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D E C I S I O N
of 31 July 2003

Case Number: T 0088/99 - 3.3.1

Application Number: 92300888.2

Publication Number: 0498591

IPC: C07C 51/487

Language of the proceedings: EN

Title of invention:

Process for the production of terephthalic acid

Patentee:

E.I. DU PONT DE NEMOURS AND COMPANY

Opponent:

BP Corporation North America Inc.

Headword:

Therephthalic acid/DU PONT

Relevant legal provisions:

EPC Art. 84, 56

Keyword:

"Clarity (yes) - objections do not arise out of the amendments made"

"Inventive step (yes) - non-obvious solution of the technical problem"

Decisions cited:

-

Catchword:

-



Case Number: T 0088/99 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 31 July 2003

Appellant: E.I. DU PONT DE NEMOURS AND COMPANY
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 23 November 1998
revoking European patent No. 0498591 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: A. J. Nuss
Members: J. M. Jonk
S. C. Perryman

Summary of Facts and Submissions

I. The Appellant (Proprietor of the patent) lodged an appeal against the decision of the Opposition Division revoking the European patent No. 0 498 591 (European patent application No. 92 300 888.2), the independent Claim 1 as granted reading as follows:

"A process in which terephthalic acid is produced by oxidising para xylene to terephthalic acid in a liquid reaction medium which comprises acetic acid and in which terephthalic acid is separated from the liquid reaction medium as a crude solid, dissolved in a liquid comprising water to produce a solution and purified by a process which comprises contacting the solution under reducing conditions with hydrogen and a heterogeneous catalyst for the reduction of at least some impurities, the solution is cooled after the said reduction to precipitate solid purified terephthalic acid, aqueous mother liquor is separated from the precipitate characterised in that the aqueous mother liquor is treated to produce a less pure precipitate comprising terephthalic acid and a second mother liquor and the following steps are employed:

- (a) the less pure precipitate is returned directly or indirectly to the reaction medium; and
- (b) at least part of the said mother liquor is, directly as such or indirectly after treatment, used to dissolve the crude solid; and/or the said second mother liquor is passed to fractional distillation and treated water is recovered from the said fractional distillation and is used to

wash the precipitate recovered from the solution after the reduction step."

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

- (1) English translation of JP-A-52-128344, and
- (2) US-A-3 452 088.

III. The decision of the Opposition Division was based on Claim 1 filed with letter dated 15 July 1998 and Claims 2 to 8 as granted, Claim 1 reading as follows:

"A process in which terephthalic acid is produced by oxidising para xylene to terephthalic acid in a liquid reaction medium which comprises acetic acid and in which terephthalic acid is separated from the liquid reaction medium as a crude solid, dissolved in a liquid comprising water to produce a solution and purified by a process which comprises contacting the solution under reducing conditions with hydrogen and a heterogeneous catalyst for the reduction of at least some impurities, the solution is cooled after the said reduction to precipitate solid purified terephthalic acid, aqueous mother liquor is separated from the precipitate, the aqueous mother liquor is treated to produce a less pure precipitate comprising terephthalic acid and a second mother liquor, and the less pure precipitate is returned directly or indirectly to the reaction medium, characterised in that at least part of said second

mother liquor is, indirectly after treatment, used to dissolve the crude solid, said treatment comprising passing the second mother liquor to the fractional distillation column used for fractionally distilling the acetic acid and water evaporated from the reaction medium, and recovering treated water therefrom."

- IV. The Opposition Division held that the subject-matter of said new Claim 1 was novel, that the amendments to Claim 1 as granted met Article 123 EPC, and that the subject-matter of present Claim 1 also met the requirement of sufficiency within the meaning of Article 83 EPC.

However, it concluded that the subject-matter of the Claim 1 before it did not involve an inventive step in the light of document (1) and common general knowledge. In this context, it considered that the aim of treating the second mother liquor as obtained in the claimed process in order to dispense with the disposal of said mother liquor could be regarded as the technical problem underlying the patent in suit, and that the solution of this problem by distilling said second mother liquor in the fractional distillation column used for the fractional distillation of the acetic acid and water evaporated from the reaction medium before being used for dissolving the crude terephthalic acid crystals was obvious to the skilled person.

- V. Oral proceedings before the Board were held on 31 July 2003.

VI. The Appellant defended the patentability of the subject-matter of the patent in suit on the basis of Claim 1 submitted during the oral proceedings before the Board and Claims 2 to 7 as granted.

This new Claim 1 corresponded to the one considered by the Opposition Division, except that after "acetic acid" (line 3) the following features were inserted:

", wherein a mixture of acetic acid and water is removed from the reaction medium by evaporation, water is fractionally distilled from the mixture and acetic acid is recycled to the reaction medium,".

He argued that the technical problem underlying the patent in suit in the light of document (1) was related to the advantages achieved with respect to this closest prior art, in particular, a reduction of the amount of contaminated waste water, a reduction of the impurities in the waste water, and an improvement of the yield of fibre grade terephthalic acid. The cited prior art documents and also document

(7) GB-A-1 152 575

mentioned by him during the opposition proceedings, did not provide any incentive to its solution as claimed in present Claim 1. In particular, they did not provide any pointer to the fractional distillation of the second aqueous mother liquor, let alone to the recycling of the purified water for dissolving the crude terephthalic acid before the hydrogenation step

and the recycling of valuable materials together with the recovered acetic acid to the oxidation reactor.

- VII. The Respondent (Opponent) argued that present Claim 1 did not meet the requirements of Article 84 EPC, since by using the expression "at least part of said second mother liquor" in present Claim 1 it would not be clear to the skilled person how much of the second mother liquor was actually passed to the fractional distillation column and how much of the recovered water was actually recycled to dissolve the crude crystalline terephthalic acid.

Furthermore, he argued with respect to inventive step that, starting from the closest prior art document (1) and in view of the problematic disposal of contaminated water, the claimed process was *prima facie* obvious to the skilled person, in particular in the light of the teaching of documents (2) and (7) disclosing the use of recycled water as a solvent for dissolving the crude terephthalic acid to be subjected to the hydrogenation treatment. In support of this contention he referred to the Ashton and Horwood declarations filed 30 September 1997 and 3 October 1997, respectively. Furthermore, he submitted in this context that the Appellant did not provide any evidence substantiating the alleged advantages of the claimed invention compared to the process of document (1), and that it was clear to the skilled person that passing a very small amount of the second mother liquor to the fractional distillation and/or its feeding to the top of the fractional distillation column, i.e. at a position providing only a low number of the theoretical plates, would not lead to a relevant positive technical effect. He concluded

that for these reasons the technical problem underlying the patent in suit could only be seen in the provision of a further process for preparing terephthalic acid.

VIII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the set of Claims 1 to 7 submitted at the oral proceedings on 31 July 2003.

The Respondent requested that the appeal be dismissed.

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

Reasons for the Decision

1. The appeal is admissible.

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

2.1 Present Claim 1 is supported by the application as filed as follows:

(a) by Claim 1;

(b) by page 2, lines 18 to 22, with respect to the inserted features indicated under point VI above;
and

(c) by page 3, lines 1 to 6, page 4, lines 20 to 22, and page 5, line 36 to page 6, line 4, concerning the features indicated in the characterising part of the claim.

- 2.2 The subject-matter of present Claims 2 to 7 is supported by the originally filed Claims 2 to 7, respectively.
- 2.3 Therefore, the amended subject-matter of the present claims does not contravene Article 123(2) EPC, which only requires that no subject-matter extending beyond the application as filed is added by an amendment to a European patent or patent application.
- 2.4 Furthermore, since the process of Claim 1 as granted is restricted to
- (a) the fractional distillation of at least part of the second mother liquor together with the mixture of water and acetic acid evaporated from the reaction medium,
 - (b) the recycling of the separated acetic acid to the reaction medium, and
 - (c) the use of at least part of the water recovered from the fractional distillation column to dissolve the crude solid,

it is the Board's position that the subject-matter of the present claims does not contravene Article 123(3) EPC either.

- 2.5 In this context, the Board notes that also the Respondent did not raise an objection with respect to the admissibility of the amendments either.

3. *Clarity under Article 84 EPC*

3.1 The Respondent contended that the subject-matter of present Claim 1 was not clear in view of the expression

"at least part of the second mother liquor"

rendering it unclear how much of the second mother liquor was passed to the fractional distillation column and how much recovered water was used to dissolve the raw crystalline terephthalic acid.

3.2 In this context, the Board notes that according to the established jurisprudence of the Boards of Appeal, Article 102(3) EPC requires that amended subject-matter be examined by both instances, i.e. by the Opposition Division and by the Board of Appeal, in order to ascertain that the EPC, including Article 84, is not contravened. However, having regard to the provisions of Article 100 EPC indicating the grounds for opposition, said Article 102(3) EPC does not allow objections to be based upon Article 84 EPC if they do not arise out of the amendments made.

3.3 In the present case, the allegedly unclear passage in present Claim 1 concerns features which were already claimed in Claim 1 as granted by the wording:

"characterised in that at least part of said second mother liquor is, indirectly after treatment, used to dissolve the crude solid"

which includes a treatment of the second mother liquor by fractional distillation (via stream 20) as a mandatory feature, and a recycling to the reslurry section (via stream 30) and a purging (via stream 21) as optional features (see page 4, lines 8 to 11 of the description of the patent in suit).

3.4 Thus, in view of the above considerations, the Board concludes that present amended Claim 1 does not contravene Article 84 EPC.

4. *Novelty*

4.1 After examination of the cited prior art, the Board has reached the conclusion that the subject-matter of the present claims is novel. Since novelty was not in dispute, it is not necessary to give reasons for these findings.

5. *Inventive step*

5.1 Article 56 EPC states that an invention is held to involve an inventive step if, having regard to the state of the art (in the sense of Article 54(2) EPC), it is not obvious to a person skilled in the art.

5.2 For deciding whether or not a claimed invention meets this criterion, the Boards of Appeal consistently apply the problem and solution approach, which involves essentially 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.

- 5.3 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 the disclosure of document (1).

This document is concerned with a process for preparing terephthalic acid corresponding to that as claimed in Claim 1 of the patent in suit, except that the process of the patent in suit comprises the treatment and use of at least part of the second mother liquor as specified in present Claim 1.

- 5.4 Having regard to this closest prior art the Appellant considered that the process of present Claim 1 has essentially the advantages that the amount of process water and of contaminated waste water was reduced, that the purification expenditure of the waste water was reduced or could even be avoided, and that the yield of pure terephthalic acid was further improved.

Thus, in the light of the closest state of the art, the technical problem underlying the application in suit can be seen in the provision of a process for preparing terephthalic acid whereby these advantages are achieved (see also page 2, lines 2 to 13 and lines 43 to 51, of the patent in suit).

- 5.5 This technical problem is solved by the process as defined in Claim 1 of the patent in suit, in particular by fractionally distilling at least part of the second mother liquor, recycling the recovered acetic acid

fraction to the reaction medium, and recovering the water fraction and using it for dissolving the crude terephthalic acid solid.

Having regard to the facts (i) that the at least part of the water fraction of the second mother liquor is used for dissolving the crude terephthalic acid and therefore reduces the total amount of process water needed, (ii) that the water fraction to be purged does not contain the high boiling impurities separated in the fractional distillation column, and (iii) that the recycling of the acetic acid fraction together with the high boiling impurities comprising residual terephthalic acid and oxidation reaction intermediates as useful materials to the reaction medium of the oxidation step will credibly lead to an improvement of yield of terephthalic acid, the Board finds that this technical problem has been solved.

In this context, the Respondent submitted that passing a very small amount of the second mother liquor to the top of the fractional distillation column, i.e. at a position giving a low number of the theoretical plates, would not lead to a relevant positive technical effect. Consequently, a reformulation of the technical problem was necessary, since the above defined technical problem would not be solved within the whole scope of present Claim 1.

However, in accordance with Article 84 and Rule 29 EPC, claims define the matter for which protection is sought, whereby the essential features of the claimed subject-matter needed to achieve the desired technical effects normally determine the borders of the invention rather

than the details of the invention within those borders. It is, therefore, practically inevitable that embodiments of inventions at their borders may provide the desired technical effects at a low level. Moreover, as long as those effects have not been shown to be inexistent, which is the case here, it does not matter whether the claimed solution encompasses embodiments achieving them only to a lesser degree since, otherwise, a fair scope of protection in accordance with Article 69 EPC would not be realised. Furthermore, in view of these considerations, the Board also cannot accept the Respondent's submission since it is based on unrealistic embodiments especially construed to achieve a minimal effect, and for this reason lacks a sufficient substantiation.

- 5.6 The question now is whether the solution of the technical problem as defined above by the process of present Claim 1 would have been obvious to the skilled person in view of common general knowledge and the cited prior art.
- 5.7 As indicated above, document (1) does not provide any teaching as to how to deal with the second mother liquor obtained after recovery of the secondary impure terephthalic acid crystals to be recycled to the oxidation step (see paragraphs 2 and 3 under "3. Detailed Description of the Invention"). Therefore, this document is of no help when trying to solve the above defined technical problem.

5.8 Document (7) particularly relates to the purification of crude solid terephthalic acid, in which the crude solid terephthalic acid is converted to fibre grade terephthalic acid by dissolving the crude terephthalic acid in water and by contacting the so obtained solution under reducing conditions with hydrogen and a heterogeneous catalyst for the reduction of at least some of the impurities (see also page 1, line 70 to page 2, line 12). However, it is less relevant in assessing inventive step than document (1), since it does not provide any incentive to the skilled person to recover a secondary impure terephthalic acid precipitate within the meaning of document (1) and its recycling to the oxidation step, let alone to recover a second mother liquor resulting from the separation of said secondary impure terephthalic acid precipitate and to treat it by fractional distillation.

It is true, that document (7) discloses the recovery of purified terephthalic acid by crystallisation in a crystallisation section as illustrated in Figures 2 and 3, in which (i) the crystallisation is performed in three steps at different crystallisation rates, (ii) water is flashed off and passed into a condenser from which it is recycled via a recycle solvent drum to dissolve the crude solid terephthalic acid, (iii) the mother liquors resulting from the respective crystallisation steps are combined and discarded, and (iv) purified fibre grade terephthalic acid is recovered (see page 6, line 123 to page 7, line 78, as well as the specific disclosure with respect to the Figures 2 and 3). However, it is clear that such a recovery of fibre grade pure terephthalic acid and recycling of water has nothing to do with the claimed

solution of the technical problem underlying the patent in suit involving a fractional distillation of the so-called second mother liquor in a particular fractional distillation column and recycling the so recovered water fraction to dissolve the solid crude terephthalic acid.

- 5.9 Document (2), which is a continuation-in-part of the parent application of document (7), is essentially concerned with a method for preparing fibre grade terephthalic acid containing a reduced amount of p-toluic acid as impurity by a controlled-rate evaporative cooling using particular temperature and pressure conditions in one or more stirred crystallisation zones (see column 4, lines 1 to 74). In this context, it also discloses that the aqueous mother liquors resulting from the separation of the solid crystalline product and the subsequent washing step can be recycled, in the manner taught in the parent application (which corresponds to document (7)), to be used as solvent to dissolve the crude terephthalic acid to be purified (see column 4, line 75 to column 5, line 9). Thus, having regard to the essential features of the process of present Claim 1, this document is no more relevant in assessing inventive step than document (7).
- 5.10 Furthermore, by referring to the Ashton and Horwood declarations filed 30 September 1997 and 3 October 1997, respectively, the Respondent also contended that the process of Claim 1 of the patent in suit was *prima facie* obvious to the skilled person, since the claimed recycling and treatment of the second mother liquor were actually customary and trivial details of process

design. However, the Board cannot accept this submission, because both declarations and the Respondent's contention have been made in the knowledge of the process of the patent in suit. They are based on an unallowable *ex post facto* analysis, and not on any demonstration of how the skilled person would derive the claimed features in an obvious manner from the prior art. Moreover, the Board notes that, having regard to the fact that according to document (1) representing the closest prior art second mother liquor remained after the separation of the valuable materials to be recycled to the oxidation, the person skilled in the art would *prima facie* not consider it worthwhile to recover any more of such valuable materials therefrom, let alone to recover water by fractional distillation. Under these circumstances, he would rather consider the second mother liquor as a final aqueous waste liquid, which could be discarded or subjected to a conventional waste water treatment, such as an extraction or chemical treatment, to get rid of the impurities.

- 5.11 Thus, in view of these considerations, the Board concludes that the solution of the above defined technical problem as claimed in Claim 1 of the patent in suit is not obvious to the skilled person in the light of his common general knowledge and the cited documents, and consequently involves an inventive step in the sense of Article 56 EPC.

The dependent Claims 2 to 7 relate to particular embodiments of the process of Claim 1. They are therefore also allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The matter is remitted to the first instance with the order to maintain the patent on the basis of the set of Claims 1 to 7 submitted at the oral proceedings on 31 July 2003 and a description to be adapted thereto.

The Registrar:

The Chairman:

P. Cremona

A. Nuss