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**Datasheet for the decision
of 2 March 2016**

Case Number: T 2520/12 - 3.3.05

Application Number: 06753354.7

Publication Number: 1907319

IPC: C01B31/00, B01D53/14, C01B31/20

Language of the proceedings: EN

Title of invention:

A METHOD FOR RECOVERY OF HIGH PURITY CARBON DIOXIDE FROM A
GASEOUS SOURCE COMPRISING NITROGEN COMPOUNDS

Patent Proprietor:

Union Engineering A/S

Opponents:

BASF SE
Siemens Aktiengesellschaft

Headword:

Relevant legal provisions:

EPC Art. 100(a), 54, 56

Keyword:

Novelty - (yes)

Inventive step - (yes)

Decisions cited:

T 0210/93, T 0684/02, T 2215/08

Catchword:



Beschwerdekammern
Boards of Appeal
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Case Number: T 2520/12 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 2 March 2016

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Decision under appeal:

**Interlocutory decision of the Opposition
Division of the European Patent Office posted on
25 September 2012 concerning maintenance of the
European Patent No.1907319 in amended form.**

Composition of the Board:

Chairman	H. Engl
Members:	G. Glod
	C. Vallet

Summary of Facts and Submissions

- I. The appeals lie from the interlocutory decision of the opposition division in which it found that European patent EP-B-1 907 319 in amended form, based on auxiliary request 3, met the requirements of the EPC.

The documents cited in the decision are:

D1: US 3 266 220 A
D3: EP 1 059 110 A1
D8: EP 1 159 056 B1

- II. Independent claim 1 of the granted patent reads as follows:

"1. A method for recovery of high purity carbon dioxide from a gaseous source comprising the steps of:

- a. feeding a gas (G1) comprising carbon dioxide, oxygen, and nitrogen compounds into an absorption column (A1),*
- b. absorbing the gas (G1) in an alkanolamine-containing absorbing agent, by which the gas (G1) is separated into a carbon dioxide-depleted gas (G2) and a carbon dioxide-rich liquid (L1),*
- c. pressurising and heating the liquid (L1) obtained in step b in order to provide the liquid (L2),*
- d. separating by means of flashing the liquid (L2) obtained in step c into a NO_x- and oxygen-rich gas (G3) and a NO_x- and oxygen-depleted liquid leaving the flash column (A3),*
- e. pressurising the liquid leaving the flash column (A3) in step d in order to provide the liquid (L3),*
- f. separating the liquid (L3) obtained in step e into a carbon dioxide-rich gas (G4) and a carbon dioxide-depleted liquid (L4) by means of stripping, and*
- g. purifying the gas (G4) obtained in step f in order to*

produce high purity carbon dioxide, which is substantially free of nitrogen oxides."

Independent claim 7 of the granted patent reads as follows:

"7. Use of a method according to any of the claims 1 to 6 for the production of high purity carbon dioxide."

- III. The board issued a communication pursuant to Article 15(1) RPBA in which it gave its provisional opinion that the subject-matter of the claims of the main request was novel and inventive.
- IV. Oral proceedings took place on 2 March 2016.
- V. The arguments of appellant I (patent proprietor) can be summarised as follows:

Novelty

D3 disclosed all features of claim 1 with the exception of steps c) and d). The conclusion of the opposition division that a pump inevitably built up pressure in a system was refuted, since this would not apply to any system, but only to a system where the receiving end or downstream end was closed or semi-closed. The pump would inevitably pressurise L2 only if the pressure in the flash column was higher than the pressure of the liquid L1 entering the pump. This was not the case in D3, where the pressure in the flash tank was kept subatmospheric by means of a vacuum pump.

It was not energetically efficient to increase the pressure prior to producing a vacuum.

Even if some phase separation occurred in the heat exchanger 120, the removal of oxygen from the absorbent could only occur in the flash tank.

According to claim 1, the flashing of step d) took place under the conditions where L2 had been provided, i.e. a stream having higher pressure and temperature than liquid L1.

The mere presence in D3 of the pump between the absorption column and the flash tank was not a clear and unambiguous disclosure of the stream 7 in D3 being pressurised.

The indications concerning the pressure present in claim 2 would be understood by the skilled person as bar(g) and not as bar(a).

Therefore, novelty had to be recognised.

Inventive step

Starting from D3 as closest prior art, the problem to be solved was to prevent the loss of carbon dioxide in the flash column and at the same time effectuate the efficient removal of oxygen and nitrogen oxides from the liquid in the flash column.

None of the prior art documents was concerned with the removal of oxygen and nitrogen oxides from the carbon dioxide stream while providing high purity carbon dioxide at a high yield. Neither D3 nor D1 taught an increase in pressure of the liquid stream leaving the absorption column. Rather, the pressure in the flash tank was reduced, relative to the pressure applied in

the absorption column. D8 was not directed to producing high purity carbon dioxide.

VI. The arguments of the appellant II (opponent I) can be summarised as follows:

Novelty

To fulfill the claim requirement of the "pressurising" feature, it was sufficient that the liquid L2 had a higher pressure after the pump than the liquid L1 before the pump. This was the case for all pumps.

D3 disclosed that the gas entering the absorption column had a pressure of 14.7 psia (column 4, line 7), which was equivalent to atmospheric pressure. It was also disclosed that in the flash tank the pressure was reduced from above atmospheric pressure to subatmospheric pressure (column 5, lines 11 to 13). This meant that the pressure had to be increased in between.

It was evident from D3 that oxygen was only released from the absorbent in the flash tank. However, the carbon dioxide loaded absorbent 101 was heated so that the pressure of the dissolved gaseous components increased. In order to avoid the release of these gaseous components prior to entering the flash tank, the liquid must be kept at an elevated pressure. This pressure increase was obtained by pump 8.

Inventive step

The problem of minimising the loss of carbon dioxide in the flash column while efficiently removing oxygen and nitrogen oxides from the liquid in the flash column was not solved. In the least, the minimisation of the carbon

dioxide loss was dependent on the process conditions that were not defined. The features of claim 1 were not sufficient to solve the problem posed.

In D3, depressurisation in the flash tank caused some carbon dioxide to be released, which meant that in D3, the main part of the easier desorbing gases was already released in the flash tank. In addition, there was no evidence in the form of comparative examples showing that a more efficient removal of nitrogen oxides was obtained. It was also implausible that pressurisation was driving the equilibrium of $1/2 O_2 + NO \leftrightarrow NO_2$ towards the left. The distribution of the components between the gas and liquid phase was dependent on the gas pressure and the liquid temperature after depressurisation. Such indications were not present in claim 1.

The pressure in the flash column according to the patent was higher than in the flash column of D3. This required more heating of the absorbent to obtain the same result as in D3.

The objective problem to be solved could only be seen as providing an alternative process. The process of claim 1 was within the generic disclosure of D3 so that a similar effect to that in D3 was to be expected.

In addition, D8 taught that the pressure of the absorbent leaving the absorption column could be pressurised prior to the flash tank (paragraph 21 and example 3).

An inventive step could not therefore be recognised.

VII. Opponent II is a party to the appeal proceedings as of right, but did not submit any comments.

VIII. Requests

Appellant I requested that the decision of the opposition division be set aside and the patent be maintained as granted. Alternatively, it requested that the patent be maintained in amended form based on one of auxiliary requests 1 to 3, all submitted by letter on 2 February 2016.

Appellant II requested that the decision of the opposition division be set aside and the patent be revoked in its entirety.

Reasons for the Decision

1. Article 100(a) EPC - Novelty

1.1 The board comes to the conclusion that the subject-matter of the claims of the patent as granted is novel for the reasons given below. This reasoning applies to all claims, since claims 2 to 6 are dependent on claim 1 and claim 7 relates to the use of a method according to claim 1. The "use of a method" is nothing but the very same method (T 210/93 (Reasons 3.2.3), T 684/02 (Reasons 5.6), T 2215/08 (Reasons 7.3)).

1.1.1 D3 (EP-A-1 059 110) discloses (figure 1 and column 4, line 2 to column 6, line 7) a process in which lean feed gas 1 is passed to compressor or blower 2 wherein it is compressed to a pressure generally within the range of 14.7 to 30 pounds per square inch absolute (psia).

Compressed lean feed gas 3 is passed from blower 2 into the lower portion of absorption column 4. Typically, absorbent 6 is introduced into the upper portion of absorption column 4. Absorbent 6 is a fluid comprising at least one alkanolamine species. Within absorption column 4 the lean feed gas rises in countercurrent flow against the downflowing absorbent. As the feed gas rises, most of the carbon dioxide, oxygen, and small amounts of other species such as nitrogen that it contains, are absorbed by the downflowing absorbent, resulting in a carbon dioxide depleted top vapour exiting at the top of column 4 and a carbon dioxide loaded absorbent containing dissolved oxygen leaving the bottom of column 4. The top vapour is withdrawn from the upper portion of column 4 in stream 5 and the carbon dioxide loaded absorbent is withdrawn from the lower portion of column 4 in stream 7.

Stream 7 is then passed to liquid pump 8 and from there, in stream 9, to a first heat exchanger 120 where it is heated by indirect heat exchange. The resulting heated carbon dioxide loaded absorbent undergoes deoxygenation carried out by depressurization in flash tank 102. To this effect, heated carbon dioxide loaded absorbent 101 is passed from first heat exchanger 120 to flash tank 102 where its pressure is reduced from above atmospheric pressure to subatmospheric pressure, due to vacuum pump 104. As a consequence of this depressurization, dissolved oxygen is released from the absorbent. The resulting oxygen depleted carbon dioxide loaded absorbent is withdrawn from flash tank 102 by stream 106, passed to liquid pump 107 and from there, via stream 108, to a second heat exchanger 121 wherein it is further heated by indirect heat exchange. The further heated oxygen depleted carbon dioxide loaded absorbent is passed from second heated exchanger 121, via stream

11, into the upper portion of stripping column 12, wherein carbon dioxide within the absorbent is stripped from the alkanolamine solution into upflowing vapor, which is generally steam, to produce carbon dioxide top vapor and the remaining alkanolamine absorbent. The carbon dioxide top vapour is withdrawn from the upper portion of stripping column 12 in stream 13 and passed through reflux condenser 47, wherein it is partially condensed. The resulting stream 14 is passed to reflux drum or phase separator 15, wherein it is separated into carbon dioxide gas and into condensate. The carbon dioxide gas is removed from phase separator 15, in stream 16, and recovered as carbon dioxide product fluid, having a carbon dioxide concentration generally within the range of 95 to 99.9 mole percent on a dry basis.

- 1.1.2 It is not under dispute that steps a), b), e), f) and g) of claim 1 of the patent are disclosed in D3 in combination. The point of debate is whether in the process of D3, stream 7 is pressurised by liquid pump 8.
- 1.1.3 In the board's view, the skilled person reading claim 1 of the patent in suit understands that the liquid (L1) that exits the absorption column is pressurised and heated in order to provide the liquid (L2). This liquid L2, having increased temperature and pressure compared to L1, is then flashed (depressurised).

This means that in D3 it is not the pressure of the liquid entering the liquid pump 8 that has to be compared to the pressure of the liquid exiting this pump, but the pressure of the liquid exiting the absorption column that needs to be compared with the pressure of the liquid entering the flash tank 102.

- 1.1.4 In D3 the lean feed gas has a pressure of 14.7 to 30 psia before passing to the absorption column, which is above atmospheric pressure. In the flash tank the pressure is reduced from above atmospheric pressure to subatmospheric pressure. From these pressure indications it cannot be concluded that a pressurisation of the absorbent necessarily had to take place between the exit of the absorbing column and the entry of the flash tank, since it is also conceivable that the absorbent leaving the absorption column already had a pressure of above 14.7 psia, which is above atmospheric pressure. The presence of the liquid pump does not necessarily mean that a pressure increase takes place. The pump could simply ensure that the liquid is continuously transported from the absorption column through the heat exchanger to the flash tank without loss of pressure due to, for example, friction in the pipe.
- 1.1.5 The heating of the absorbent in the heat exchanger 120 could possibly lead to the release of some oxygen and carbon dioxide prior to entering the flash tank. This is not excluded by the teaching of D3, since it does not indicate that the dissolved oxygen is only released in the flash tank. The depressurisation in the flash tank leads to the release of oxygen from the absorbent, but it is not excluded that the release of some happened before. Therefore, an increase in pressure is not implied by the description of the process of D3.
- 1.1.6 Furthermore, the pressure difference in the flash tank is obtained via the vacuum pump, so an increase in pressure prior to entering the flash tank is not mandatory. It would even be energetically unfavourable to first increase the pressure and produce a vacuum afterwards. For the depressurisation it is sufficient to rely on the vacuum only.

- 1.1.7 Reference was made to the passage at column 5, lines 1 to 7, of D3:

"Stream 7 is passed to liquid pump 8 and from there in stream 9 to and through first heat exchanger 120 wherein it is heated by indirect heat exchange[...]. The resulting heated carbon dioxide loaded absorbent undergoes deoxygenation."

Here mention is made explicitly to a heating step and that the resulting absorbent is heated, but nothing is said about changes in pressure. The board does not agree with the argument that there was no need to mention a pressurising step because it was implicit and evident, since as explained above, other meaningful interpretations of the role of the pump are equally possible.

- 1.1.8 The board concludes that D3 neither explicitly nor implicitly discloses that stream 9 has an increased pressure as compared to stream 7 and that a pressurisation of the liquid exiting the absorption column is not directly and unambiguously derivable from D3.

2. Article 100(a) EPC - Inventive step

The board comes to the conclusion that the subject-matter of the claims of the patent as granted involves an inventive step for the following reasons.

2.1 Invention

The invention concerns a method for recovering high purity carbon dioxide from a gaseous source.

2.2 Closest prior art

D3 can be considered as closest prior art since it also relates to the recovery of carbon dioxide (see above point 1.1.1 and D3: paragraph 1 and column 6, lines 3 to 7).

2.3 Problem

According to the patent in suit, the problem to be solved is to provide an easier and cost effective method for the provision of high purity carbon dioxide, which is substantially free of nitrogen oxides (paragraphs 12 and 15).

2.4 Solution

As a solution to this problem, the patent proposes a method according to claim 1 characterised by the fact that the liquid exiting the absorption column is pressurised prior to entering the flash column.

2.5 Success of the solution

The process described in paragraphs 41 to 49 of the patent is a process falling within the scope of claim 1. The liquid L1 leaving the absorption column has a pressure of 1.02 bar, while the liquid L2, entering the flash column, has a pressure of 2 bar and contains 0.4 mole ppm of O₂, 0.7 mole ppm of NO₂ and 0.1 mole ppm of NO. The liquid L3 entering the stripper contains 0.01 mole ppm of O₂, while NO₂ and NO are not detectable. This means that in the flash tank, under the conditions indicated, the nitrogen oxides are removed from the liquid below the detection limit, while oxygen is

substantially removed.

It is plausible that this removal of nitrogen oxides and oxygen has a beneficial effect on the purification of the gas G4 leaving the stripper for the production of high purity carbon dioxide.

The overall process and especially the removal of nitrogen oxides and oxygen in the flash tank is dependent on the process conditions such as pressure and temperature differences between the different process steps. In that context it is to be noted that step d) of the process according to claim 1 is formulated such that the separation by means of flashing the liquid L2 leads to a NO_x- and oxygen-rich gas (G3) and a NO_x- and oxygen-depleted liquid. This requires the process conditions to be such that a meaningful separation takes place. There is no evidence, apart from speculation, that the problem was not solved when steps a) to g) were followed according to claim 1 of the patent at different process conditions than those indicated in the patent (see Table paragraph 41).

Moreover, D3 does not indicate the removal of nitrogen oxides during the flashing step. The depressurisation is said to cause at least 50 percent of the oxygen dissolved in the absorbent to be released (column 5, lines 17 to 20). The oxygen depleted carbon dioxide loaded absorbent is said to contain preferably less than 0.5 ppm oxygen (column 5, lines 27 to 29). These values do not establish that the removal of oxygen and nitrogen in D3 is at the same level as in the process of claim 1. There is no evidence that the process of D3, not having the pressurising step, but having a flash tank under vacuum, obtains the same removal of NO and O₂ in the flash column.

The argument that the conditions present in claim 2 of the patent, such as pressures of 0.1 bar, would not solve the problem is not accepted, since claim 2 must be interpreted to refer not to the absolute pressure, but to the gauge pressure.

Therefore, due to a lack of convincing evidence, the board does not agree with the argument of appellant II on whom the burden of proving that the problem was not solved over the whole range lies.

The board therefore accepts that the problem is solved.

2.6 Obviousness

It has to be decided whether or not the proposed solution can be derived from the state of the art in an obvious manner.

- 2.6.1 D1 relates to an improved process for the removal of carbon dioxide from a gaseous mixture of hydrocarbons and/or other non-acidic constituents containing carbon dioxide by the use of a selective solvent consisting essentially of at least one ether of a carbonitrile (column 1, lines 12 to 16). D2 is not concerned with the production of high purity carbon dioxide.

The feed gas is fed into an absorber, wherein carbon dioxide is absorbed from the feed mixture into the absorbent (column 4, lines 64 and 65; column 5, lines 1 to 4). The rich absorbent, containing absorbed carbon dioxide, is withdrawn from the absorber. Then, the rich absorbent undergoes a controlled pressure reduction, by being passed successively through an expansion valve and a heat exchanger into the flash chamber (column 5, lines

16 to 21). D1 does not teach the pressurisation of the liquid exiting the absorption column.

- 2.6.2 As already indicated above, D3 does not mention the removal of nitrogen oxide in the flash tank and the benefit of such a removal for the subsequent purification of carbon dioxide. In addition, D3 relies on the vacuum in the flash tank for depressurising the absorbent.

D3 does not mention the increase in pressure of the absorbent entering the flash tank compared to the pressure at the exit of the absorption column. D3 does not give any incentive to increase the pressure of the absorbent prior to entering the flash tank since this would be in conflict with the creation of a vacuum in the flash tank. It is not credible that the skilled person would have contemplated the increase of pressure prior to the flash tank for increasing the depressurisation. The conditions indicated in D3 already lead to some loss of carbon dioxide (column 5, lines 22 to 24). Further pressure changes would increase this loss. In addition, it would be energetically unfavourable to increase the pressure prior to applying a vacuum.

- 2.6.3 D8 does not relate to the recovery of high purity carbon dioxide but to the removal and recovery of carbon dioxide from exhaust gas for deposition (page 2, lines 12 to 15). Therefore, the removal of nitrogen oxides and oxygen from carbon dioxide is not of relevance.

The exhaust gas is fed to an absorber containing a chemical absorbent where the carbon dioxide is absorbed. This absorbent is further fed to a desorber to remove carbon dioxide and discharge it (claim 1). The absorbent

can be pressurised after leaving the absorption unit (page 3, lines 56 and 57). However, the goal of the desorption unit is to remove carbon dioxide from the absorbent, while the goal of the flash tank in D3 is to remove mainly oxygen from the absorbent. Applying the teaching of D8 to D3 would mean that the carbon dioxide would be almost completely removed in the flash tank, which runs against the object of the process of D3. The process of D8 is completely different and the board cannot recognise why the skilled person would consider D8 when trying to solve the problem posed.

2.6.4 In summary, none of the prior art documents provides a teaching which would lead to the proposed solution.

2.7 This reasoning also applies to claims 2 to 6 that are dependent on claim 1 and to claim 7 that relates to the use of a method according to claim 1 (see point 1.1 above).

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The patent is maintained as granted.

The Registrar:

The Chairman:



C. Vodz

H. Engl

Decision electronically authenticated