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Datasheet for the decision of 28 May 2020

Case Number: T 1717/15 - 3.3.02

Application Number: 03000846.0

Publication Number: 1329448

IPC: C07D201/04, C07D201/06

Language of the proceedings: EN

Title of invention:
Procsess for producing laurolactam from cyclododecanone

Patent Proprietor:
Ube Industries, Ltd.

Opponent:
Evonik Operations GmbH

Headword:

Relevant legal provisions:
EPC Art. 83

Keyword:
Sufficiency of disclosure - (yes)

Decisions cited:
G 0009/91, G 0003/14

Catchword:

Europäisches Patentamt
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Beschwerdekammern

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Case Number: T 1717/15 - 3.3.02

D E C I S I O N
of Technical Board of Appeal 3.3.02
of 28 May 2020

Appellant: (Patent Proprietor)	Ube Industries, Ltd. 1978-96, O-Aza Kogushi Ube-shi, Yamaguchi-ken 755-8633 (JP)
Representative:	Hoeger, Stellrecht & Partner Patentanwälte mbB Uhlandstrasse 14c 70182 Stuttgart (DE)
Respondent: (Opponent)	Evonik Operations GmbH Rellinghauserstrasse 1-11 45128 Essen (DE)
Representative:	Evonik Degussa GmbH CI-IPM-PAT Paul-Baumann-Strasse 1 45764 Marl (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 2 July 2015 revoking European patent No. 1329448 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	M. O. Müller
Members:	M. Maremonti
	R. Romandini

Summary of Facts and Submissions

I. The appeal by the patentee ("appellant") lies from the decision of the opposition division to revoke European patent No. 1 329 448 ("the patent").

II. The patent as granted contains four claims, independent claim 1 of which reads as follows:

"*1. A process for producing laurolactam from cyclododecanone, comprising reacting a cyclododecacone* [sic!]*-containing starting material with a hydroxylamine salt of a mineral acid to prepare cyclododecanoneoxime, and converting the resultant cyclododecanoneoxime to laurolactam through a Beckmann rearrangement reaction, wherein*

(1) the cyclododecanone-containing starting material further contains, as an impurity, at least one member selected from oxygen atom-containing organic compounds having 12 carbon atoms and cycloaliphatic unsaturated hydrocarbon compounds having 12 carbon atoms;
(2) before the step of producing the cyclododecanoneoxime, the cyclododecanone-containing starting material, is pre-treated with an aqueous solution of an alkali metal hydroxide, toluenesulfonic acid or γ-alumina at a temperature of 70 to 230°C; and
(3) the resultant pre-treated reaction mixture is subjected to a precision distillation,

to thereby control a content of each of the oxygen atom-containing organic compounds having 12 carbon atoms and the cycloaliphatic unsaturated hydrocarbon compounds having 12 carbon atoms contained, as an impurity, in the cyclododecanone-containing starting material to 300 ppm or less."

Claims 2 to 4 define specific embodiments of the process set out in claim 1.

III. The opposition division came to, *inter alia*, the following conclusion on the then pending main request (patent as granted) and first auxiliary request:

- The subject-matter of claim 1 as granted and claim 1 according to the first auxiliary request was not disclosed in the patent in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art.

IV. In its statement setting out the grounds of appeal, the appellant contested the reasoning of the opposition division and submitted, *inter alia*, that the claimed subject-matter was sufficiently disclosed. It also re-filed the three auxiliary requests already filed before the opposition division.

V. In its reply to the appeal, the opponent raised objections under Article 100(b)/83 EPC against, *inter alia*, the main request and the first auxiliary request.

For its argumentation concerning insufficiency of disclosure, the opponent especially relied on experiments 10 to 12 and figure 1 as filed on 7 April 2015 before the opposition division.

By letter dated 29 May 2019, the opponent withdrew its opposition against the patent. Therefore, it is no longer a party to these appeal proceedings.

VI. The appellant was summoned to oral proceedings in accordance with its request.

In preparation for the oral proceedings, the board issued a communication in which it expressed its

preliminary opinion that the claimed subject-matter did not appear to be sufficiently disclosed in the patent.

VII. In its response dated 21 April 2020, the appellant maintained that the claimed subject-matter was sufficiently disclosed and corroborated its argumentation by filing new experimental results and a figure 1 showing the chemical reactions involving the impurities contained in the starting cyclododecanone-containing material. It also filed new second and third auxiliary requests.

VIII. In a telephone conversation dated 25 May 2020, the appellant was informed of the board's preliminary opinion that the subject-matter of the first auxiliary request appeared to meet the requirements of Articles 83, 84 and 123(2) and (3) EPC.

IX. By letter dated 26 May 2020, the appellant withdrew its previous main request to maintain the patent as granted and filed a new main request (see below).

On the same day, the board issued a communication informing the appellant that the oral proceedings had been cancelled.

X. The appellant's requests

As its main request, the appellant requests that the board affirm that claims 1 to 4 of the first auxiliary request as filed on 12 November 2015 comply with the provisions of Articles 83, 84 and 123 EPC, and remit the case to the opposition division for the assessment of inventive step.

Alternatively, the appellant maintained its second and third auxiliary requests as filed by letter dated 21 April 2020.

XI. The arguments of the appellant, where relevant to the present decision, may be summarised as follows.

- Figure 1 included in the former opponent's submission of 7 April 2015 displayed the vapour pressure vs. temperature curves of cyclododecanone ("CDON") and the possibly present impurity cyclododecenone ("CDENON"). It could be seen that the vapour pressure curves for CDON and CDENON were very similar. Due to this small volatility difference, distillation separation of CDENON from CDON appeared not to be possible.

- However, the present invention overcame the problem of low volatility differences between CDON and CDENON by the pretreatment defined in step (2) of the claimed process.

- In step (2), CDENON was converted to a high boiling point product ("HBP"). This HBP was the addition product resulting from a Michael addition of a CDON molecule to a CDENON molecule. The HBP produced could then easily be separated from CDON in the distillation step (3) of the claimed process.

- This Michael addition reaction was shown in figure 1 of the submission dated 21 April 2020. It would have belonged to the general knowledge of a skilled person in the field of organic chemistry who would have understood this reaction to occur in step (2) of the claimed process.

- Since this reaction was reversible, the skilled person would also have recognised that inappropriate reaction conditions in step (2) would lead to the reverse reaction, resulting in an unsuccessful removal of the target impurity.

- In experiment 10 of the former opponent (submission of 7 April 2015), the pretreatment step (2) had not been appropriately performed since the content of CDENON only slightly decreased from 0.39% to 0.34%.

- This meant that the Michael addition did not progress sufficiently. In particular, the adopted operating conditions were mild (150°C for 6 hours), which resulted in scarce progress of the reaction.

- In contrast, example 1 of the patent demonstrated that CDENON could be reduced by the claimed process to a content below 300 ppm in accordance with the requirement of claim 1.

- This result was confirmed by the additional examples 1 and 2 filed on 21 April 2020. Example 1 included a pretreatment with sodium hydroxide according to step (2), performed at 200°C for 3 hours, and a distillation according to step (3) of the claimed process. Example 2 was comparative since pretreatment step (2) was not included.

- The obtained results demonstrated that by selecting appropriate operating conditions for step (2), e.g. a higher temperature (200°C vs. 150°C used by the former opponent), CDENON was not detectable after pretreatment and distillation. This meant that under the adopted conditions, the Michael addition reaction successfully progressed to form the HBP product, which was then removed by distillation.

- In example 2, not including the pretreatment step (2), CDENON and other impurities could not be removed.

- Therefore, these examples confirmed that the content of each impurity of the CDON-containing starting material, especially CDENON, might be controlled to 300 ppm or less as required by claim 1. The subject-matter of claim 1 was thus sufficiently disclosed in the patent.

- A remittal of the case to the opposition division for assessment of inventive step of the claimed subject-matter was thus justified.

Reasons for the Decision

Main request - sufficiency of disclosure under Article 83 EPC

1. Claim 1 of the main request recites as follows. The amendments to claim 1 as granted (II *supra*) have been highlighted by the board.

1. A process for producing laurolactam from cyclododecanone, comprising reacting a cyclododecacone [sic!]-containing starting material [...] to prepare cyclododecanoneoxime, and converting the resultant cyclododecanoneoxime to laurolactam [...], wherein

(1) the cyclododecanone-containing starting material further contains, as an impurity, [...] *having 12 carbon atoms;*
(2) before the step of producing the cyclododecanoneoxime, the cyclododecanone-containing starting material, is pre-treated with an aqueous solution of an alkali metal hydroxide~~, toluenesulfonic acid or γ-alumina~~ at a temperature of 70 to 230°C; and
(3) the resultant pre-treated reaction mixture is subjected to a precision distillation,

to thereby control [...] *to 300 ppm or less.*"

Therefore, the pretreatment step (2) has been limited to treatment with an aqueous solution of an alkali metal hydroxide.

In the following, cyclododecanone is referred to as "CDON"; cyclododecenone, which may be the impurity referred to in point (1) of claim 1, referred to as "CDENON".

2. According to Article 83 EPC, the European patent application (European patent) shall disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art.

2.1 The opposition division (impugned decision, reasons, 3 and 4) came to the conclusion that the process defined in claim 1 above (corresponding to claim 1 of the first auxiliary request underlying the impugned decision) was not workable over the whole claimed scope, contrary to the requirements of Article 83 EPC. This conclusion (reasons 3.3 of the appealed decision) was based on experiment 10 included in the former opponent's submission of 7 April 2015 (hereinafter "experiment 10"). This experiment had shown that by carrying out a process under operating conditions falling under those of claim 1 at issue, the content of various impurities, *inter alia*, CDENON, could not be controlled to 300 ppm or less as required by claim 1.

2.2 Experiment 10 was submitted by the former opponent. It was carried out on a mixture containing 98.35 wt-% CDON and various additional substances meeting the definition of "*impurities*" given in claim 1 at issue, namely "*oxygen atom-containing organic compounds having 12 carbon atoms and cycloaliphatic unsaturated hydrocarbon compounds having 12 carbon atoms*". The

tested mixture contained 0.39% CDENON, i.e. the same impurity as present in example 1 of the patent (paragraph [0074]).

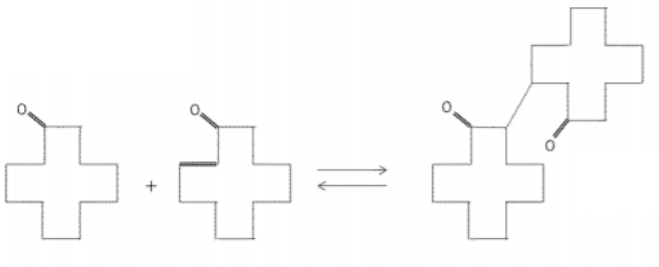
2.3 The board acknowledges that the results of experiment 10 show that by subjecting this mixture to a pretreatment with an aqueous solution of sodium hydroxide at 150°C for 6 hours, the concentration of CDENON is only reduced to 0.34%. Moreover, CDENON cannot be reduced to 300 ppm or less in a subsequent distillation step in line with the fact that CDON and CDENON have substantially identical boiling points (figure 1 of the former opponent's submission dated 7 April 2015, displaying the vapour pressure vs. temperature curves of CDON and CDENON), thus rendering separation by distillation highly problematic as acknowledged in the patent itself (paragraph [0004]).

2.4 However, example 1 of the patent (paragraph [0074]) reports that by subjecting a CDON-containing material comprising 700 ppm CDENON to a pretreatment with an aqueous solution of sodium hydroxide at 200°C for 5 hours and a subsequent distillation, the content of CDENON is reduced to 80 ppm, thus meeting the requirement of claim 1 at issue.

2.5 This result has been confirmed by example 1 filed by the appellant during these appeal proceedings (letter dated 21 April 2020, VII *supra*). Here (example 1, table 2 under "*NaOH Treatment*") a CDON-containing material also comprising CDENON was subjected to a pretreatment with an aqueous solution of sodium hydroxide at 200°C for 3 hours. No CDENON could be detected after this treatment step. Nor was any CDENON detectable after the subsequent distillation step (continuation of table 2 under "*Precision Distillation*").

2.6 Thus, the amount of the impurity CDENON is at least reduced (all experiments discussed above including experiment 10), and under certain pretreatment conditions it is reduced to a level as low as required by claim 1 by a pretreatment with sodium hydroxide (example 1 of the patent and example 1 filed by the appellant by letter dated 21 April 2020).

2.7 This conclusion is further supported by mechanistic considerations. More specifically, as submitted by the appellant (XI *supra*), the following Michael addition reaction between CDON and CDENON takes place in the presence of sodium hydroxide under appropriate temperature conditions, leading to the elimination of CDENON and the formation of a high boiling point(HBP) compound:



CDON CDENON HBP compound

This HBP compound can then be easily separated by the subsequent distillation step as defined in claim 1 at issue, thus overcoming the problem of the substantially identical boiling points of CDON and CDENON.

2.8 The board acknowledges that a claimed invention, here the claimed process for producing laurolactam from CDON, has to be sufficiently disclosed within the meaning of Article 83 EPC over the entire claimed scope and that the operating conditions of experiment 10 in terms of temperature of the pretreatment step (2) fall under the conditions required by claim 1 at issue. More specifically, the pretreatment with sodium hydroxide in

experiment 10 was carried out at 150°C for 6 hours, while claim 1 requires a temperature of 70 to 230°C and does not limit the pretreatment time. Nevertheless, *inter alia*, the CDENON content could not be reduced to 300 ppm or less as required by claim 1.

However, as set out above, the pretreatment in experiment 10 at **150°C** for 6 hours led at least to a reduction of the CDENON amount. It is reasonable to assume that the skilled person - wanting to even further reduce the CDENON amount before the subsequent distillation step - would have known, on the basis of common general knowledge, that this result could be achieved by e.g. increasing the temperature of the pretreatment. This can also be derived from the patent, where in example 1 a pretreatment at **200°C** for 5 hours led to a reduction of the CDENON level as required by claim 1. Hence, the skilled person carrying out the experiment submitted by the opponent and realising that the amount of CDENON is not as required by claim 1 would have found the appropriate operating conditions, especially of the pretreatment step (2), on the basis of information provided in the patent and common general knowledge.

2.9 The board is aware that claim 1 at issue is not limited to CDENON as the impurity in the CDON-containing starting material. However, the problem of the similarity of the boiling points (2.3 *supra*) was only raised by the former opponent with regard to CDENON. The board has no reason to doubt that the skilled person would have been able to control the content of other impurities to 300 ppm or less as required by claim 1 at issue by finding appropriate operating conditions for the pretreatment step (2) and the distillation step (3) of claim 1, based on the information provided in the patent and common general

knowledge. This is further confirmed by example 3 of the patent, reporting the elimination of the impurity cycloundecanecarboxyaldehyde by a process according to claim 1 at issue.

2.10 The same reasoning applies to the remaining claims 2 to 4, all dependent on claim 1, for which no specific objections as to sufficiency of disclosure are mentioned in the decision under appeal.

3. For the reasons set out above, the board concludes that the subject-matter of the main request meets the requirements of Article 83 EPC.

Main request - compliance with Articles 84, 123(2) and (3) EPC

4. Claim 1 of the main request (1 *supra*) only differs from claim 1 as granted (II *supra*) in that two out of three alternatives for the pretreatment step (2) have been deleted. This deletion does not introduce any lack of clarity within the meaning of Article 84 EPC. The remaining subject-matter as defined in the claims of the main request is identical to the subject-matter of the claims as granted and thus it is not open to clarity objections (decision G 3/14, OJ 2015, page 102, order).

5. As mentioned above, claim 1 of the main request (1 *supra*) only differs from claim 1 as granted (II *supra*) in that two out of three alternatives for the pretreatment step (2) have been deleted. This deletion restricts the claimed subject-matter so that the requirements of Article 123(3) EPC are met.

6. The remaining subject-matter as defined in the claims of the main request is identical to the subject-matter of the claims as granted. The ground for opposition under Article 100(c) EPC was not invoked by the former

opponent, nor was it introduced by the opposition division. Therefore, the board has no power to examine the compliance of the claimed subject-matter with Article 123(2) EPC (decision G 9/91, OJ 1993, 408, reasons 10 and 11 and order).

Remittal under Article 111(1) EPC

7. The decision under appeal has only dealt with the issue of sufficiency of disclosure within the meaning of Article 100(b) EPC in combination with Article 83 EPC. Since the appellant's main request has been found to meet the requirements of Article 83 EPC, the board considers it appropriate to make use of its discretion under Article 111(1) EPC and remit the case to the opposition division for the assessment of inventive step, not dealt with in the impugned decision, the presence of which had been contested by the former opponent under Article 100(a) EPC.

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 for the assessment of inventive step of the subject-matter of the claims according to the first auxiliary request filed by the appellant on 12 November 2015.

The Registrar: The Chairman:

N. Maslin M. O. Müller

Decision electronically authenticated