (3), gives the person skilled in the art concrete incentives on how to

solve the objective problem underlying the patent in suit as defined in the above point 5.4, last paragraph, namely by maintaining the water concentration at a level such as now claimed and by cooling by means of latent heat of evaporation at a temperature and a reduced pressure at values encompassed by the claimed ranges, thus arriving at the process of the claimed invention without involving any inventive activity.

5.7 Therefore, in the Board's judgement, the subject-matter of the claim represents an obvious solution to the problem underlying the patent in suit.

6. As a result, the Appellant's request is not allowable as the subject-matter of the claim lacks inventive step pursuant to Article 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

N. Maslin

A. Nuss