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File Number: T 219/90 - 3.3.1

Application No.: 83 300 662.0

Publication No.: 0 087 870

Title of invention: Process for the production of acetic anhydride and
acetic acid

Classification: C07C 51/56

DECISION
of 8 May 1991

Proprietor of the patent: BP Chemicals Limited

Opponent: Hoechst AG

Headword: Acetic anhydride/BP

EPC Article 56

Keyword: "Inventive step (denied)"

Headnote



Case Number : T 219/90 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 8 May 1991

Appellant :
(Proprietor of the patent)

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Representative :

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Respondent :
(Opponent)

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Decision under appeal :

Decision of Opposition Division of the European
Patent Office of 5 December 1989, posted on
5 February 1990 revoking European patent
No. 0 087 870 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
J.-C. Saisset

Summary of Facts and Submissions

I. European patent No. 0 087 870 in respect of European patent application No. 83 300 662.0, which was filed on 10 February 1983, was granted on 17 April 1985 (cf. Bulletin 85/16) on the basis of seven claims.

II. On 7 December 1985 a notice of opposition was filed in which the revocation of the patent was requested on the ground that its subject-matter did not involve an inventive step. The opposition was supported, inter alia, by DE-A-2 450 965 (2).

In the course of the opposition proceedings, the Respondent (Opponent) also referred to DE-A-2 940 753 (1), DE-A-2 441 502 (5) and DE-A-2 610 036 (6).

III. By a decision delivered orally on 5 December 1989, with written reasons dispatched on 5 February 1990, the Opposition Division revoked the European patent. The Opposition Division held that the claimed subject-matter was novel but it did not involve an inventive step in the light of the disclosure in documents (1) and (2). The Opposition Division considered there was a clear incentive to carry out the carbonylation of methyl acetate in the presence of a predetermined amount of water in order to solve the technical problem underlying the disputed patent of providing a carbonylation process in which both acetic anhydride and acetic acid are produced simultaneously in desired amounts, since it is generally recognised that the relative proportions of anhydride and acid obtained depend upon the amount of water present during the carbonylation reaction.

Furthermore, the skilled person would realise that the disclosure of the carbonylation of wet methyl acetate in document (2) could be applied to the process according to document (1), since these processes are technically related to one another.

- IV. An appeal was lodged against the decision on 21 March 1990 with payment of the prescribed fee. In the statement of grounds of appeal filed on 2 June 1990 and during the oral proceedings held on 8 May 1991, the Appellant emphasised that the claimed process was an integrated series of six steps which has been operating successfully on a commercial scale for the last eighteen months. The flexibility of the process to respond quickly to market conditions is achieved by varying the amount of water in the feed to the carbonylation stage. This also avoids the necessity of drying the methyl acetate and removing water from the other feed streams.

Although the Appellant admitted that five of the six steps of the claimed process were disclosed in document (1), he contended that it cannot be considered to be a trivial modification of this prior art process to eliminate its essential dehydration stage since this goes against the teaching of this document and that of documents (5) and (6).

In the Appellant's view, documents (1) and (2) contain contrary teachings and a skilled person would only ignore the teaching of document (1) with the benefit of hindsight, particularly since there are no Examples in document (2) of the use of conditions other than anhydrous ones. Furthermore, document (2) only suggested that minor amounts of water may be tolerated.

The Appellant also argued that the publication dates of the various documents supported his view that the use of wet methyl acetate was not obvious since the later published document (1) chose to dry the methyl acetate in the face of acknowledged difficulties.

- V. The Respondent argued that the claimed process only differed from the process described in document (1) in step (3). However, document (2) discloses that the carbonylation of methyl acetate containing 1 to 5% by weight of water produces a product containing the equivalent amounts of acetic acid. Moreover, the amount of water and/or methanol necessary in the feedstock to the carbonylation reactor to give the desired amount of acetic acid in the product can easily be calculated.

The proposed limitation to rhodium carbonylation catalysts did not render the process inventive since the nature of the catalyst only affected the reaction parameters, not the products of the reaction.

- VI. The Appellant requested that the decision under appeal be set aside and the patent be maintained on the basis of Claims 1 to 7 as granted except for the replacement of the word "metallic" by "rhodium" in Claim 1. The only independent claim in accordance with this request reads as follows:

"A process for the production of acetic anhydride with or without the net co-production of acetic acid from methanol and carbon monoxide in a series of esterification, carbonylation and separation steps comprising:

- 1) reacting methanol with recycle acetic acid in an esterification step to form an esterification product

containing predominantly methyl acetate, water and optionally unreacted methanol,

- 2) removing part of the water from the esterification product,
- 3) reacting the esterification product still containing water with carbon monoxide in a carbonylation step in the presence as catalyst of free or combined rhodium carbonylation catalyst and as promoter of free or combined halogen to form a carbonylation product containing acetic acid and acetic anhydride,
- 4) separating the carbonylation product by fractional distillation into a low boiling fraction containing carbonylation feed and volatile carbonylation promoter components, acetic acid and acetic anhydride fractions, and a higher boiling fraction containing carbonylation catalyst components,
- 5) recycling the low boiling fraction containing carbonylation feed and carbonylation promoter components and the higher boiling fraction containing carbonylation catalyst components to the carbonylation step and,
- 6) recycling at least part of the acetic acid fraction to the esterification step."

Alternatively, the Appellant requested that the patent be maintained on the basis of Claims 1 to 6 filed on 2 June 1990 or Claims 1 to 5 filed on 16 January 1991 with Claim 1 of each set of claims amended in the same manner as Claim 1 of the main request. Claim 1 of the first auxiliary request differs from Claim 1 of the main request in that in step 2 water is removed to produce a product

containing at least 6% w/w water based on the weight of methyl acetate and methyl alcohol if present. Claim 1 in accordance with the second auxiliary request also requires that the recycle acetic acid forms at least 50% of the acetic acid fed to the esterification step.

The Respondent requested that the appeal be dismissed.

VII. At the conclusion of the oral proceedings the Board's decision to dismiss the appeal was announced.

Reasons for the Decision

1. The appeal is admissible.
2. There are no formal objections under Article 123 EPC to any of the sets of claims in accordance with the Appellant's three requests. The replacement of the term "metallic" by "rhodium" in Claim 1 in accordance with each request is justified by the disclosure on page 7, lines 20 and 21 of the published patent application (cf. also column 5, line 57 of the printed patent specification). Claims 2 to 7 of the main request correspond to Claims 2 to 7 as filed and granted.

The requirement in Claim 1 in accordance with the first auxiliary request with respect to the minimum amount of water present in the feed to step 3 of the process is supported by originally filed and granted Claim 4. Claims 2 to 6 of this request correspond to Claims 2, 3 and 5 to 7 as filed and granted.

Claim 1 of the second auxiliary request represents a combination of originally filed and granted Claims 1 and 4. Claims 2 to 5 of this request correspond to Claims 2 and 5 to 7 as filed and granted.

3. The patent in suit concerns a process for the production of acetic anhydride from methanol and carbon monoxide by an integrated series of esterification, carbonylation and separation steps. Document (1), which represents the closest state of the art, discloses a process in which acetic anhydride is prepared by carbonylating substantially anhydrous methyl acetate which has been obtained by drying wet methyl acetate resulting from the esterification of acetic acid with methanol (cf. Claims 2 and 3).

3.1 This prior art process, however, was considered to have the disadvantages that it was necessary to dry the methyl acetate before feeding it to the carbonylation zone and that it lacked the flexibility to respond to the changing demands of the market for both acetic anhydride and acetic acid (cf. the paragraph bridging columns 2 and 3 and column 9, line 62 to column 10, line 3 of the disputed patent specification).

In the light of this closest prior art, the technical problem underlying the patent in suit is to be seen in providing a process which overcomes the above-mentioned disadvantages.

According to the disputed patent, this technical problem is solved by carbonylating methyl acetate containing a certain amount of water to produce a mixture of acetic anhydride and acetic acid and using at least a part of the acetic acid so obtained to produce methyl acetate.

In view of the Examples in the disputed patent, the Board is satisfied that this technical problem is successfully solved.

4. After examination of the cited prior art the Board has reached the conclusion that the claimed subject-matter is novel. Since novelty is not in dispute, it is not necessary to give detailed reasons for this finding.
5. It still remains to be decided whether the subject-matter of the disputed patent involves an inventive step.
- 5.1 Document (1) discloses a process for the dehydration of wet methyl acetate by contacting it with acetic anhydride in an amount of at least stoichiometrically equivalent to the water present in the wet methyl acetate (cf. Claim 1). The acetic anhydride required for this drying process may be obtained by carbonylating the anhydrous methyl acetate (cf. Claim 2). According to Claim 3 of this document, wet methyl acetate is prepared by esterifying methanol with the acetic acid separated from the mixture of acetic anhydride and acetic acid leaving the carbonylation zone.

This document further discloses that the large bulk of the acetic anhydride remaining after the removal of a small portion to dry the wet methyl acetate may be used to produce, for example, cellulose acetate or vinyl acetate (cf. the paragraph bridging originally numbered pages 17 and 18). The acetic acid resulting as a by-product from both these processes may be employed as the acid feed to the esterification reaction to supplement the acetic acid produced in the dehydration step (cf. first complete paragraph on page 18). Thus, the acetic acid required for the esterification of the methanol is obtained from the dehydration of the wet methyl acetate with acetic anhydride and subsequent operations involving the use of the acetic anhydride prepared in the carbonylation stage.

From the whole disclosure of document (1) it is clear that its primary aim is to provide a process for the manufacture of acetic anhydride which can be readily integrated with processes involving its use in the production of other valuable chemicals. Since the skilled person is aware from his common general knowledge as reflected in the last paragraph on page 6 of document (2) that the presence of water in the carbonylation feed does not affect the activity of the carbonylation system but merely reduces the yield of acetic anhydride, he would see this possible reduction in the yield of the desired product as the only reason for the measures taken to ensure that the methyl acetate is substantially anhydrous.

In the Board's judgement, this is also the reason for the use of anhydrous or substantially anhydrous conditions in the processes disclosed in documents (5) and (6) (cf. document (5), page 5, lines 20 to 23; document (6), page 6, lines 9 to 12).

- 5.2 Document (2) discloses a process for the preparation of acetic anhydride by carbonylating methyl acetate in the presence of a rhodium carbonylation catalyst and iodine in free or combined form (cf. the claim and fourth complete paragraph on page 3). According to the third complete paragraph on page 2 of this document, the methyl acetate may contain methanol or small quantities of water, for example 1 to 5% of water. However, the presence of either of these compounds in the carbonylation feed results in the formation of not only acetic anhydride but also acetic acid in an amount approximately equivalent to the quantity of methanol or water employed.

There is no contradiction between the teaching of document (2) and that of documents (1), (5) and (6) since the latter documents are directed to the preparation of

acetic anhydride as the sole product of the carbonylation reaction, hence the need for substantially anhydrous conditions. Document (2), on the other hand, envisages the production of both acetic anhydride and acetic acid and, thus, the presence of water may be tolerated, at least to a certain extent.

The mere fact that document (1), which was published after document (2), insists on employing substantially anhydrous methyl acetate is of no significance, since the use of anhydrous or wet methyl acetate is dependent upon whether it is desired to obtain a maximum yield of acetic anhydride or a mixture of acetic anhydride and acetic acid.

Therefore, document (2) teaches, even in the absence of any Examples, that the presence of water in the carbonylation feed does not have a negative influence on the rhodium carbonylation catalyst and that, provided one is prepared to accept the production of both acetic anhydride and acetic acid, it is not necessary to use substantially anhydrous methyl acetate.

In the Board's judgement, the teaching of document (2) provides a clear pointer to the solution of the technical problem of overcoming the above-mentioned disadvantages of the process disclosed in document (1). Therefore, the subject-matter of Claim 1, in accordance with the main request, does not involve an inventive step.

- 5.3 Claim 1 in accordance with the first auxiliary request contains the additional feature that water should be removed from the esterification product to such an extent that a product is obtained which contains at least 6% w/w water based on the weight of methyl acetate and methanol, if present. In the Board's view, the reference to 6% as a

lower limit for the amount of water present does not render the claimed process inventive since in document (2) the quantity of water that may be present is only given by way of example. Therefore, the reference to 5% of water would not deter the skilled person from carrying out the process with more than this amount of water. Furthermore, based on his common general knowledge of the chemical reactions the methyl acetate, water and methanol undergo in the carbonylation zone, the skilled person is able to calculate the levels of water and/or methanol necessary to obtain the desired acetic anhydride/acetic acid product ratios.

Therefore, in the Board's judgement, Claim 1 of the first auxiliary request is also unallowable.

- 5.4 Claim 1 in accordance with the second auxiliary request contains the additional feature that the recycle acetic acid forms at least 50% of the acetic acid fed to the esterification step.

The amount of acetic acid produced by the process is entirely dependent upon the amount of water and/or methanol present in the feed to the carbonylation reactor, therefore, by controlling the composition of the carbonylation feed, the skilled person is able to determine the amount of acetic acid available for recycle to the esterification step. The determination of the optimum level of recycle in any particular circumstances would form part of the normal duties of the skilled person.

Therefore, in the Board's judgement, the subject-matter of Claim 1, in accordance with the second auxiliary request, does not involve an inventive step.

6. The Board has no reason to doubt that a plant using the process of the disputed patent has been operated successfully on a commercial scale for at least eighteen months and that several companies have expressed an interest in obtaining licenses to operate the process. However, in the circumstances of the present case, commercial success alone cannot be regarded as indicative of inventive step even if the Board were satisfied that the success was derived from technical features of the process and not from extraneous causes.

Order

For these reasons, it is decided that:

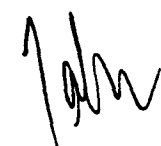
The appeal is dismissed.

The Registrar:



E. Görgmaier

The Chairman:



K.J.A. Jahn