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DECISION
of 5 November 2003

Case Number: T 0540/01 - 3.3.1

Application Number: 93402815.0

Publication Number: 0616992

IPC: C07C 29/38

Language of the proceedings: EN

Title of invention:

Process for the preparation of 1,3-butylene glycol

Patentee:

Daicel Chemical Industries, Ltd.

Opponent:

Celanese International Corporation

Headword:

Butyleneglycol/DAICEL

Relevant legal provisions:

EPC Art. 56, 114(2), 123(2) and (3)

Keyword:

"Documents late filed (no) - filed with grounds of appeal"
"Amendments (allowable)"
"Inventive step (yes) - closest prior art as acknowledged in
the patent specification - fair comparison - technical effect
not addressed in prior art"

Decisions cited:

-

Catchword:

-



Case Number: T 0540/01 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 5 November 2003

Appellant I: Daicel Chemical Industries, Ltd.
(Proprietor of the patent) 1, Teppo-Cho
Sakai-shi
Osaka-fu (JP)

Representative: Portal, Gérard
Cabinet Beau de Loménie
158, rue de l'Université
F-75340 Paris Cédex 07 (FR)

Appellant II: Celanese International Corporation
(Opponent) 1601 West LBJ Freeway
Dallas
Texas 75234 (US)

Representative: James, Anthony Christopher W.P.
Carpmaels & Ransford
43 Bloomsbury Square
London WC1A 2RA (GB)

Decision under appeal: Interlocutory decision of the Opposition
Division of the European Patent Office posted
16 March 2001 concerning maintenance of
European patent No. 0616992 in amended form.

Composition of the Board:

Chairman: A. J. Nuss
Members: R. Freimuth
S. U. Hoffmann

Summary of Facts and Submissions

I. The Appellant I (Proprietor of the patent) and the Appellant II (Opponent) lodged appeals against the interlocutory decision of the Opposition Division posted on 16 March 2001 which found that European patent No. 616 992 in the form as amended according to the then pending main and first auxiliary request did not satisfy the requirements of the EPC, but that it could be maintained in the form as amended according to the second auxiliary request.

II. Notice of Opposition had been filed by Appellant II requesting revocation of the patent as granted in its entirety for lack of novelty and of inventive step based on the documents:

(1) GB-A-853 266 and

(2) Encyclopaedia of Chemical Processing and Design, Vol. 1, pages 116, 117 and 153 to 157 (1976).

The Opposition Division held that the claims as amended according to the then pending main request were not in keeping with the provisions of Article 123(2) EPC and that the amended set of claims according to the then pending first auxiliary request did not satisfy Rule 57a EPC since it comprised fresh dependent claims which were not a direct result of the grounds for opposition. The claims according to the second auxiliary request were found to comply with Articles 54, 84 and 123 EPC which was not disputed by the Appellant II. They also involved inventive step since certain specific requirements of the distillation

(lines 1-4-5 and 1-4-6 and decanter 1-4-4) and the low level of crotonaldehyde in the acetaldehyde of less than 0.1% could not have been deduced from the prior art.

III. At the oral proceedings before the Board held on 5 November 2003 the Appellant I defended the maintenance of the patent in suit in amended form on the basis of a single "main request" consisting of a sole claim which read as follows:

"1. A process for the preparation of 1,3-butylene glycol by steps (a), (b) and (c):

(a) aldol condensation step of acetaldehyde in the presence of an alkali catalyst to obtain a crude reaction solution primarily containing aldoxane, acetaldehyde, water and small amounts of crotonaldehyde;

(b) thermal decomposition step of aldoxane contained in the crude reaction solution, in an aldoxane decomposition column (1-3) to obtain paraldol while distilling off a distillate containing a mixture of acetaldehyde, water and small amounts of crotonaldehyde from the crude reaction solution; and performing a distillation of said mixture to recover acetaldehyde on the one hand, and crotonaldehyde on the other hand; and

(c) hydrogenation step of paraldol to obtain 1,3-butylene glycol in the presence of a catalyst;

characterised in that said process further comprises:

(d) performing said distillation in an acetaldehyde-refining column (1-4) including a reboiler (1-4-1), a condenser (1-4-2), and a condenser (1-4-3) and a decanter (1-4-4) between a discharging line (1-4-5) and a recirculating line (1-4-6), wherein a distillate discharged from the discharging line (1-4-5) is condensed in the condenser (1-4-3), and then a resulting condensate is separated into two layers of liquid in the decanter (1-4-4); crotonaldehyde is primarily included in the upper layer of liquid [stream (E)] of the two layers of liquid, followed by supplying to a recovery step or a waste line, and resulting in being removed from acetaldehyde to be recirculated to the aldol condensation step (a); and the lower layer of liquid [stream (D)] of the two layers of liquid primarily includes from 5 to 15 % by weight of crotonaldehyde, and from 85 to 95 % by weight of water, both of which are recirculated to the side portion of the acetaldehyde-refining column (1-4) through a recirculating line (1-4-6) in order to effectively recover crotonaldehyde; and

(e) recirculating refined acetaldehyde having a content of less than 0.1% crotonaldehyde based on acetaldehyde to step (a)."

IV. The Appellant I submitted that the sole amended claim was in keeping with the requirements of Article 123(2) EPC as the amendments resulted from the original description, in particular pages 7 and 9, and the original drawings.

The process of document (1) did not comprise the decomposition of aldoxane and the hydrogenation of

paraldol and therefore disqualified as closest prior art. Starting, thus, the assessment of inventive step from the teaching of both documents cited on page 2, lines 41 and 42 of the patent specification as closest prior art, which described a process acknowledged in the precharacterising portion of present claim 1, the problem underlying the patent in suit was to prepare odorless 1,3-butylene glycol in higher yields. The examples and comparative examples comprised in the patent specification demonstrated that this aim of improving the yield has been successfully achieved by the claimed process. None of the documents cited so far in the proceedings addressed this problem and described the particular steps (d) and (e) according to claim 1 in order to solve it. Therefore the claimed process was not obvious.

The Appellant I objected to the admission in the proceedings of the Appellant II's documents (4) to (6) due to their late filing.

- V. The Appellant II submitted that document (1) described the process for preparing 1,3-butylene glycol specified in the precharacterising portion of claim 1, i.e. a process comprising the decomposition of aldoxane, the purification of acetaldehyde and the hydrogenation of paraldol, though using a terminology different to that used in patent in suit. Steps (a) to (d) of the claimed process were perfectly obvious vis-à-vis that closest prior document having regard to the skilled person's common general knowledge specified for example in the fresh document (4) to (6) which were cited in the Statement of the Grounds of Appeal:

- (4) Encyclopaedia of Chemical Processing and Design, Vol. 13, pages 231 to 238 (1981),
- (5) Ullmann's Encyclopaedia of Industrial Chemistry, Vol. 8, pages 83 to 90 (1987) and
- (6) H. Z. Kister, Distillation Operation, MacGraw-Hill 1989, pages 356 to 358, 498, 557, 558, 595 and 596.

The step (e) of the claimed invention specifying a content of less than 0.1% crotonaldehyde in the acetaldehyde was not a technical feature of the invention but merely a result to be obtained. There was nothing inventive in selecting a low target maximum for the content of crotonaldehyde in the recycled acetaldehyde.

The same conclusion in respect of obviousness arose when starting from the teaching of both documents cited in the patent specification as the closest prior art. The teaching of those documents as acknowledged in the patent specification was not disputed by the Appellant II. However, the purported aim of the patent in suit to provide odorless 1,3-butylene glycol in higher yields was not successfully solved, on the one hand, and solved in an obvious way, on the other. Thus, on the one hand, according to Tables 1 and 2 of the patent specification, the content of crotonaldehyde in stream (B) from the bottom of the aldoxane decomposition column was lower in comparative example 2 than in example 7 according to the invention and, on the other hand, document (1) already taught to purify the recirculated acetaldehyde from crotonaldehyde (page 2, lines 75 and following). The engineering of that

purification step was conventional in the art and described for example in documents (4) and (6). Thus, the objective problem underlying the patent in suit was less ambitious, namely to provide merely an alternative process for preparing 1,3-butylene glycol, and this problem was solved in an obvious way.

The fresh documents (4) to (6) were filed in due time, namely together with the Statement of the Grounds of Appeal.

VI. The Appellant II requested that the decision under appeal be set aside and that the patent be revoked.

The Appellant I requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request filed during the oral proceedings on 5 November 2003.

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

Reasons for the Decision

1. The appeals are admissible.
2. *Late filed evidence (Article 114(2) EPC)*

Documents (4) to (6) are new evidence cited for the first time in the Appellant II's Statement of the Grounds of Appeal. The Appellant I objected to admitting these documents into the proceedings for the reason that they were late filed while providing

detailed comments thereon in his letter of reply dated 8 February 2002.

Those documents were prompted by and intended to overcome the Opposition Division's essential argument set out in the decision under appeal that the specific requirements of the distillation, i.e. the lines (1-4-5) and (1-4-6) and the decanter (1-4-4), supported the inventive step of the claimed invention. Furthermore documents (4) to (6) are standard textbooks reflecting the common technical knowledge of the skilled person.

The submission by an Appellant of fresh documents in the Statement of the Grounds of Appeal to overturn the appealed decision is to be considered as a normal action of a losing party (see decision T 1072/98, point 2.3 of the reasons, not published in OJ EPO). Thus, in the present case, the fresh documents (4) to (6) submitted with the Appellant II's Statement of the Grounds of Appeal are not filed late in the sense of Article 114(2) EPC. This finding is underpinned by the fact that those documents address common general knowledge thereby causing no undue delay or burden to the appeal proceedings.

Therefore, documents (4) to (6) are to be taken into consideration in the appeal proceedings.

3. *Amendments (Article 123(2) and (3) EPC)*

The amendment to claim 1 as granted of decomposing aldoxane, contained in the crude reaction solution, in an aldoxane decomposition column (1-3) finds support on page 6, lines 27 and 28 and on page 7, lines 22 and 23

of the application as filed. Performing a distillation of the mixture of acetaldehyde, water and crotonaldehyde to recover separately acetaldehyde and crotonaldehyde is based on original Figure 1, streams (F) and (E). The specific installations to perform that distillation indicated in the characterising portion of claim 1 is found on page 7, lines 8 to 10 of the application as filed. The particular operation of the condenser (1-4-3) and the decanter (1-4-4) is supported by original page 9, lines 4 to 15. Thus, all the amendments made to claim 1 as granted comply with the requirements of Article 123(2) EPC.

Those amendments of claim 1 as granted bring 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.

4. *Novelty*

The novelty of the patent in suit was not at issue in this appeal. Although raised as a ground for opposition by him, the Appellant II concurred in appeal proceedings with the finding of the Opposition Division rejecting this ground. Nor does the Board see any reason to take a different view. Hence, it is unnecessary to go into more detail in this respect.

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 Claim 1 of the patent in suit is directed to a process for preparing 1,3-butylene glycol starting with an aldol condensation of acetaldehyde to yield the intermediate aldoxane, decomposing that intermediate to paraldol which is then hydrogenated. The documents JP-A-212384/1987 and JP-A-246529/1987 which are cited and acknowledged in the specification of the patent in suit on page 2, lines 41 to 52 as the closest prior art, refer both to the same type of preparation process. Uncontested by the Appellant II, they describe a preparation process wherein aldoxane resulting from an aldol condensation is thermally decomposed yielding a crude reaction solution primarily consisting of paraldol while distilling off acetaldehyde, followed by catalytically reducing (with hydrogen) paraldol to prepare 1,3-butylene glycol. The unreacted acetaldehyde, together with crotonaldehyde generated in the thermal decomposition step, is recirculated to the aldol condensation.

5.3 Those documents, though having not been expressly addressed in the notice of opposition, nevertheless form part of the opposition appeal proceedings since any document indicated in a contested patent as closest prior art is automatically included therein (see decision T 536/88, OJ EPO 1992, 638, point 2.1 of the reasons). Where the patent in suit indicates a

particular piece of prior art as being closest to the claimed invention and the starting point for determining the problem underlying the patent in suit, then the Board should adopt this as the starting point for the purpose of a problem-solution analysis unless it turns out that there is closer state of the art of greater technical relevance (see e.g. decisions T 800/91, point 6 of the reasons; T 68/95, point 5.1 of the reasons).

Thus, the Board considers, in agreement with the Appellant I that in the present case the process for preparing 1,3-butylene glycol described in both documents specified above represents the closest state of the art and, hence, takes it as the starting point when assessing inventive step.

- 5.4 The Appellant II, while not disputing the above findings, addressed also document (1) as representing the closest prior art. That document is directed to a process for preparing 1,3-butylene glycol starting with an aldol condensation of acetaldehyde yielding acetaldol which is steam stripped to remove unreacted acetaldehyde, and then hydrogenating the stripped acetaldol. Therefore document (1) neither specifies that process to proceed via the intermediate aldoxane and, thus, to thermally decompose aldoxane in a decomposition column, nor to hydrogenate paral dol. For those reasons the process disclosed in document (1) is further away from the claimed invention than the documents addressed in point 5.2 above.

The Appellant II alleged that the process of document (1) necessarily and implicitly comprised the

unspecified steps and features, namely the formation of aldoxane, its thermal decomposition in a decomposition column and the hydrogenation of paral dol. Aldoxane was formed automatically and the steam stripping corresponded to a thermal decomposition of aldoxane yielding what was described as paral dol in the claimed invention. However, the Appellant II, when offering his interpretation of document (1), has merely speculated without providing substantiating facts or corroborating evidence. The burden of proving the facts it alleges lies with the party invoking these facts. If a party, whose arguments rest on these alleged facts, is unable to discharge its onus of proof, it loses thereby. In the absence of any pertinent evidence presented by him, the Appellant II has not discharged the burden of proof which is upon him, with the consequence that the Board cannot accept his view.

5.5 The drawbacks of the conventional process for preparing 1,3-butylene glycol according to the closest prior art (cf. point 5.2 above) lie in recirculating crotonaldehyde generated in the thermal decomposition step of aldoxane to the aldol condensation together with unreacted acetaldehyde, unpreferably resulting in the generation of various impure components by a reaction with acetaldehyde in the aldol condensation step (patent specification page 2, lines 49 to 52).

Thus, the technical problem underlying the claimed invention as indicated in the specification of the patent in suit on page 2, lines 4 to 6 and page 3, lines 19 to 20, and as submitted by the Appellant I at the oral proceedings before the Board, consists in providing an improved process for preparing 1,3-

butylene glycol in which the generation of by-products is decreased resulting in obtaining an odorless 1,3-butylene glycol at higher yields.

- 5.6 As the solution to this problem, the patent in suit proposes a process for preparing 1,3-butylene glycol as defined in claim 1 which is characterised by step (d), i.e. by performing a distillation of crude acetaldehyde in an acetaldehyde-refining column (1-4) including a reboiler (1-4-1), a condenser (1-4-2), and a condenser (1-4-3) and a decanter (1-4-4) between a discharging line (1-4-5) and a recirculating line (1-4-6), wherein a distillate discharged from the discharging line (1-4-5) is condensed in the condenser (1-4-3), and then a resulting condensate is separated into two layers of liquid in the decanter (1-4-4); crotonaldehyde is primarily included in the upper layer of liquid [stream (E)] of the two layers of liquid, followed by supplying to a recovery step or a waste line, and resulting in being removed from acetaldehyde to be recirculated to the aldol condensation step (a); and the lower layer of liquid [stream (D)] of the two layers of liquid primarily includes from 5 to 15 % by weight of crotonaldehyde, and from 85 to 95 % by weight of water, both of which are recirculated to the side portion of the acetaldehyde-refining column (1-4) through a recirculating line (1-4-6) in order to effectively recover crotonaldehyde, and which is further characterised by step (e), namely by recirculating the refined acetaldehyde having a content of less than 0.1% crotonaldehyde to the aldol condensation step.

5.7 The Appellant I and the Appellant II were divided on the matter of whether or not the evidence presented in the specification of the patent in suit convincingly demonstrates that the proposed solution successfully solves the problem underlying the invention of preparing odorless 1,3-butylene glycol at higher yields.

The Appellant I relied on the experimental report comprised in the specification of the patent in suit demonstrating that the yield of 1,3-butylene glycol in the process according to the invention, i.e. examples 1 and 2, was improved vis-à-vis the process according to the closest prior documents (cf. point 5.2 above), i.e. comparative examples 1 and 2. In examples 1 and 2 the yield of odorless 1,3-butylene glycol is 76,6% and 78%, respectively, while in comparative examples 1 and 2 the yield thereof is lower, namely 59,6% and 70%, respectively. That experimental report compares the yield of two processes for obtaining odorless 1,3-butylene glycol both differing from each other exclusively in the presence or absence of steps (d) and (e) as defined in claim 1. Therefore, the comparison of the experimental data for examples 1 and 2 and comparative examples 1 and 2 indicated in that test report truly reflects the impact of the process modifications distinguishing the solution suggested by the patent in suit from the closest prior documents. This specific comparison is, thus, a fair basis for the assessment of inventive step. For these reasons, the Board is satisfied that the problem underlying the patent in suit has been successfully solved.

The Appellant II addressed example 7 and comparative example 2 in order to challenge the successful solution of the problem underlying the invention. He submitted that the content of crotonaldehyde in the crude solution discharged from the bottom of the aldoxane decomposition column [stream (B)] was higher in example 7 according to the invention, namely 2,3%, than in comparative example 2 according to the prior art, namely 1,7%. However, the problem underlying the patent in suit consists in obtaining odorless 1,3-butylene glycol in higher yields (cf. point 5.5 above), not in reducing the content of crotonaldehyde in a particular intermediate process stream, e.g. stream (B). Furthermore the operation conditions in example 7 and comparative example 2 are substantially different, e.g. the reaction temperature / the retention time being 117°C / 13 min and 75°C / 86 min, respectively, thereby making any direct comparison between both examples void.

5.8 Finally, it remains to be decided whether or not the proposed solution to the problem underlying the patent in suit is obvious in view of the cited state of the art.

5.8.1 The closest prior art documents (see point 5.2 above) to start from teach a process wherein unreacted acetaldehyde is recirculated to the aldol condensation together with crotonaldehyde. They do not give any incentive to modify that process by steps (d) and (e) as defined in claim 1 and to increase thereby the yield. Thus, those documents, on their own, do not render obvious the solution proposed by the claimed invention

5.8.2 Though document (1) is directed to a process for preparing 1,3-butylene glycol and describes *inter alia* the distillation of a crude mixture of acetaldehyde and crotonaldehyde to recover acetaldehyde for further use (page 2, lines 75 to 81), that document does not address the technical problem underlying the patent in suit of obtaining odorless 1,3-butylene glycol in higher yields (see point 5.5 above). For this simple reason document (1) cannot give any hint on how to solve that technical problem since a skilled person would not take the teaching of that document into consideration when looking for a solution to the problem underlying the invention.

Furthermore, the skilled person would not in any event arrive at the process according to claim 1 when combining the teaching of the closest prior documents (cf. point 5.2 above) with that of document (1). Document (1) describes a mere distillation of the crude mixture of acetaldehyde and crotonaldehyde to recover acetaldehyde whereas the process of the invention requires in characterising step (d) *inter alia* the additional use of a decanter which is operated following particular process features, and in characterising step (e) a maximum content of 0.1% crotonaldehyde in the refined acetaldehyde. Thus, the skilled person when combining the teachings of the closest prior documents and of document (1) would thereby not arrive at the claimed process. Therefore, the Appellant II's obviousness objection based on document (1) cannot convince the Board.

5.8.3 The Appellant II also addressed documents (4) and (6) reflecting common general knowledge and describing standard features in the field of separation techniques thereby implying a less ambitious problem of the invention, namely to provide merely an alternative process for preparing 1,3-butylene glycol. However, the technical problem underlying the patent in suit is different and consists in the provision of a process for preparing odorless 1,3-butylene glycol in higher yields (see point 5.5 above). Both documents do not address this technical problem and for that very reason they cannot give any hint on how to solve it; therefore a skilled person would not take the teaching of those documents into consideration when looking for a solution to the problem underlying the invention.

Furthermore, the combination of the teaching of documents (4) or (6) with that of the closest prior documents (cf. point 5.2 above) would not result in the process as defined in claim 1. Document (4) describes a series of stills for refining crotonaldehyde whereas the process of the invention requires in step (d) the additional use of a particularly operated decanter and in step (e) a maximum content of 0.1% crotonaldehyde in the refined acetaldehyde. Document (6) is directed to distillation operations in general describing *inter alia* a removal of water from a side stream of a hydrocarbon or fusel oil distillation column by means of a decanter. However, that document is silent about any particular operation of the decanter as required in the process of claim 1, namely of separating in a decanter a specific side stream into two layers of liquid, the upper layer being crotonaldehyde and the lower layer primarily including from 5 to 15% by weight

of crotonaldehyde, and from 85 to 95% by weight of water, both of which being recirculated to the side portion of the acetaldehyde-refining column, and it is silent about a maximum content of 0.1% crotonaldehyde in the refined acetaldehyde.

Hence, the skilled person when combining the teaching of the closest prior documents with that of document (4) or (6) would thereby not arrive at the claimed process. Therefore, the Appellant II's obviousness objection based on those documents is devoid of merit.

5.8.4 To summarize, in the Board's judgment, none of the documents addressed above renders the claimed invention obvious, either taken alone or in combination.

The Appellant II not relying on further prior art in order to support his objection of obviousness, the Board is satisfied that none of the other documents in the proceedings renders the proposed solution obvious.

5.9 For these reasons the Board concludes that the subject-matter of the sole claim involves an inventive step within the meaning of Articles 52(1) and 56 EPC.

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 on the basis of the main request filed during oral proceedings of 5 November 2003 and a description yet to be adapted.

The Registrar:

The Chairman:

N. Maslin

A. Nuss