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D E C I S I O N
of 29 October 2002

Case Number: T 0546/00 - 3.3.1

Application Number: 94108218.2

Publication Number: 0632007

IPC: C07C 53/12

Language of the proceedings: EN

Title of invention:

Method of removing iodine compounds from crude acetic anhydride or mixture of crude acetic anhydride and crude acetic acid

Patentee:

DAICEL CHEMICAL INDUSTRIES, LTD.

Opponent:

BP Chemicals Ltd

Headword:

Crude acetic anhydride/DAICEL

Relevant legal provisions:

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

Keyword:

"Inventive step (no) - obvious to try - no deterrent teaching in the art"

Decisions cited:

-

Catchword:

-



Case Number: T 0456/00 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 29 October 2002

Appellant: BP Chemicals Ltd
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Representative: Hoey, Shona
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Respondent: DAICEL CHEMICAL INDUSTRIES, LTD.
(Proprietor of the patent) 1, Teppo-cho
Sakai-shi,
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Representative: Grünecker, Kinkeldey, Stockmair & Schwanhäusser
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 13 April 2000
rejecting the opposition filed against European
patent No. 0 632 007 pursuant to Article 102(2)
EPC.

Composition of the Board:

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

Summary of Facts and Submissions

I. The Appellant (Opponent) lodged an appeal on 25 May 2000 against the decision of the Opposition Division posted on 13 April 2000 rejecting the opposition against European patent No. 632 007 which was granted on the basis of four claims, the only independent claim 1 and dependent claim 2 reading as follows:

"1. A method for producing acetic anhydride or a mixture of acetic anhydride and acetic acid and for removing iodine compounds therefrom wherein crude acetic acid anhydride or a mixture of crude acetic anhydride and crude acetic acid is formed by reacting dimethyl ether and/or methyl acetate, and optionally water and/or methanol, with carbon monoxide in the presence or absence of hydrogen in the presence of a rhodium compound as a catalyst and methyl iodide as a cocatalyst and an iodide as a reaction promoter, which comprises the steps of heat-treating the crude acetic anhydride or the mixture of crude acetic anhydride and crude acetic acid in the presence of methanol and/or methyl acetate in a treatment tank at 110 to 200°C for 5 to 60 minutes, wherein the amount of methanol and/or methyl acetate is 1 to 1000 mol per mol of the iodine compound to be treated and distilling the heat-treated crude acetic anhydride or the heat-treated mixture of crude acetic anhydride and crude acetic acid.

2. The method of claim 1, wherein the crude acetic anhydride or the mixture of crude acetic anhydride and crude acetic acid to be heat-treated is obtained by taking a crude reaction liquid out of the reactor, subjecting the crude reaction liquid to flash

evaporation to give a vapor comprising acetic anhydride and acetic acid, introducing the vapor into a distillation column, and taking out the crude acetic anhydride or the mixture of crude acetic anhydride and crude acetic acid through a side cut near the bottom of the distillation column."

II. Notice of Opposition had been filed by the Appellant requesting revocation of the patent as granted in its entirety for the ground of lack of inventive step. *Inter alia* the following documents were submitted in opposition proceedings:

- (1) US-A-4 039 395,
- (2) JP-B-61/8811, considered in the form of its English translation,
- (3) US-A-4 628 041,
- (4) Catalysis Today, Vol. 13, pages 73 to 91 (1992)
- (5) US-A-5 169 982,
- (6) EP-A-535 825 and
- (8) JP-B-58/116436, considered in the form of its English translation.

III. The Opposition Division held that the subject-matter claimed involved an inventive step in the light of the documents cited. The documents (5) and (8) represented the closest prior art as they related to the removal of iodine impurities from acetic anhydride by a chemical treatment. Starting the assessment of inventive step

from that state of the art the problem underlying the patent in suit was seen in the provision of another process for the same purpose. The solution provided by claim 1 consisted in the use of methanol and/or methyl acetate in a pretreatment step, followed by distillative separation. The documents (1) to (3) disclosed the use of either methanol or methyl acetate for a similar purpose, namely the reduction of iodine impurities in acetic acid streams. A combination of the teaching of documents (1) or (2) with document (5) appeared as one of the possibilities the skilled person could have used, but there was no indication that he would have regarded the use of methanol and/or methyl acetate as being the method of choice. The shown improvement of the present invention when using a pretreatment step was unexpected in view of the examples 6 and 7 of document (5). Therefore a combination of that document with documents (1) or (2) would not have led to the claimed subject-matter since the skilled person could not have seen any reason in document (5) to prefer the use of a pretreatment tank. Document (3) was not directed to the removal of traces of iodine impurities but rather to the recovery of rhodium from a fraction which could contain up to 24 wt% of iodides.

IV. The Respondent (Proprietor of the patent) defended the maintenance of the patent in suit on the basis of the claims as granted and subsidiarily on the basis of three amended claims submitted as auxiliary request on 3 March 2000, claim 1 reading as follows:

"1. A method for producing acetic anhydride or a mixture of acetic anhydride and acetic acid and for removing iodine compounds therefrom, wherein crude

acetic acid anhydride or a mixture of crude acetic anhydride and crude acetic acid is formed by reacting dimethyl ether and/or methyl acetate, and optionally water and/or methanol, with carbon monoxide in the presence or absence of hydrogen in the presence of a rhodium compound as a catalyst and methyl iodide as a cocatalyst and an iodide as a reaction promoter, which comprises the steps of

- heat-treating the crude acetic anhydride or the mixture of crude acetic anhydride and crude acetic acid in the presence of methanol and/or methyl acetate in a treatment tank at 110 to 200°C for 5 to 60 minutes, wherein the amount of methanol and/or methyl acetate is 1 to 1000 mol per mol of the iodine compound to be treated, and wherein the crude acetic anhydride or the mixture of crude acetic anhydride and crude acetic acid to be heat-treated is obtained by taking a crude reaction liquid out of the reactor, subjecting the crude reaction liquid to flash evaporation to give a vapor comprising acetic anhydride and acetic acid, introducing the vapor into a distillation column, and taking out the crude acetic anhydride or the mixture of crude acetic anhydride and crude acetic acid through a side cut near the bottom of the distillation column, and

- distilling the heat-treated crude acetic anhydride or the heat-treated mixture of crude acetic anhydride and crude acetic acid."

At the oral proceedings before the Board held on 29 October 2002 the Respondent conceded that the subject-matter of claim 1 according the auxiliary request was identical to that of claim 2 according to

the main request. In respect of that claimed embodiment he started from document (6) as the closest state of the art in the assessment of inventive step. This document was directed to a purification process for removing iodine impurities from acetic anhydride comprising a pre- and a post-flash evaporation/distillation step. The problem underlying the patent in suit was to provide an alternative purification process for removing iodine impurities from acetic anhydride having a similar purification efficiency. The claimed process differed from the known process in using a chemical reaction in a separate treatment tank for that purpose, namely a reaction with methanol or methyl acetate. The question was where the skilled person should look for an alternative purification process. Documents (1) and (2) were directed to the purification of acetic acid with methanol or methyl acetate. Since document (5) indicated at column 1, line 51 to column 2, line 3 that agents used for purifying acetic acid could not be used for purifying acetic anhydride, the skilled person was deterred from applying the teaching of documents (1) and (2) to acetic anhydride as they were directed to the treatment of acetic acid. Furthermore, neither document (5) nor documents (1) and (2) gave a hint to use a separate treatment tank as required in the patent in suit. Though document (3) referred to the purification of an acetic acid product stream from iodine impurities using a flash distillation followed by a treatment with methyl acetate, this product stream was different from that treated in the claimed process. The product stream treated in document (3) comprised rhodium catalyst and lithium ions stemming from the synthesis step of acetic acid and the rhodium catalyst was supposed to interfere with the purification

process. Iodine impurities were enriched in that product stream with the consequence that it contained before and after purification a higher amount of iodine impurities than the product stream treated in the patent in suit. Therefore the invention was not obvious in view of that state of the art.

The Respondent further submitted at the oral proceedings before the Board that the object of the present invention comprised the effective removal of hydrogen iodide (patent specification page 3, line 21). When questioned on the difference in impurities in respect of the prior art, he conceded that the impurity hydrogen iodide was mandatorily present and had to be removed in the purification process according to the invention as well as according to document (6) since that impurity was inevitably formed during the precedent synthesis reaction.

- V. The Appellant, with respect to the embodiment claimed in the auxiliary request and claim 2 of the main request, submitted that document (6) represented the closest prior art. That document was directed to the purification of acetic anhydride from iodine impurities using a conventional flash distillation. As shown in figure 2 of document (6) this flash distillation was identical to that of the claimed process. Starting from that document in the assessment of inventive step, the further document (3) taught a purification process for removing iodine impurities from acetic acid using methyl acetate after a flash distillation. That purification process showed an extraordinary efficiency since it was applied to a product stream having a high load of iodine impurities. The chemical purification of acetic acid and of acetic anhydride were closely

related as the iodine impurities to be removed from either product comprised essentially hydrogen iodide. The impurity hydrogen iodide was well known to be compulsorily formed in the preceding synthesis reaction of both acetic acid and acetic anhydride and, thus, to be removed in the purification process of documents (3) and (6) as well as according to the invention. The inevitable formation of the impurity hydrogen iodide was not in dispute between the Parties and even shown in the reaction scheme on page 86 of document (4). Remaining rhodium catalyst originating from the preceding synthesis reaction and comprised in the product stream treated in document (3) did not affect the purification process of removing iodine impurities from that product stream with methyl acetate. Thus, the skilled person was guided to the claimed process by using methyl acetate after a flash distillation in order to purify acetic anhydride without involving an inventive step.

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

The Respondent requested as main request that the appeal be dismissed and the patent be maintained as granted and as auxiliary request that the patent be maintained as amended on the basis of the auxiliary request submitted on 3 March 2000.

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

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments (Article 123 EPC)*

In claim 1 according to the auxiliary request the features of claim 2 as granted, i.e a flash evaporation followed by a specific distillation, have been incorporated into granted claim 1. That amendment finds support in claim 2 of the application as filed and, thus, complies with the requirements of Article 123(2) EPC.

That amendment of claim 1 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. *Inventive step*

The sole issue arising from this appeal consists in deciding whether or not the subject-matter of the claims of the patent in suit as granted according to the main request or of the claims as amended according to the auxiliary request involves an inventive step.

3.1 Independent claim 1 of the auxiliary request is directed to a preferred embodiment of the main request, namely to the subject-matter of dependent claim 2 as granted. Thus, the subject-matter claimed in the auxiliary request is covered by that of claim 1 of the main request. In case the embodiment according to the auxiliary request lacked inventive step, such a line of requests would mandatorily result in the conclusion that the preceding main request, which encompasses that obvious embodiment, at least to that extent, cannot

involve an inventive step either. For this reason, it is appropriate that the auxiliary request, in particular the subject-matter of claim 1 thereof, is examined first as to its inventive ingenuity.

- 3.2 The patent in suit is directed to a process for producing acetic anhydride, optionally in admixture with acetic acid, and for removing iodine compounds therefrom which process comprises the steps of preparing crude acetic anhydride by reacting a carbonylation feedstock with carbon monoxide in the presence of a rhodium catalyst, methyl iodide cocatalyst and an iodide reaction promoter, subjecting the crude reaction liquid to flash evaporation followed by a distillation, taking out the crude acetic anhydride through a side cut near the bottom of the distillation column, heat-treating the crude acetic anhydride and distilling the heat-treated crude acetic anhydride. The patent in suit aims at an efficient removal of iodine impurities from the product acetic anhydride.

A similar process already belongs to the state of the art in that document (6) describes a process for producing a mixture of acetic anhydride and acetic acid and for removing iodine contaminants therefrom (claims 2, 8 and 9; examples 3 and 4). That process comprises the steps of preparing crude acetic anhydride by reacting a carbonylation feedstock with carbon monoxide in the presence of a rhodium catalyst, methyl iodide cocatalyst and an iodide reaction promoter, e.g. hydrogen iodide (page 3, line 23). The crude reaction liquid is then subjected to a pre-flash vaporisation and the vapour fraction thereof passed into a distillation column (claim 2; page 6, lines 17 to 51,

and figures 1 and 2). Acetic anhydride having substantially reduced iodine content is removed one or two trays from the bottom of the distillation column (page 6, lines 50 and 51; examples 3 / 4 on page 7, lines 55 and 56; figure 2, line 12). A post-flash vaporisation and distillation of the acetic anhydride follows (claim 2; page 6, lines 52 and 53). Document (6) achieves a substantial reduction of iodine contamination in the final product down to 1.3 ppm iodide (claim 2; page 2, line 57; example 3).

For these reasons, the Board considers, in agreement with the Appellant and the Respondent, that the disclosure of document (6) specified above represents the closest state of the art, and, hence, the starting point in the assessment of inventive step.

3.3 In view of this state of the art the problem underlying the patent in suit, as correctly formulated by the Respondent, consists in providing a further purification process for removing iodine impurities from acetic anhydride having a similar purification efficiency.

3.4 As the solution to this problem the patent in suit proposes a purification process for removing iodine impurities from flash distilled crude acetic anhydride and optionally acetic acid which process is characterised by heat-treating the crude acetic anhydride in the presence of methanol and/or methyl acetate in a treatment tank at 110 to 200°C for 5 to 60 minutes, wherein the amount of methanol and/or methyl acetate is 1 to 1000 mol per mol of the iodine compounds to be treated.

3.5 The Appellant never disputed that the claimed process successfully achieves the efficient removal of iodine compounds; and the Board is not aware of any reason for challenging this finding. The specification of the patent in suit demonstrates in examples 1 and 2 the low iodine contamination of acetic anhydride purified according to the claimed process. For these reasons, the Board is satisfied that the problem underlying the patent in suit has been successfully solved.

3.6 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.

When starting from the purification process known from document (6) it is a matter of course that the person skilled in the art, seeking to provide an alternative purification process for removing iodine impurities, would turn his attention to that prior art just addressing that technical problem. He would take document (3) into consideration which aims at removing iodine compounds from a product stream containing acetic acid (claim 1, steps 3 and 4; column 2, lines 14 and 15). He would be struck in particular by the efficiency of the process of that document since it "removes substantially all of the iodide" (column 3, line 36).

That document (3) teaches to remove iodine compounds from a product stream which has been subjected to a flash distillation by adding an excess of methyl acetate, thereby converting the methyl acetate to methyl iodide which is removed by subsequent distillation (claim 1, steps 3 and 4). The treatment

with methyl acetate is operated at a temperature of 130 to 190°C (claim 2) for a sufficient time (column 3, line 30) and the molar excess of methyl acetate to total iodine is e.g. 15:1 (example 2, table 1 including footnote 2).

The Board concludes from the above that document (3) gives the person skilled in the art a concrete hint on how to solve the problem underlying the patent in suit to provide a further purification process for removing iodine impurities (cf. point 3.3 *supra*), namely by heat-treating a flash distilled mixture of acetic anhydride and acetic acid known from document (6) with methyl acetate for a sufficient time at a temperature and a molar excess of methyl acetate which lie within the claimed ranges of 110 to 200°C and 1 to 1000 mol, thereby arriving at the solution proposed by the patent in suit. Therefore, in the Board's judgement, it was obvious to try to follow the avenue indicated in the state of the art with a reasonable expectation of success without involving any inventive ingenuity. The numerical time range of 5 to 60 minutes indicated in claim 1 for that heat-treatment can neither provide the claimed process with any inventive ingenuity as that range is arbitrary and the determination of a time range to be considered "sufficient" is anyhow within the routine of a skilled person, which finding was not disputed by the Respondent.

3.7 For the following reasons the Board cannot accept the Respondent's arguments designed to support inventive step.

3.7.1 The Respondent argued that the product stream treated in document (3) was quantitatively different from that

treated in the claimed process since the iodine impurities were enriched in the former with the consequence that it contained before and also after purification a higher amount of iodine impurities than the product stream treated in the patent in suit.

However, claim 1 does not specify any particular limit for the level of iodine impurities in the product stream to be treated as well as in the final product. Thus, the Appellant's argument based on an alleged mandatory and significant difference in the level of iodine impurities between the process of document (3) and of the patent in suit is not supported by the facts. Moreover, the skilled person is all the more likely to pursue the course indicated in document (3) as the process of that document was taught to be very efficient since it removes substantially all of the iodine.

- 3.7.2 The Respondent argued furthermore that the product stream treated in document (3) was qualitatively different from that treated in the claimed process since the former comprised rhodium catalyst and lithium ions originating from the synthesis step. He concluded that the rhodium catalyst was supposed to trouble a purification step.

It is true that the product stream treated in the purification step of claim 1 is substantially free of rhodium catalyst and lithium, contrary to that of document (3). The Board, however, cannot share the Respondent's conclusion drawn from that finding. The teaching of document (3) itself is evidence to the contrary as the purification process of that document is successful in the presence of rhodium catalyst.

Thus, there is no reason to speculate about or to suppose any troubling of the purification step by remaining rhodium catalyst, as the Respondent does and even less in the absence of it in the product stream purified according to the claimed process.

- 3.7.3 Moreover, the Respondent alleged that the contaminants and impurities to be removed in the process of document (3) were qualitatively different from those removed in the claimed process where *inter alia* hydrogen iodide was removed (specification of the patent in suit, page 3, line 22). Therefore, document (3) was not to be taken into account when looking for a solution to the problem underlying the patent in suit.

However, the product stream treated in the purification process of document (3) is likewise contaminated with *inter alia* hydrogen iodide (column 2, lines 20 and 30) and that purification process removes substantially all of the iodine impurities, including explicitly hydrogen iodide (column 3, line 37). Hence, the Respondent's allegation is not supported by the facts; on the contrary the skilled person had even an additional incentive to consider document (3) for solving the problem underlying the patent in suit, thereby rendering the claimed invention obvious.

- 3.7.4 The Respondent submitted that document (5) indicated at column 1, line 51 to column 2, line 3 that agents for purifying acetic acid could not be used for purifying acetic anhydride, thereby deterring the skilled person from applying a purification agent known for acetic acid in a purification process of acetic anhydride.

However, the teaching of document (5) addressed by the Respondent simply indicates that acetic anhydride undergoes saponification when using an aqueous alkaline solution as purifying agent (column 1, line 67 to column 2, line 3). That document specifies at column 1, line 66 and 67 that this statement is limited exclusively to that particular agent, i.e. to an aqueous alkaline solution. Document (3) directed to the purification of an acetic acid containing product stream, however, teaches to use a different agent, namely methyl acetate. For that reason, the person skilled in the art is not deterred from applying the teaching of document (3), i.e. using methyl acetate as purifying agent, in the process for purifying acetic anhydride known from the closest prior art document (6) in order to solve the problem underlying the patent in suit.

- 3.8 For these reasons, the solution to this problem proposed in claim 1 is obvious in the light of the prior art.
4. As a result, the Respondent's auxiliary request is not allowable for lack of inventive step pursuant to Article 56 EPC.
5. The main request covers the subject-matter of claim 1 of the auxiliary request in the form of the preferred embodiment of claim 2 as granted. Therefore the considerations having regard to inventive step given in points 3.2 to 3.6 *supra* and the conclusion drawn in point 3.8 *supra* with respect to the auxiliary request applies also to the main request, i.e. the subject-matter claimed is obvious and does not involve an inventive step.

6. In these circumstances, the Respondent's main request shares the fate of the auxiliary request in that it too is not allowable for lack of inventive step pursuant to Article 56 EPC.

Order

For these reasons it is decided:

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar:

The Chairman:

N. Maslin

A. Nuss