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D E C I S I O N
of 17 June 2004

Case Number: T 0996/01 - 3.3.1

Application Number: 95111625.0

Publication Number: 0696565

IPC: C07C 53/08

Language of the proceedings: EN

Title of invention:
Method of purifying acetic acid

Patentee:
DAICEL CHEMICAL INDUSTRIES, LTD.

Opponent:
BP Chemicals Ltd.

Headword:
Distillation/DAICEL

Relevant legal provisions:
EPC Art. 56

Keyword:
"All requests - inventive step (no) - obvious solution"

Decisions cited:
-

Catchword:
-

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Case Number: T 0996/01 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 17 June 2004

Appellant: (Proprietor of the patent)	DAICEL CHEMICAL INDUSTRIES, LTD. 1, Teppo-cho Sakai-shi Osaka (JP)
Representative:	Grünecker, Kinkeldey Stockmaier & Schwanhäusser Anwaltssozietät Maximilianstrasse 58 D-80538 München (DE)
Respondent: (Opponent)	BP Chemicals Ltd. Britannic House, 1 Finsbury Circus London EC2M 7BA (GB)
Representative:	Brooke, Caron BP International Limited Patents and Agreements Division Chertsey Road Sunbury-on-Thames Middlesex TW16 7LN (GB)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted 26 June 2001 revoking European patent No. 0696565 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: A. J. Nuss
Members: P. P. Bracke
S. C. Perryman

Summary of Facts and Submissions

I. The appeal lies from the Opposition Division's decision to revoke European patent No. 0 696 565 since the patent in suit did not meet the requirement of inventive step over the disclosure of *inter alia* document

(1) GB-A-1 294 432,

in combination with the common general knowledge as represented by documents

(2) Buckley et al., "Design of Distillation Column Control Systems", Chapter 8 "Minimizing Energy Requirements", pages 181-192, 1985; and

(7) Ullmann's Encyclopaedia of Industrial Chemistry, Vol. B3, Fifth Edition, 1988, VCH Verlagsgesellschaft, pages 12-10 to 12-13.

The decision was based on sets of claims defined as "Main Request" and "Auxiliary Request".

The set of claims according to "Main request" consisted of 5 claims, with the only independent claim reading:

"1. A method for purifying a crude acetic acid containing at least one component selected from the group consisting of organoiodine compounds, metalloiodine compounds, iodide ions, unsaturated compounds and carbonyl compounds as an impurity, which comprises the steps of:

a) purifying said crude acetic acid in a first distillation column operated at atmospheric or higher-pressure; and then

b) further purifying the acetic acid obtained from step (a) in a second distillation column having at least 30 plates by operating the distillation column under a reduced pressure of not less than 5.33 kPa (40 mmHg) at a reflux ratio of at least 4;

wherein overhead vapor from the first column is used as the heat source for a reboiler of the second column."

The set of claims according to "Auxiliary request" consisted of 5 claims, with the only independent claim reading:

"1. A method for purifying a crude acetic acid containing at least one component selected from the group consisting of organoiodine compounds, metalloiodine compounds, iodide ions, unsaturated compounds and carbonyl compounds as an impurity, which comprises the steps of:

a) purifying said crude acetic acid in a first distillation column operated at atmospheric or higher-pressure; and then

b) further purifying the acetic acid obtained from step (a) in a second distillation column having at least 30 plates by operating the distillation column under a pressure ranging from 5.33 to 53.3 kPa (40 to 400 mmHg) at a reflux ratio of at least 4;

wherein overhead vapor from the first column is used as the heat source for a reboiler of the second column."

II. In particular, the Opposition Division found that starting from the closest state of the art, represented by document (1), the problem underlying the present invention was the provision of an energy-saving economical purification method of acetic acid. Since the use of the overhead heat of a distillation column as reboiler heat for another distillation column was known from documents (2) and (7), the claimed method was obvious over the cited prior art.

III. With letter of 12 May 2004, the Appellant (Proprietor of the patent) filed sets of claims according to a Second and a Third Auxiliary Request.

The Second Auxiliary Request consisted of 5 claims, with the only independent claim reading:

"1. A method for purifying a crude acetic acid obtained by carbonylation of methanol, said crude acetic acid comprising at least one component selected from the group consisting of organoiodine compounds, metalloiodine compounds iodide ions, unsaturated compounds and carbonyl compounds as an impurity, wherein the method comprises the steps of:

a) purifying said crude acetic acid in a first distillation column operated at atmospheric or higher-pressure; and then

b) further purifying the acetic acid obtained from step (a) in a second distillation column having at least 30 plates by operating the distillation column under a pressure ranging from 5.33 to 53.3 kPa (40 to 400 mmHg) at a reflux ratio of at least 4;

wherein overhead vapor from the first column is used as the heat source for a reboiler of the second column."

The Third Auxiliary Request consisted of 5 claims, with the only independent claim reading:

"1. A method for purifying a crude acetic acid obtained by carbonylation of methanol, said crude acetic acid comprising at least one component selected from the group consisting of organoiodine compounds, metalloiodine compounds iodide ions, unsaturated compounds and carbonyl compounds as an impurity, wherein the method comprises the steps of:

a) purifying said crude acetic acid in a first distillation column operated at atmospheric or higher-pressure; and then

b) further purifying the acetic acid, which is obtained in step (a) by removing a heavy fraction from said acetic acid, in a second distillation column having at least 30 plates by operating the distillation column under a pressure ranging from 5.33 to 53.3 kPa (40 to 400 mmHg) at a reflux ratio of at least 4;

wherein overhead vapor from the first column is used as the heat source for a reboiler of the second column."

Moreover, at the oral proceedings, which took place on 17 June 2004, the Appellant filed a set of five claims according to the Fourth Auxiliary Request, with the only independent claim reading:

"1. A method for purifying a crude acetic acid obtained by carbonylation of methanol, said crude acetic acid comprising at least one component selected from the group consisting of organoiodine compounds, metalloiodine compounds iodide ions, unsaturated compounds and carbonyl compounds as an impurity, wherein the method comprises the steps of:

a) purifying said crude acetic acid in a first distillation column operated at atmospheric or higher-pressure; then

(a1) leading overhead steam output obtained in step (a) into a condenser and condensing there; and then

b) further purifying the acetic acid, which is obtained in step (a1) steam, in a second distillation column having at least 30 plates by operating the distillation column under a pressure ranging from 5.33 to 53.3 kPa (40 to 400 mmHg) at a reflux ratio of at least 4;

wherein the overhead vapor from the first column is used as the heat source for a reboiler of the second column."

IV. The Appellant accepted that document (1) represented the closest state of the art and that starting therefrom the problem underlying the invention could be seen in providing an energy-saving economical method

for purifying acetic acid. However, since the claimed method could not be arrived at by combining the teaching of document (1) with the teaching of any of the cited prior art documents, the Appellant was of the opinion that the claimed method was not obviously derivable from that prior art.

V. The Respondent essentially argued that it was known from documents (2) and (7) to use overhead heat from a distillation column as the heat source for a reboiler of another column. Therefore, the claimed method was obviously derivable from the teaching of document (1) with any of the teachings of documents (2) and (7).

VI. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the requests defined as "Main Request" or as "Auxiliary Request" in the decision under appeal, or the sets of claims filed as Second and Third Auxiliary Requests with letter of 12 May 2004 or the set of claims filed as Fourth Auxiliary Request in the oral proceedings on 17 June 2004.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. *Article 123(2) EPC and novelty*

Since the Board came to the conclusion that none of the requests meets the requirement of inventive step, it is

not necessary to give any reasoning as to whether the requirements of Article 123(2) EPC and of novelty are met.

3. *Inventive step*

3.1 Main request

3.1.1 In accordance with the “problem-solution approach” applied by the Boards of Appeal to assess inventive step on an objective basis, it is in particular necessary to establish the closest state of the art forming the starting point, to determine in the light thereof the technical problem which the invention addresses and solves and to examine the obviousness of the claimed solution to this problem in view of the state of the art.

3.1.2 It was not contested that document (1), which is referred to on page 2, lines 35 to 40, of the patent in suit, represented the closest state of the art.

Document (1) discloses a method of purifying carboxylic acids containing halogen or halogen-containing contaminant by introducing the contaminated stream into a first distillation zone intermediate the ends thereof, removing a product stream from an upper part of the first distillation zone and introducing this product stream into a second distillation zone intermediate the ends thereof, removing a carboxylic acid product stream from the lower part of the second distillation zone, this carboxylic acid product stream being substantially free of the halogen or halogen-containing contaminant, and removing a fraction containing the contaminant from

the upper part of the second distillation zone (page 1, lines 46 to 63). Although atmospheric, superatmospheric and subatmospheric pressures may be used in both distillation zones (see page 3, lines 20 to 25) it is also stated on page 3, lines 31 to 36, that for purifying acetic acid the two distillation zones are usually operated at atmospheric or slightly superatmospheric pressure.

According to page 2, lines 41 to 46, of the patent in suit, such known method has the disadvantage that a considerably large amount of thermal energy is to be consumed.

Thus, starting from the disclosure in document (1), the problem underlying the invention can be seen in providing an **energy-saving** economical method of purifying acetic acid.

3.1.3 The patent in suit claims to solve this problem by the method in Claim 1, which differs essentially from the method disclosed in document (1) by using overhead vapor from the first column as the heat source for a reboiler of the second column and by operating the first distillation column at atmospheric or higher-pressure and the second distillation column at reduced pressure.

3.1.4 Since the Board came to the conclusion that in the light of the teachings of the cited documents a skilled person seeking to solve the above-mentioned problem would have arrived at the method of Claim 1 in an obvious way, it is not necessary to give any reasoning as to whether it has been made plausible that this

problem has been effectively solved by all methods embraced within Claim 1.

3.1.5 Using the overhead vapor from a first distillation column as the heat source for a reboiler of a second distillation column was generally known.

Indeed, document (2) mentions such energy recovery in the introductory part on page 181 and specifically discloses in Figure 8.3 a design of two distillation columns wherein the overhead heat of the first distillation column is used as the heat source for the reboiler of a second distillation column.

Moreover, document (7), which is concerned with methods of increasing energy efficiency of distillation, teaches in the first paragraph of the left-hand column on page 12-13 that the thermodynamic efficiency may be increased by repeated use of a given quantity of energy and discloses in Figure 9 a series of distillation columns, each at a different **decreasing** pressure, wherein all the heat is applied to the first column and its overhead vapors are condensed by reboiling the next column.

Thus, starting from the method for purifying acetic acid disclosed in document (1), a skilled person looking for saving energy would have recovered the overhead heat of the first distillation column for reboiling the next column and he would have chosen a lower pressure in the second distillation column than in the first distillation column.

3.1.6 The Appellant contested that a skilled person would have taken the disclosures of documents (2) and (7) into consideration, since document (1) only discloses a distillation scheme wherein the overhead stream of the first distillation column is further purified in a second distillation column, whereas in Fig. 8.3 of document (2) the bottom streams are further purified and Fig. 9 of document (7) concerns a series of distillation columns for fractionating the same feed in a parallel arrangement.

However, in the present case, the skilled person is necessarily one with a background in distillation technology and with knowledge of the different methods for saving energy in distillation methods. As the origin of the heat energy is immaterial for its use in a reboiler, a skilled person looking for saving energy in the distillation method disclosed in document (1) would not only consider methods for saving energy in distillation systems designed specifically for the further purification of the overhead stream, as the one disclosed in document (1), but would consider any disclosure concerning recovery of heat of any distillation column. Such skilled person would thus clearly take documents (2) and (7) into consideration.

3.1.7 The Appellant also submitted that the claimed method had the additional unexpected advantage that the amount of acetic acid anhydride produced as by-product in the second distillation column is reduced when reducing the pressure in the second column, resulting in a higher yield of acetic acid.

As support of this submission the Appellant referred to the experimental data provided with letter of 31 October 2001 and presenting the amount of acetic acid anhydride after 90 minutes, 2, 3 or 4 hours boiling at 760, 650, 400 and 100 mmHg. The Appellant alleged that with those experimental data, obtained by changing the pressure and the heating time of the second distillation column and determining the amount of formed acetic acid anhydride per hour by gas chromatograph, it was shown that the amount of acetic acid anhydride produced as by-product in the second distillation column was reduced when reducing the pressure in the second column.

However, it is generally known that the boiling temperature of a chemical fluid decreases with reduced pressure and that the chemical degradation, i.e. the formation of undesired compounds, decreases with reduced temperature.

Since those experimental data thus only confirm what could be expected, such data are not suitable for showing any unexpected effect.

3.1.8 Therefore, Claim 1 and, thus, the main request cannot be considered to meet the requirement of inventive step.

3.2 Auxiliary Request

3.2.1 Claim 1 of the Auxiliary Request only differs from Claim 1 of the Main Request in that the upper limit of the pressure range in the second distillation column is restricted to 53.3 kPa (400 mmHg).

3.2.2 The Appellant submitted that it had been shown in the examples of the patent in suit that a significant effect of saving energy is obtained when operating the second distillation column at a pressure of 53,3 kPa (400 mmHg).

However, since in the sole example in the patent in suit according to the invention only a distillation is described wherein the second distillation column is operated at 53,3 kPa (400 mmHg), in the absence of any proper comparison, it has not been made plausible in that example that an unexpected effect would have been obtained by operating the second distillation column at a pressure of 53,3 kPa (400 mmHg).

3.2.3 Moreover, the Appellant submitted that it followed from the experimental data provided with letter of 31 October 2001 that a more significant decrease of the formation of acetic acid anhydride could be observed at pressures of not more than 53,3 kPa (400 mmHg) than at 760 and 650 mmHg.

This submission is based on a figure obtained by plotting the rate of decrease of anhydride formation against pressure. Since, however, it may not be unambiguously derived from that plot that the gradient of the curve below 400 mmHg is steeper than between 760 and 650 mmHg, as submitted by the Respondent and not contested by the Appellant, the experimental data provided with letter of 31 October 2001 are not suitable for showing an effect for selecting 400 mmHg as the upper limit of the pressure range.

3.2.4 Since, thus, the selection of the upper limit of the pressure range does not result in an additional technical effect, Claim 1 and, thus, the Auxiliary Request cannot be considered to meet the requirement of inventive step for the reasons given for the Main Request.

3.3 Second Auxiliary Request

The method in Claim 1 of the Second Auxiliary Request is further specified by the fact that the crude acetic acid is obtained by carbonylation of methanol.

Since the distillation method described in document (1) was specifically designed for purifying carboxylic acids obtained by the reaction of alcohols and carbon monoxide (see page 1, lines 14 to 31), the Second Auxiliary Request cannot be considered to meet the requirement of inventive step for the reasons given for the Main Request and the Auxiliary Request.

3.4 Third Auxiliary Request

In comparison with the Second Auxiliary Request, the method of Claim 1 is further specified by the fact that a heavy fraction is removed from acetic acid in the first distillation column.

Since in the first distillation column described in document (1) a light fraction is removed from an upper part thereof, a heavy fraction is necessarily removed in the lower part of that first distillation column.

Thus, the Third Auxiliary Request cannot be considered to meet the requirement of inventive step for the reasons given for the Main Request and the Auxiliary Request.

3.5 Fourth Auxiliary Request

In comparison with the Second Auxiliary Request, the method of Claim 1 is further specified by the fact that the overhead steam output in step a) is lead into a condenser and condensing there.

Since, however, the condensation of the overhead stream of the first distillation column is inherent to the use of the overhead vapor from the first distillation column as the heat source for a reboiler of the second column, Claim 1 and thus the fourth auxiliary request cannot be considered to meet the requirement of inventive step for the reasons given for the Main Request.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

N. Maslin A. Nuss