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Bezeichnung der Erfindung: Process for recovery of olefinic nitriles

Title of invention:

Titre de l'invention :

Klassifikation / Classification / Classement : C07C120

### ENTSCHEIDUNG / DECISION

vom / of / du 20 August 1985

~~Anmelder / Applicant / Demandeur :~~

Patentinhaber / Proprietor of the patent / The Standard Oil Company

Titulaire du brevet :

Einsprechender / Opponent / Opposant : Bayer AG

Stichwort / Headword / Référence :

EPU / EPC / CBE Art.56,114(2)

"Inventive step - problem not plausibly solved"

"facts or evidence not submitted in due time - later evaluation  
of documents submitted in time"

Leitsatz / Headnote / Sommaire

**Europäisches  
Patentamt**  
Beschwerdekammern

**European Patent  
Office**  
Boards of Appeal

**Office européen  
des brevets**  
Chambres de recours



**Case Number: T 186 / 83**

**DECISION**  
**of the Technical Board of Appeal 3.3.1**  
**of 20 August 1985**

**Appellant:**  
**(Opponent)**  
BAYER AG LEVERKUSEN  
Konzernverwaltung RP  
Patentabteilung  
Bayerwerk  
D-5090 Leverkusen

**Representative:**

**Respondent:**  
**(Proprietor of the patent)**  
The Standard Oil Company  
Midland Building  
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USA

**Representative:**  
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**Decision under appeal:**  
Decision of the Opposition Division of the European Patent Office  
dated 5 September 1983 rejecting the opposition filed against  
European patent No. 566 pursuant to  
Article 102(2) EPC

**Composition of the Board:**

**Chairman:** K. Jahn  
**Member:** F. Antony  
**Member:** O. Bossung

Summary of Facts and Submissions

1. European Patent Application No. 78 100 473.4, which had been filed on 21.07.78, claiming USA priority of 29.07.77, was granted as European Patent No. 566 on 02.09.81 with twelve claims. Claim 1 reads :

"A process for the recovery and purification of acrylonitrile or methacrylonitrile produced by the ammoxidation reaction of propylene or isobutylene, molecular oxygen and ammonia in the presence of ammoxidation catalysts, comprising :

- a) contacting the ammoxidation reactor effluent containing acrylonitrile or methacrylonitrile, acetonitrile, and impurities with an aqueous quench liquid in a quench system to produce a gaseous quench effluent from said quench system;
- b) absorbing said gaseous quench effluent in water to form an aqueous solution;
- c) feeding the aqueous solution to an intermediate tray of a distillation column having a plurality of trays, using solvent water introduced in the top of said column to perform a water extractive distillation, wherein an overhead vapor stream of acrylonitrile or methacrylonitrile with some water is removed from the top of the column, and a liquid stream containing water and impurities is removed from the bottom of the column;
- d) removing a first sidestream from the lower half of said column to recover acetonitrile;

- e) feeding at least a part of the liquid bottoms from said column to the quench system as quench liquid, characterized by removing a vapor stream containing water from the lower fourth of said column so reducing the size of the bottom stream."

Independent Claim 5, directed to a modification of the process of Claim 1, reads as follows :

"A process for the recovery and purification of acrylonitrile or methacrylonitrile produced by the ammoxidation reaction of propylene or isobutylene, molecular oxygen and ammonia in the presence of ammoxidation catalysts, comprising :

- a) contacting the ammoxidation reactor effluent containing acrylonitrile or methacrylonitrile, acetonitrile, and impurities with an aqueous quench liquid in a quench system to produce a gaseous quench effluent from said quench system;
- b) absorbing said gaseous quench effluent in water to form an aqueous solution;
- c) feeding the aqueous solution to an intermediate tray of a first distillation column having a plurality of trays, using solvent water introduced in the top of said first column to perform a water extractive distillation, wherein a first overhead vapor stream of acrylonitrile or methacrylonitrile with some water, is removed from the top of the first column, and a liquid stream containing acetonitrile, impurities and water is removed from the bottom of the first column;

d) feeding at least a part of the liquid bottoms from said first column to a second column wherein distillation is performed to remove a second overhead vapor stream of acetonitrile and water from the top of the second column, and a second liquid stream containing water and impurities from the bottom of the second column.

e) feeding at least part of the second liquid bottoms stream to the quench system of (a) as the aqueous quench liquid, characterized by removing a vapor stream from the lower fourth of said second column so reducing the size of the bottom stream."

II. On 21.05.82, the Opponents (Appellants) lodged opposition against the patent, requesting its complete revocation, alleging anticipation thereof by the document

(1) DE-A-2 050 722.

III. By the Decision of 05.09.83 the Opposition Division rejected the opposition.

The Opposition Division considered the invention according to independent Claims 1 and 5 novel with respect to the only citation relied on by the Opponents, because the claimed characterising feature pertains to removing a vapour stream containing water from the lower fourth of the relevant column, so reducing the bottom stream, a feature not disclosed in the citation. The Opponents having only asserted that the claimed process was "anticipated" by the citation (i.e. not novel), the Opposition Division's decision did not discuss the question of inventive step and rejected the opposition on the sole basis that the patent was novel.

- IV. A notice of appeal was filed by the Appellants (Opponents) against the decision of the Opposition Division on 21.10.83, and the fee for appeal paid. The Statement of Grounds submitted together with the notice of appeal can be summarized essentially as follows :

The Appellants continue to rely on their only citation (1), but have modified their attack to the extent that they now assert that the claimed process is obvious. They point out that the removal of a vapour stream containing water from above the bottom of the respective column can be clearly deduced from (1), even though, in the latter, the said removal has been separated from the said column and transferred to an evaporation unit. This separation is said to have two advantages, namely reduction of volatile toxic compounds and use of the energy of the vapour stream. The claimed process is thus alleged to be only a less favourable embodiment of the process of (1).

The Appellants further contend that, according to their own tests and in contrast to what the patent claims, the condensate of the said vapour stream is not suitable for treatment in a biopond, the concentration of cyanide ions being too high and requiring first an at least 1:100 dilution with water.

- V. The Respondents (Patentees) contest the Appellants' arguments. They emphasize that, according to (1) there is no offtake of vapour from the lower part of any column, especially as the flow from the bottom line 3 (Fig. 1) is in liquid form. Therefore, in their view, a vapour stream from the lower fourth of the column cannot be suggested by (1).

Also, in the system of (1), there is no recycling to a quencher, because no quencher is mentioned. The recycling of (1) is to an absorber, so that a very substantial proportion of the material fed into the system is recycled. Consequently, the material is subjected to multiple high temperature treatment, which inevitably leads to polymerisation and accumulation of materials difficult to purify. In the system of (1) there is no mention of acetonitrile recovery either.

They consider

(2) US-A-3 399 120

the closest prior art and additionally refer to

(3) US-A-3 936 360.

The Respondents insist that the condensate of the vapour stream removed near the bottom of their column requires no detoxification, because it contains fewer cyanide ions than the Opponents assert; a dilution of 1:1 being sufficient for purification in a biopond.

VI. By letter received on 29.07.85, the Respondents stated that they did not intend to be represented at the Oral Proceedings appointed for 20.08.85. In a further letter received on 13.08.85 the Respondents contended that, if the Appellants were to attempt to introduce new facts or significant substantive issues at this stage, this should be rejected, having regard to Article 114(2) EPC. They request that the appeal should be dismissed and the patent be maintained.

VII. In the Oral Proceedings, the Appellants reaffirmed their previous arguments. In particular, the Appellants continued to deny that the most important alleged effect of the claimed process, i.e. suitability for biopond treatment of the condensate of the vapour stream characterising the claimed process, is in fact achieved. In this connection they pointed to the comparative results of Example 2 of (1). They requested that the decision under appeal be set aside and the patent be revoked.

#### Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC; it is thus admissible.
  
2. The invention relates to a process for the recovery and purification of acrylonitrile or methacrylonitrile produced by the ammoxidation reaction of propylene or isobutylene, molecular oxygen and ammonia in the presence of ammoxidation catalysts. Considering the preambles of Claims 1 and 5 together, the following features - somewhat simplified - are mentioned :
  - (A) contacting the reactor effluent with an aqueous quench liquid in a quench system, to produce a gaseous quench effluent;
  
  - (B) absorbing said gaseous effluent in water, to form an aqueous solution;
  
  - (C) subjecting the said solution to a water extractive distillation by feeding it to an intermediate tray of a (first) distillation column, using solvent water introduced at the top of said (first) column to perform said extractive distillation, wherein a (first) overhead



vapour stream of acrylonitrile or methacrylonitrile with some water is removed from the top of the column, and a (first) liquid stream containing water and impurities is removed from the bottom of said (first) column; and either

- (D1) removing and recovering acetonitrile in a (first) sidestream from the lower half of said (first) column, or
- (D5) removing acetonitrile with the said (first) liquid stream, feeding the said stream to a second column wherein distillation is performed, to remove a (second) overhead vapour stream of acetonitrile from the top, and a (second) liquid stream containing water and impurities from the bottom of said (second) column;
- (E) feeding to the quench system as quench liquid at least part of the liquid bottom stream from said (first) column if there is no second column, or from said second column if such exists.

The characterising clause supplements the above combination of features by one of

- (F) removing a vapour stream containing water from the lower fourth of said (first) column if there is no second column, or of said second column if such exists, so reducing the size of the respective bottom stream.
3. Of the three citations, (3) is considered the closest prior art, as it is the only document which discloses a quench system and recycling thereto of a bottom stream from a column. Of the above features, the combination of the following ones is known from (3), the bracketed references indicating passages where the respective disclosure can be

found : A (column 4, lines 12 to 13; reference numeral 2 of the drawing), B (column 4, lines 31 to 33; reference numeral 9 of the drawing) and E (Abstract; column 1, lines 42 to 44; column 4, lines 50 to 53, and column 4, line 66, to column 5, line 3; lines 30, 31, 32 of the drawing). While features C, D (D1 or D5) and F are not specifically disclosed in (3), it is made clear in column 2, lines 38 to 47, that the "intermediate system" (comprising absorber and columns) is "not critical". Therefore, the disclosure in (3) of features A + B + E is relevant for the claimed process as well.

4. Starting from (3), the technical problem (object) underlying the invention is to be investigated.

From the description of the patent in suit, a twofold object might be deduced: reduction of the size of the waste stream leaving the quench column 102 through conduit 106, thereby reducing the energy needed to concentrate such stream prior to its disposal, e.g. by incineration; and achieving such reduction by removal of an aqueous phase in a form suitable for being sent to a biopond or a similar biological treatment.

5. According to the claimed process, this two-fold object is to be achieved through combination of above features C, D (D1 or D5) and F with the total of the features known from (3), viz. A + B + E, the critical feature of the invention being F.

- 5.1 It is clear and beyond dispute that the first element of the above two-fold object, i.e. reduction of the waste stream size, is in fact achieved by feature F; for, removal of a vapour stream must concentrate, and therefore reduce the size of, the remaining aqueous liquid.

5.2 It is, however, contested by the Appellants that the condensate of the vapour stream removed in accordance with feature F is suitable, without extreme dilution, for treatment in a biopond or the like.

5.2.1 More particularly, the Appellants state that, in following the procedure disclosed by the patent in suit, they have obtained a condensate with a cyanide concentration of 1500 ppm, requiring an at least 1:100 dilution before being suitable for treatment in a biopond (page 6, paragraph 2, of the Statement of Grounds for the appeal).

5.2.2 According to the Respondents, the respective value measured is 367 ppm (fourth line of the extreme right-hand column in the table on page 4 of the submission dated 19.03.84). They further contend (numbered paragraphs 23 and 24 on page 7 of submission dated 05.12.84) that it is not possible to predict whether a given effluent can be treated successfully by a biological technique simply from chemical analysis for particular components. The only way to determine this is to test it, and in the present case it be a fact that the (condensate of the) vapour stream in question is sufficiently low in toxicity to permit biological purification without any detoxification steps. (Apparently this statement is to be qualified, with reference to the last but two paragraph on page 4 of the Respondents' submission dated 19.03.84, in that a 1:1 dilution prior to biological purification is necessary.)

5.2.3 In refuting of this argument, the Appellants have, during the Oral Proceedings, drawn the Board's attention

.../...

to Example 2 (pages 16\* to 17) of document (1): That Example relates to a comparative experiment in which the condensate of the vapour obtained in concentrating the bottom stream of an acetonitrile stripping column comparable to column 126 (Fig. 1) of the patent in suit is investigated as to, inter alia, its cyanide content and the dilution necessary before biological treatment. The cyanide content is established as 440 ppm - a result very similar to the 367 ppm measured by the Respondents in their experiment -, and it is stated that an at least 1:30 dilution is necessary for biological treatment. (Alternatively, as shown by Example 3, detoxification as suggested by the teaching of (2) leads to a condensate with only 3 ppm cyanide which can be subjected to biological treatment without dilution.)

- 5.2.4 Having regard to the Respondents' letter of 12.08.85, the Board has considered whether the aforementioned new argument should be taken into consideration or should be disregarded under Art. 114(2) EPC.

It is to be noted that Article 114(2) EPC, unlike Article 114(1) EPC, does not mention "arguments". It follows that relevant arguments presented for the first time in oral proceedings must be taken into account by the Board of Appeal. A party who does not attend such proceedings of its own volition after being duly summoned to do so runs the risk that new arguments will be presented in its absence and will be found convincing by the Board. It has no ground for complaint if this happens.

Any invocation of the discretionary provision of Article 114(2) EPC must in the present case fail; for there is no question of new facts or evidence having been submitted. Document (1) was cited at the very

\* page numbers here and hereinafter as renumbered in official document (1)

beginning of the opposition proceedings and again of the appeal proceedings so that its contents do not constitute new facts. All the Appellants did during the Oral Proceedings was, to draw the Board's attention to a particular portion of the document, and rely upon experimental results there given. While it would have been desirable for them to have done so at an earlier stage, they cannot be denied the right to do so at any stage. The results given in the citation are important for the evaluation of the results reported by the Respondents and, therefore, must be taken into consideration.

- 5.2.5 Having regard to the experimental results of Example 2 of (1), the Board cannot accept as plausible the Respondents' assertion that the condensate of the vapour stream removed in accordance with feature F (line 132 of Fig. 1; line 166 of Fig. 2) is sufficiently low in toxicity to permit biological purification by usual methods (no other methods being disclosed) without substantial dilution or detoxification steps. The only substance referred to by both parties in connection with the toxicity of the waste water concerned is cyanide or HCN (in whatever form). Thus, it cannot be accepted that the (virtually) same cyanide concentration should in one, but not in another case, prohibit biological treatment. The arguments of the Respondents in paragraphs 23 and 24 on page 7 of their submission dated 25.12.84 (already referred to above) are too indefinite and unsubstantiated to serve as a basis for disregarding the said experimental results of (1).

Furthermore, it must be assumed that, by not attending the Oral Proceedings, the Respondents have forgone their opportunity to present comments in response to any of the Appellants' oral contentions. Oral Proceedings may

continue in the absence of a duly summoned party (Rule 71(2) EPC), and a voluntarily absent party cannot justly complain if a decision is given against it in its absence. This Board must decide on the basis of all facts and submissions actually presented to it, and these are not such, in its judgement, that the Respondents' written submissions could outweigh the experimental results in (1) pointed out by the Appellants.

- 5.3 An alleged effect not substantiated by an Applicant or Patentee has to be disregarded in determining the object of an invention (see the Board's Decision T 20/81 "Shell/Aryloxybenzaldehydes", OJ 6/1982, page 217). Accordingly, once the suitability for biological treatment of the condensate of the vapour stream removed in accordance with feature F is disregarded, the object of the invention can only be seen in reduction of the size of the waste stream leaving the quench column 102 through conduit 106, (and of the resulting energy consumption for its concentration prior to, e.g. incineration).
6. The teaching of the claimed process is novel. Neither of the documents (3), (2) and (1), taken singly, discloses all of the features A to F of the claimed combination. Moreover, novelty is no longer in dispute, so that more detailed discussion thereof is unnecessary.
7. It must therefore be investigated whether combination of features A + B + E disclosed by (3), with features C, D (D1 or D5) and F not disclosed thereby, did or did not require an inventive step.
- 7.1 Document (2) discloses a process and apparatus whose constructional details are very similar to those of the patent in suit. In particular, it provides for water

extractive distillation in full accordance with feature C above (see Fig. 1 in conjunction with column 3, line 74, to column 4, line 35; Fig. 2 in conjunction with column 4, lines 23 et seq.), and for removal and recovery of acetonitrile in two alternative ways corresponding to features D1 and D5 above, respectively (see, respectively, Fig. 2 in conjunction with column 5, lines 43 et seq.; and Fig. 3 in conjunction with column 6, lines 20 et seq.). While the passages referred to speak of "water-miscible impurities" rather than specifically of acetonitrile, it is clear that, in essence, acetonitrile is concerned, and this is also acknowledged on pages 2 and 3 of the Respondents' submission dated 05.12.84 (see 7th horizontal column on page 2).

7.2 As (3) not only discloses that the quench system thereof (features A + E) can be combined with an "intermediate system" - consisting of an absorber (feature B) and distillation columns - which is "not critical" (column 2, lines 38 to 47), but also refers to (2) as one of three prior art systems which can be improved by incorporation of the said quench system (column 1, lines 12 et seq.), the combination of features A + B + E on the one hand with features C + D (D1 or D5) on the other hand was almost anticipated and certainly quite obvious.

7.3 It remains to be seen whether feature F as such or its combination with features A to E required an inventive step, having in mind the above object of the invention.

None of the cited documents discloses feature F. However, (1), which - as expressly stated on page 10, paragraph 3 - can be applied to all kinds of acrylonitrile processes, does disclose removal from the bottom of a column in which either acetonitrile alone or acrylonitrile, acetonitrile and hydrogen cyanide are separated from washing water

(page 10, last paragraph), of a liquid stream, which is then fed into an evaporator, the vapour from which (after condensation by heat exchange irrelevant in this context) can be subjected to biological treatment (page 11, 12th to 5th lines from the bottom), whereas a concentrated liquid is removed from the bottom of the evaporator and may be incinerated (page 11, last line, to page 12, line 2). In connection with a particular process recirculation of the said concentrated liquid, for use in an earlier step of the process - washing of the pre-cooled gaseous effluent from the reactor - is also envisaged by this citation (page 15, penultimate paragraph).

Instead, feature F of the invention relates to removal of water in the form of vapour, directly from the extraction distillation column or stripping column, near its bottom (in its lower fourth, preferably below its first tray), doing away with the detoxification column provided by (1) and with the separate evaporator thereof.

However, as the object of the invention, as far as it is to be taken into consideration, is simply reduction of the volume of the waste stream (without regard to the toxicity or otherwise of the vapour condensate), it must have been within the ambit of ordinary skill to so simplify the device of (1), direct removal of vapour being the most obvious expedient to reduce the volume of any liquid solution.

An inventive step cannot, therefore, be recognized for either feature F alone or its combination with the combined features A to E already shown to be obvious.

7.4 Accordingly, Claims 1 and 5 lack inventive step and cannot be maintained.



8. Dependent Claims 2 to 4 and 6 to 12 need not be investigated in any more detail because the Board must decide on a request as a whole. There being no auxiliary request to uphold the patent with a limited scope, e.g. with the scope of one or more of Claims 2 to 4 and 6 to 12, the patent must be totally revoked.

Order

For these reasons, it is decided :

1. The decision under appeal is set aside.
2. The European Patent is revoked.

