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D E C I S I O N
of 24 February 2005

Case Number: T 0482/01 - 3.3.1

Application Number: 95109122.2

Publication Number: 0687662

IPC: C07C 53/08

Language of the proceedings: EN

Title of invention:

Process for producing high purity acetic acid

Patentee:

DAICEL CHEMICAL INDUSTRIES, LTD.

Opponent:

BP Chemicals Ltd

Headword:

Acetic acid production/DAICEL

Relevant legal provisions:

EPC Art. 54, 56, 113(2), 114(2)

Keyword:

"Admissibility of late filed documents (no)"

"Admissibility of a late filed request (yes)"

"Novelty (yes)"

"Inventive step (yes) - non-obvious alternatives"

Decisions cited:

-

Catchword:

-



Case Number: T 0482/01 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 24 February 2005

Appellant:
(Opponent)

BP Chemicals Ltd
Britannic House,
1 Finsbury Circus
London EC2M 7BA (GB)

Representative:

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Respondent:
(Proprietor of the patent)

DAICEL CHEMICAL INDUSTRIES, LTD.
1, Teppo-cho
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Osaka (JP)

Representative:

Grünecker, Kinkeldey,
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Decision under appeal:

Decision of the Opposition Division of the
European Patent Office posted 9 March 2001
rejecting the opposition filed against European
patent No. 0687662 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: R. Freimuth
Members: J. M. Jonk
R. T. Menapace

Summary of Facts and Submissions

I. The Appellant (Opponent) lodged an appeal against the decision of the Opposition Division rejecting the opposition against the European patent No. 0 687 662 (European patent application No. 95 109 122.2), the claims reading as follows:

"1. A process for producing high purity acetic acid, comprising the steps of continuously reacting methanol with carbon monoxide in the presence of a rhodium catalyst, an iodide salt, and methyl iodide, wherein the reaction is carried out while maintaining an acetaldehyde concentration in the reaction liquid of 400 ppm or lower."

"2. The process of claim 1, wherein the acetaldehyde is removed from the process liquid being circulated into a reactor."

"3. The process of claim 1 or 2, comprising the steps of separating the reaction liquid into a volatile phase containing acetic acid, methyl acetate and methyl iodide and a low volatile phase containing the rhodium catalyst, distilling the volatile phase to obtain a product mixture containing acetic acid and the overhead containing methyl acetate and methyl iodide, and recirculating said overhead into the reactor, wherein the overhead or a condensate of said overhead is contacted with water to separate it into an organic phase containing methyl acetate and methyl iodide and an aqueous phase containing the carbonyl impurities containing acetaldehyde, and recirculating said organic phase into the reactor."

"4. The process of any of claims 1 to 3, wherein the overhead is distilled at a top temperature of 55°C or higher, at a reflux tank temperature of 25°C or higher, at a pressure of 98 kPa (1 kg/cm²) or more, to separate and remove acetaldehyde, before recirculating the purified overhead into the reactor".

"5. The process of any of claims 1 to 3, wherein the overhead is distilled at a top temperature of less than 55°C and a reflux tank temperature of less than 25°C in the presence of an alcohol".

"6. The process as described in claim 5, wherein methanol is introduced at a lower position than a stage charged with the overhead containing acetaldehyde and methyl iodide".

II. The opposition was filed against the patent as a whole, and based on the grounds of lack of novelty and lack of inventive step as indicated in Article 100(a) EPC. It was supported by several documents including:

- (1) EP-A-0 487 284,
- (2) US-A-4 102 922, and
- (3) Handbook of Chemistry and Physics, CRC Press Inc., 56th Edition (1975-1976), pages C-365 and C-405.

III. The Opposition Division held that the subject-matter of Claim 1 of the patent in suit was novel and involved an inventive step, since the cited prior art did not provide any incentive to control the acetaldehyde concentration in the reaction liquid at a value of 400 ppm or lower in order to achieve an acetic acid

product having a reduced amount of carbonyl and iodide impurities.

IV. Oral proceedings before the Board were held on 24 February 2005. The Appellant, who was duly summoned, did not attend the oral proceedings as announced by his facsimile dated 13 January 2005.

V. The Appellant based his objections against the patentability of the claimed subject-matter indicated in his Grounds of Appeal not only on the cited documents (1) to (3), but also on documents

(4) US-A-5 214 203, and

(5) US-A-5 001 259,

which had been acknowledged in the specification of the patent in suit as prior art.

He submitted in writing:

- (a) that the subject-matter of Claim 1 as granted lacked novelty in the light of documents (1) and (5),
- (b) that the subject-matter of Claim 1 as granted, if considered novel, lacked inventive step having regard to the teaching of document (1), since this document was related to the same technical problem, namely, to avoid an undesirable high concentration of aldehyde impurities in the acetic acid product, and because the solution of this problem as claimed in the patent in suit was a matter of mere

experimental routine in view of the reduction of such impurities in the methyl iodide recycle stream achieved as indicated in said document (1);

- (c) that the subject-matter of Claim 2 as granted, i.e. the removal of acetaldehyde from the process stream recirculated to the carbonylation reactor, had been disclosed in document (1);
- (d) that the subject-matter of Claim 3 as granted, i.e. the extraction of the overhead from the splitter column with water in order to separate acetaldehyde and other carbonyl impurities from the organic phase before its recirculation into the reactor, was obvious to the skilled person in the light of document (1), since according to this document said overhead phase was treated by an aqueous phase inevitably causing a separation of any unconverted acetaldehyde by passing into the aqueous phase being discharged;
- (e) that the subject-matter of Claim 4 as granted, i.e. the distillation of the overhead of the splitter column under the particular distillation conditions as specified in the claim in order to separate the carbonyl impurities before recirculating the overhead into the reactor, was obvious to the skilled person in the light of documents (1) and (2), since the removal of impurities from the splitter overhead was known from document (2), and because he would have understood in reading document (1) that it would be advantageous to distil the splitter overhead in which the carbonyl impurities were concentrated to

minimise their recirculation into the reactor and that the finding of suitable distillation conditions would be a matter of mere routine experimentation; and

- (f) that the subject-matter of Claim 5 as granted, i.e. the distillation of the splitter overhead under the lower temperature conditions as specified in the claim and in the presence of an alcohol, was obvious to the skilled person for the same reasons as indicated with respect to the subject-matter of Claim 4 as granted, and because it would be obvious to the skilled person that clogging in the distillation column due to the forming of metaldehyde and paraldehyde could be prevented by the introduction of an alcohol in view of the known solubility of metaldehyde and paraldehyde in alcohols as taught by document (3).

VI. During oral proceedings, the Respondent (Patentee) defended the patentability of the subject-matter of the patent in suit on the basis of a new set of Claims 1 and 2, independent Claim 1 resulting from combining the subject-matter of Claims 1 to 5 as granted reading as follows:

"A process for producing high purity acetic acid, comprising the steps of continuously reacting methanol with carbon monoxide in the presence of a rhodium catalyst, an iodide salt and methyl iodide, wherein the reaction is carried out while maintaining an acetaldehyde concentration in the reaction liquid of 400 ppm or lower and wherein the acetaldehyde is removed from the process liquid being circulated into

the reactor, said process comprising the steps of separating the reaction liquid into a volatile phase containing acetic acid, methyl acetate and methyl iodide and a low volatile phase containing the rhodium catalyst, distilling the volatile phase to obtain a product mixture containing acetic acid and the overhead containing methyl acetate and methyl iodide, and recirculating said overhead into the reactor,

wherein the overhead or a condensate of said overhead is contacted with water to separate it into an organic phase containing methyl acetate and methyl iodide and an aqueous phase containing the carbonyl impurities containing acetaldehyde, and recirculating said organic phase into the reactor, or

wherein the overhead is distilled at a top temperature of 55°C or higher, at a reflux tank temperature of 25°C or higher, at a pressure of 98 kPa (1 kg/cm²) or more, to separate and remove acetaldehyde, before recirculating the purified overhead into the reactor, or

wherein the overhead is distilled at a top temperature of less than 55°C and a reflux tank temperature of less than 25°C in the presence of an alcohol."

- VII. The Respondent argued that the late cited documents (4) and (5) should not be admitted into the appeal proceedings, since they did not represent the closest prior art and, in addition, were not relevant for assessing novelty and inventive step.

Furthermore, he defended the patentability of the process of present Claim 1 essentially arguing that the cited state of the art did not provide any incentive to the skilled person that high purity acetic acid having a reduced content of carbonyl and iodide impurities could be obtained by the process as claimed, wherein the reaction is carried out while maintaining an acetaldehyde concentration in the reaction liquid of 400 ppm or lower using one of the three alternative process steps for removing acetaldehyde and other carbonyl impurities from the splitter overhead as indicated in the claim before recirculating the overhead into the reactor.

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

The Respondent requested that the appeal be dismissed and that the patent be maintained on the basis of Claims 1 and 2 as submitted at the oral proceedings.

IX. At the conclusion of the oral proceedings the Board's decision was pronounced.

Reasons for the Decision

1. The appeal is admissible.
2. *Admissibility of late filed documents into the proceedings.*
 - 2.1 The Respondent submitted that the late cited documents (4) and (5) should not be admitted into the appeal proceedings, since they related to a different technical problem and, consequently, were not relevant in assessing inventive step.
 - 2.2 According to Article 114(2) EPC the EPO may indeed disregard facts or evidence which are not submitted in due time by the parties concerned. In this context, a considerable body of jurisprudence has been developed by the boards of appeal showing that the main criterion for deciding on the admissibility of late-filed documents is their relevance, i.e. their evidential weight in relation to other documents already in the proceedings. Accordingly, the boards of appeal normally consider that a document acknowledged in the European patent as the closest state of the art for the purpose of formulating the technical problem set out in the description forms part of the opposition appeal proceedings even if it has not been expressly cited within the opposition period. With respect to the relevant jurisprudence concerning the admissibility of late-filed documents, the Board refers to the Case Law of the Boards of Appeal of the EPO, 4th edition 2001, VI. F. 1 - 6, pages 324 to 335).

2.3 Documents (4) and (5), which were cited by the Appellant for the first time in his Grounds of Appeal, have been acknowledged in the description of the patent in suit as state of the art.

However, the documents are merely mentioned in the patent in suit as background art (see page 2, lines 8 to 25), whereas document (1) is considered therein as the closest prior art for the purpose of formulating the technical problem to be solved (see page 2, lines 38 to 46, and page 3, lines 7 and 8). Therefore, they do not automatically form part of the proceedings. Moreover, they are apparently less relevant than the cited document (1) already forming part of the proceedings.

Furthermore, in considering the disclosure of the documents, the Board concludes that, *prima facie*, there are no clear reasons derivable from the documents to suspect that they would prejudice the patentability of the subject-matter of the patent in suit. In fact, the Appellant based a novelty objection merely on document (5) indicating the production of acetic acid containing only 91 ppm propionic acid (Table III), but from this disclosure it cannot directly and unambiguously be concluded that the production was carried out while maintaining an aldehyde concentration in the reaction liquid of 400 ppm or lower.

2.4 Thus, in applying the criteria developed by the Boards of Appeal for deciding on the admissibility of late-filed documents as indicated above (point 2.2) the Board does not admit the documents (4) and (5) into the proceedings.

3. *Admissibility of the late-filed request*

3.1 The Respondent filed his present request consisting of a new Claim 1 and a dependent Claim 2 corresponding to the dependent Claim 6 as granted at a very late stage, namely during the oral proceedings before the Board and in the absence of the Appellant who did not attend these proceedings as previously announced. One of the issues to be decided is, therefore, whether or not admitting the new request into the proceedings violates the Appellant's right to be heard (Article 113(2) EPC).

Present Claim 1 was filed by the Respondent after a discussion with the Board of the objections raised by the Appellant concerning inventive step and in particular after hearing the Board's opinion on this issue.

Moreover, present Claim 1 comprises as amendments with respect to the subject-matter of Claim 1 as granted only those technical features specified in the dependent Claims 2 to 5 as granted, which have been discussed during the opposition proceedings and also have been dealt with by the Appellant in his Statement of the Grounds of Appeal. By such a restriction the Appellant could not be taken by surprise and a decision could be based on the claims of the present request without getting into conflict with what has been explained in the opinion G 4/92 (OJ EPO 1994, 149) in respect of Article 113(1) EPC.

3.2 Under these circumstances, the Board considers it appropriate to exercise its discretion to admit the present request into the proceedings.

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

4.1 Present Claim 1 results from combining Claims 1 to 5 as granted. Moreover, its subject-matter is also supported in that it is a combination of the subject-matter of Claims 1 to 5 of the patent application as filed.

Present Claim 2 corresponds to Claim 6 of both the patent in suit and the application as filed.

4.2 Therefore, the Board finds that the subject-matter of the present claims meets the requirements of Article 123(2) and (3) EPC.

5. *Novelty*

5.1 Document (5) not having been admitted into the proceedings, the only issue to be dealt with in respect of novelty is, whether the subject-matter of present Claim 1 lacks novelty in view of document (1).

5.2 Document (1) relates, like the patent in suit, to a process for producing pure acetic acid by continuously reacting methanol with carbon monoxide in the presence of a rhodium catalyst, an iodide salt, and methyl iodide, which is characterised by treating the methyl iodide recycle stream to the carbonylation reactor to reduce the amount of carbonyl impurities in the methyl iodide recycle stream and, consequently, to diminish the forming of tars in the reaction fluid which have a

detrimental effect on the catalyst activity (see page 3, lines 21 to 44).

The treatment of the methyl iodide recycle stream is carried out by:

- (a) contacting the recycle stream which contains the carbonyl impurities including aldehydes, such as acetaldehyde, crotonaldehyde and butyraldehyde, with an amine compound, preferably an aqueous hydroxylamine salt, and a base, so that the carbonyl impurities are reacted with hydroxylamine to form oximation products which are soluble in the aqueous phase (see page 5, lines 37 to 45, and page 6, lines 24 to 33),
- (b) directing the reaction products to a decanter for separating the organic phase from the aqueous phase containing unreacted hydroxylamine salt as well as most of the oximation products (see page 6, lines 34 to 41),
- (c) directing the separated organic phase containing methyl iodide-rich recycle, minor amounts of water as well as trace amounts of hydroxylamine compound, oximes and other impurities which do not separate with the aqueous phase withdrawn from the decanter, to a distillation tower for removal of these impurities from the recycle (see page 6, lines 41 to 44), and
- (d) distilling the organic phase in the presence of added water in order to obtain a purified methyl iodide recycle stream which can be recycled to the

carbonylation reactor and a bottom stream comprising the separated aqueous oximes as well as other impurities such as alkanes (see page 6, lines 44 to 56).

5.3 Thus, the subject-matter of present Claim 1 is novel in view of document (1), since this document does not directly and unambiguously disclose the maintenance of an aldehyde concentration in the reaction liquid of 400 ppm or lower, let alone a treatment of the methyl iodide recycle stream as indicated in the three alternative process steps of present Claim 1.

6. *Inventive step*

6.1 For deciding whether subject-matter claimed involves an inventive step, the Boards of Appeal consistently apply the problem and solution approach, which essentially consists in identifying the closest prior art, determining in the light thereof the technical problem which the claimed invention addresses and successfully solves, and examining whether or not the claimed solution to this problem is obvious for the skilled person in view of the state of the art.

6.2 The Board considers, in agreement with the parties to the proceedings, that the closest state of the art with respect to the claimed subject-matter of the patent in suit is document (1).

This document discloses, as indicated above under point 5.2, a process for producing pure acetic acid, in which the methyl iodide recycle stream to the carbonylation reactor is treated to reduce the amount

of carbonyl impurities therein and, consequently, the forming of tars in the reaction fluid which have detrimental effect on the catalyst activity.

6.3 Starting from the teaching of this closest state of the art, the Board considers, in agreement with the parties to the proceedings, that the technical problem underlying the patent in suit consists in providing a further process for preparing acetic acid, in which the concentration of carbonyl impurities in the methyl iodide recycle stream is reduced (see also page 2, lines 38 to 46, and page 2, line 58 to page 3, line 1, of the patent in suit).

6.4 According to Claim 1 of the patent in suit this technical problem is essentially solved by maintaining an aldehyde concentration in the reaction liquid of 400 ppm or lower using one of the three alternative treatments of the methyl iodide recycle stream to reduce the acetaldehyde concentration therein, namely:

- (a) the extraction of the overhead from the distillation column (splitter) with water to separate it into an organic phase containing methyl acetate and methyl iodide and an aqueous phase containing the carbonyl impurities containing acetaldehyde,
- (b) the distillation of said overhead at a top temperature of 55°C or higher, at a reflux tank temperature of 25°C or higher, at a pressure of 98 kPa (1 kg/cm²) or more, to separate and remove acetaldehyde, or

(c) the distillation of said overhead at a top temperature of less than 55°C and a reflux tank temperature of less than 25°C in the presence of an alcohol.

6.5 Furthermore, in view of the examples of the patent in suit, the Board is satisfied that the technical problem as defined above has been successfully solved within the whole area claimed. This has not been disputed by the Appellant.

6.6 The question now is whether the solution of the technical problem underlying the patent in suit proposed by the process of Claim 1 comprising the three alternative treatments of the methyl iodide recycle stream is obvious in view of the cited prior art.

6.7 In challenging the inventive step of the extraction treatment indicated under point 6.4 (a) above, the Appellant only contended that the extraction of the overhead of the splitter column with water in order to separate acetaldehyde and other carbonyl impurities from the organic phase before its recirculation into the reactor, was obvious to the skilled person in the light of document (1), since according to this document said overhead phase was treated by an aqueous phase inevitably causing a separation of any unconverted acetaldehyde by its passing into the aqueous phase being discharged.

However, document (1) does not disclose an extraction of acetaldehyde and other carbonyl impurities from the overhead of the splitter column, but instead a decantation step to separate an aqueous phase

containing oximation products, obtained by a previous conversion of the carbonyl impurities in the overhead with an amine compound, from an organic phase, and a subsequent distillation of the separated organic phase containing methyl iodide-rich recycle, minor amounts of water as well as trace amounts of hydroxylamine compound, oximes and other impurities which do not separate with the aqueous phase withdrawn from the decanter, in order to remove these impurities from the recycle stream (see point 5.2, paragraphs (a) to (d) above). Moreover, there is no indication in document (1) that the aqueous phase from the decanter would contain unconverted acetaldehyde as suggested by the Appellant.

Therefore, document (1) does not provide any pointer to the skilled person that the technical problem underlying the patent in suit as defined above could be solved by applying a simple extraction with water without a previous conversion of the carbonyl impurities to oximation products.

- 6.8 In challenging the inventive step with respect to both alternative distillation treatments of the overhead from the splitter column as indicated under point 6.4 (b) and (c) above, the Appellant submitted that such distillations were obvious to the skilled person in the light of documents (1), since in reading this document he would have understood that in order to reduce the concentration of carbonyl impurities in the recycle stream it would be advantageous to distil the splitter overhead in which the carbonyl impurities are concentrated, and that the finding of suitable distillation conditions would be a matter of mere routine experimentation. In this context, he emphasised

that the removal of impurities from the splitter overhead was already known from document (2).

However, this submission fails, since document (1) discloses that it was difficult to remove the minor amounts of carbonyl impurities by conventional means such as distillation, since the impurities have boiling points close to that of acetic acid and acetic anhydride products (see page 3, lines 6 to 8), and it proposes therefore the process as set out under point 5.2 above as the solution to this problem. Thus, document (1) rather leads away from distilling the overhead from the splitter column to remove the carbonyl impurities.

Furthermore, document (2) does not disclose a separation of carbonyl impurities, let alone their separation by distillation. In fact, it relates to a process for separating alkanes from vaporised carbonylation products by introducing said vaporised carbonylation products into a first distillation zone to remove an overhead product and a bottom product, separating said overhead product into a light phase and a heavy phase, removing a slip stream of said heavy phase, introducing it into a second distillation zone and distilling so as to remove an overhead stream free of alkanes and a bottoms stream consisting essentially of alkanes, and recycling said alkane-free overhead stream to the separation zone (see column 2, lines 29 to 58). Thus, for the simple reason that this document neither addresses the technical problem underlying the patent in suit, nor discloses a reduction of carbonyl impurities in a recycle stream, it cannot give any

incentive to the skilled person to the claimed solution of the technical problem underlying the patent in suit.

- 6.9 Document (3), referred to by the Appellant with respect to the distillation embodiment of the present process indicated under point 6.4 (c) above, and in particular regarding the addition of an alcohol in order to avoid clogging in the distillation column, only discloses some data concerning the solubility of metaldehyde and paraldehyde in solvents including ethanol.

Thus, apart from the fact that this document does not address the technical problem underlying the present patent either, it is already irrelevant for recognising inventive step, because the distillation step as such is already non-obvious for the reasons set out under point 6.8 above.

- 6.10 Therefore, documents (1), (2) and (3), taken alone or in combination, do not provide an incentive to the skilled person to arrive at the claimed solution of the above defined technical problem underlying the application in suit.

- 6.11 In conclusion, the subject-matter of present Claim 1, and by the same token, that of the dependent Claim 2, 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 department of first instance with the order to maintain the patent on the basis of Claims 1 and 2 submitted as sole "main request" at the oral proceedings, and a description and figures yet to be adapted.

The Registrar:

The Chairman:

N. Maslin

R. Freimuth