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D E C I S I O N
of 31 January 1995

Case Number: T 1093/92 - 3.2.3

Application Number: 85308312.9

Publication Number: 0183446

IPC: F25J 3/04

Language of the proceedings: EN

Title of invention:
Nitrogen generation

Patentee:
UNION CARBIDE CORPORATION

Opponent:
LINDE AG, Wiesbaden

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (yes)"

Decisions cited:
-

Catchword:
-

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Case Number: T 1093/92 - 3.2.3

D E C I S I O N
of the Technical Board of Appeal 3.2.3
of 31 January 1995

Appellant: (Proprietor of the patent)
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Representative:
-

Decision under appeal:
Decision of the Opposition Division of the European Patent Office dated 23 September 1992, issued in writing on 13 October 1992, revoking European patent No. 0 183 446 pursuant to Article 102(1) EPC.

Composition of the Board:

Chairman: C. T. Wilson
Members: J. du Pouget de Nadaillac
L. C. Mancini

Summary of Facts and Submissions

I. The present appeal is directed against the decision of the Opposition Division dated 23 September 1992, issued in writing on 13 October 1992, revoking European patent No. 0 183 446 (based on European application No. 85 308 312.9).

In its grounds, the Opposition Division held that the subject-matters of Claims 1 of the main and subsidiary requests, amended with respect to granted Claim 1, either lack novelty or do not involve an inventive step. Documents D1 and D2, hereinafter listed, are mentioned in the contested decision.

II. The Appellant (Proprietor) filed the appeal on 14 December 1992 and paid the appeal fee on the same date. The Statement of the Grounds was received on 8 February 1993, accompanied by four sets of claims, as main and auxiliary requests, together with new pages of the description. A correction is requested for page 4 at line 50 of the description, where "about" should read "above".

III. Oral proceeding took place on 31 January 1995. During these proceedings, the wording of Claim 1 of the main request was amended so as to take account of objections under Articles 84 and 123 EPC raised by the Respondent (Opponent). Finally, the Appellant filed a single new set of claims, together with new pages of description and drawings.

IV. The single independent claim of the set of claims reads as follows:

"1. A process for the production of nitrogen at relatively high yield and purity by cryogenic rectification of feed air comprising avoiding the need to employ a nitrogen recycle stream by the steps of:-

(1) compressing the entire feed air to a pressure greater than the operating pressure of a rectification column which is operating at a pressure in the range of from 241 to 1000 kPa (from 35 to 145 psi), and wherein feed air is separated into nitrogen-rich vapour and oxygen-enriched liquid;

(2) expanding a major portion of the feed air to the operating pressure of the column and introducing it into the column at a point at least one tray above the bottom of the column where the oxygen-enriched liquid collects, the major portion comprising from 55 to 90 per cent of the feed air;

(3) condensing a minor portion of the feed air, at a pressure of from 69 to 621 kPa (from 10 to 90 psi) greater than that at which the column is operating, by indirect heat exchange within the column with oxygen-enriched liquid in the bottom of the column, the minor portion comprising from 10 to 45 percent of the feed air;

(4) introducing the resulting condensed minor portion of the feed air into the column at an intermediate point and at a point at least one tray above the point where the major portion of the feed air is introduced into the column;

(5) condensing a first portion of the nitrogen-rich vapour by indirect heat exchange with vaporizing oxygen-enriched liquid;

(6) passing at least some of the resulting condensed nitrogen-rich portion to the column at a point at least one try above the point where the minor portion of the feed air is introduced into the column;

(7) recovering substantially the entire remaining second portion of the nitrogen-rich vapour as product nitrogen;

with refrigeration being provided by said expansion of the major portion of the feed air prior to introduction into the column; and precluding recycle of any portion of the product nitrogen or of any waste gases produced by the process back to the distillation column."

V. During the appeal proceedings, following prior art documents cited either in the examination or in the opposition proceedings were considered:

D1: The Russian technical magazine "KISLOROD", No. 2, 1959, pages 9-14, accompanied by a translation into the German language.

D2: DE-A-3 035 844

E1: US-A-4 464 188

E2: US-A-4 382 366

E3: US-A-3 518 839

VI. The Appellant submitted the following arguments:

Document E1 is the state of the art closest to the present invention, since it aims at obtaining nitrogen gas with high efficiency. However, the process disclosed in this document needs nitrogen recycle streams. By contrast, the present invention avoids recycling nitrogen by using for the refrigeration needs a major portion of the feed air before its fractionation and that contributes to increase the nitrogen recovery. The other cited prior art documents relate to processes, which are not comparable, since they produce different

products or the same product, however in another state. In this technical field, each category of production process has its own specific conditions, which cannot automatically be applied in another category. Document D1 aims at producing liquid nitrogen, and moreover at a low yield, which is the opposite to the object of the present invention. The production of liquid nitrogen requires much higher power input than the production of gaseous nitrogen. Document D2 indeed describes a process which produces oxygen and nitrogen, but this last is only a by-product used for providing refrigeration and, then, discharged as waste stream. It cannot provide a hint to the present invention, which uses for the same purpose a feed air portion. In this prior art, moreover, the boiler of the fractionating column is outside the column, when in the present invention it is inside, so that a vapour with a higher nitrogen content is obtained increasing also the nitrogen yield. Documents E1 and E2, which aim at producing other air products, do not teach to use the major portion of the feed air for refrigeration purposes.

VII. The Respondent contested an inventive step of the claimed invention by arguing as follows:

Claim 1, as worded, does not exclude other refrigeration sources than the one claimed and, in this respect, the description of the patent in suit mentions the compression of the minor air feed. Thus, the compression step of Claim 1 is not necessarily linked to the following refrigeration step and can therefore be any kind of cómpression.

Document D2 may be seen as the closest prior art. Its classification is the same as the one of the present invention and, in the process disclosed in this

document, nitrogen is produced in addition to oxygen. As soon as products are wanted, the aim is to produce them at relatively high purity and high recovery. Moreover the object of document D2, namely to obtain a relatively pure oxygen, results in the production of a relatively pure nitrogen at the top of the column. The purity and yield values reached in the present invention are quite usual in this technical field and most of the steps mentioned in the contested Claim 1 can be found in the process according to Document D2. Thus, this document is very close.

Two distinguishing features are to be seen in Claim 1: the boiler inside the column and the expansion of the major portion of the feed air for the refrigeration need of the process. With respect to the first difference, it appears clearly for the person skilled in the art that the teaching of document D2 is not restricted to boilers outside the column, even when this arrangement is shown in the drawings, which is essentially done only because it is easier to draw it so. All technical books teach that it is quite equivalent to have the boiler outside or inside the column, considering the process itself. The choice is only made according to the room available in the bottom of the column. In both cases, the vapour product is in equilibrium with the liquid.

When, as is the case with the present invention, it is wished to get rid of the nitrogen recycle streams in a process according to document D2, it is in any way necessary to find another refrigeration source. At the output of the column, the oxygen stream is at a too low pressure to be used therefor. Thus, what remains is the feed air, and the skilled person sees immediately that only the major stream can provide sufficient refrigeration. Therefore, the skilled person was in a one-way street situation and the present invention must

be considered as obvious, especially as it is well known to use a feed air portion for this object as shown by documents E1 and E2.

Alternatively, if it is supposed that Document E1 constitutes the closest prior art, the sole difference is mainly to be seen in the expansion step, which, as seen above, is obvious. It is then well within the ability of the skilled person to determine the relative proportions of the major and minor feed air streams disclosed in document D1, so that the needed refrigeration is obtained.

VIII. The Appellant requested the decision under appeal be set aside and the patent be maintained on the basis of the amended description, claims and figures filed at oral proceedings.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. New Claim 1 is a combination of granted Claims 1 and 3. Moreover, this claim contains additional features, such as:

- a more precise indication of the introduction points into the column of both air portions, the major portion being introduced "at a point at least one tray above the bottom of the column" and the minor portion" at a point at least one tray above the point of introduction of the major portion";

- the arrangement of the boiler inside the column and the pressure at which the condensation of the minor portion occurs, namely the "pressure of from 69 to 621 Kpa (from 10 to 90 psi) greater than that at which the column is operating".

Moreover, the last advantage mentioned at the end of Claim 1 is clarified, in that the claimed process precludes substantial recycling of any portion of the product nitrogen or of any waste gases produced by the process.

All these amendments are supported either by the description or by the drawings. In particular, the description of the patent in suit clearly indicates that each line 13, 14 or 15 in Figure 1 represents at least one tray, and the first line 13 in this figure is located just above the condenser 10, thus just above the sump of the column. This point was no longer disputed by the Respondent during the oral proceedings.

The description has been amended in order to comply with the new claims and to acknowledge prior art documents D1 and D2.

Hence, the new claims and description comply with the requirements of Article 123(2) and (3) EPC.

3. Neither of the cited prior art documents describes a process comprising all the steps according to Claim 1, so that this process is new. In particular, step (1) of present Claim 1 distinguishes clearly the claimed process from the process according to document D2, which, according to the contested decision, destroyed the novelty of the process of Claim 1 of the main and first subsidiary requests filed during the opposition proceedings.

4. *Closest prior art*

As indicated by Claim 1 the present invention relates to a process for the production of nitrogen gas at relatively high yield and purity. Therefore, the Board takes the view that document E1, in preference to document D2, represents the closest prior art:

The process described in document E1 is directed to air separation and more particularly to the recovery of pressurized, substantially pure gaseous nitrogen. The aim of this prior art is to realize said separation with a high efficiency, in particular by reducing the power consumption. The example disclosed in this prior art indicates that about 88% of the nitrogen processed by this known system is recovered as gaseous nitrogen at high pressure and at 2 ppm oxygen purity.

Document D2, in contrast, discloses an air separation process for obtaining oxygen of average purity, namely from 42 to 52 percent purity. Nitrogen gas indeed is also produced, but only as by-product: As soon as it is withdrawn from the rectification column, it is spilt into two approximately equal streams. One stream is used as power means for the air compression and the other stream is expanded to provide refrigeration of the column and is then rejected. Thus, at least 50 percent of the produced nitrogen is lost. The object of this prior art as well as its results are therefore further removed from the present invention than are those of document E1.

5. In the process according to document E1, the incoming air is first compressed to a pressure of about 4,5 bar and then split into two unequal portions. The relative proportions of these streams are not mentioned, but they can be deduced from the single example of this prior

art, which indicates a minor portion comprising about 7% of the feed air. The major feed air portion is cooled and introduced into the single rectification column "at a point at least one tray above the bottom of the column", as can be seen from the figures. Said column operates at a pressure of about 4 bar. The minor air portion undergoes another compression step in a supplemental compressor to reach a pressure of about 7,5 bar, that is to say a pressure of 3,5 bar greater than that of the column, and then reboils the sump of the column in the way according to step (3) of the contested Claim 1. Afterwards, being sub-cooled outside of the column and flashed, this minor air portion is reintroduced into the rectification column in the way mentioned in step (4) of said Claim 1. In this known process, also the steps (5) to (7) of said claim are carried out.

6. From above, it can be seen that, in the process according to the closest prior art, step (1) of the contested Claim 1 is per se fulfilled, but only a slight pressure difference exits between the compression and column pressures, corresponding to the usual pressure losses due to the flow resistances, mainly through the heat exchangers which are disposed between the compressor and the column. In the process according to Claim 1 of the patent in suit, however, step (2) calls for an expansion of a feed air portion and therefore implies that the compression of the feed air according to step (1) should be carried out at a higher pressure, that is to say a pressure which allows for an expansion of the air such that the refrigeration of the system is provided, as required at the end of Claim 1. Thus, the process according to document E1 comprises all the steps of Claim 1, apart from step (2) with its consequence on step (1). Another difference is to be seen in the given proportions of the major and minor portions of the feed

air (see steps (2) and (3) of Claim 1). The new features are therefore the expansion of the major feed air portion together with the necessary air compression and the proportions between both air portions, the minor portion according to the present invention being about 10 to 45 percent of the feed air compared to the 7 percent of the prior art. Furthermore, it should be remembered that, in the process according to E1, the minor feed air portion undergoes a particular compression before its introduction into the column sump, and a sub-cooling step before its subsequent introduction at an intermediate point of the column. These steps are not required by Claim 1.

7. In document E1, a recovery of about 88% of the total nitrogen with a purity up to 98 mole percent is given. The patent in suit (page 4, lines 55 to 61) mentions a nitrogen yield up to about 82% with a purity of at least 98 mole percent. Thus, no true difference can be seen regarding these results and, consequently, one advantage of the present invention mentioned by the Appellant, namely an increase of the nitrogen recovery, cannot be taken into account.

However, the air separation according to document E1 requires a complicated and costly system, since, downstream of the separation column, two nitrogen streams are recycled in order to reboil part of the column and to provide the necessary refrigeration, before being introduced into the top of the column as reflux. Therefore, starting from the above mentioned closest prior art, the objective problem underlying the present invention is to be seen in that a process is created which can produce nitrogen at relatively high purity and relatively high yield, even while avoiding

the need to employ nitrogen recycle streams. This last modification reduces the complication and, thus, the costs of the whole system.

This process is solved in the present invention in that the feed air is compressed at a pressure allowing the source of refrigeration to be provided by the expansion of a major portion of the feed air prior to the introduction of said air into the rectification column, said major portion comprising 55 to 90 percent and the minor portion 10 to 45 percent of the feed air.

8. It has now to be assessed whether inventive step was necessary to arrive at the process defined in Claim 1 when starting from document E1.

The introductory part of document E1 and the passage on column 6, lines 10 to 14, show that several possibilities for providing the refrigeration need of an air separation process were already known. Usually, when the process is directed to the production of one of both air dominant constituents, namely nitrogen or oxygen, then the other one is used as waste product and expanded to produce refrigeration. In the case of nitrogen production, as suggested in column 6 of document E1, the oxygen vapour at the top of the column condenser could have been used and technical books show that this technique is the most employed. The expansion of the feed air to provide refrigeration is also mentioned in document E1 as a prior art attempt, however for the production of oxygen and with a two pressure rectification column. In view of all these possibilities, the one-way street situation put forward by the Respondent for the solution of the present invention is not confirmed.

9. One teaching of the closest prior art according to E1, see column 6, lines 3 to 9, is also that the use of nitrogen recycle streams for the refrigeration need is advantageous in that the air pressure for the whole system is set by the pressure drop downstream of the column. Thus, a reduced pressure level is required, which helps to economize on the power needed for the process. As seen above, the pressure difference between the compressor and the column in this known process is small. Thus, by increasing this difference, the present invention goes against the teaching of document E1.

10. Document D1 teaches to expand a portion of about 50% of the feed air prior to its introduction into a distillation column in order to provide the refrigeration need. However, the process described in this document is directed to produce liquid nitrogen at a relatively low yield. In this technical field, it is well known that for the production of a product in a liquid form much higher pressures than for gaseous products are needed, since not only the cold due to the environment and exchange losses are to be covered, but also the refrigeration required for the production of liquids rather than gases. The refrigeration need is multiplied by at least twenty, and even more when liquid nitrogen is wanted. For this reason, in the process according to document D1, the feed air is in the first step compressed to a pressure of about 190 to 200 bar. Since the fractionating column operates at a pressure of about 4 bar, the feed air has to be expanded before being introduced into the column and, in such a case, it is usual to utilize this expansion for refrigeration purposes, as is shown by the technical books.

Moreover, it is clearly indicate in this prior art that, when gaseous nitrogen is wanted, a particular apparatus has to be added to the distillation system in order to

vaporize the nitrogen. In this process, further, the expanded feed air is introduced into the sump of the column, whereas in the present invention, it is introduced at a point above the sump.

Thus, the basic object and the process conditions of such a prior art system are quite different from those of the present invention. The high pressure, which is used, requires a great power. Therefore, the person skilled in the art, starting from the process known from E1 which provides nitrogen at a reduced power requirement, would not even consider this document D1.

11. Document D2 is not relevant regarding the solution of the present invention. Its teaching is quite similar to that of document E1 and, more specifically, nitrogen from the column, here also, is expanded to produce refrigeration for the system.

12. In document E2, an oxygen producing air separation system is disclosed, in which a single stage rectification column operating at a pressure of 3,6 bar is used. High purity oxygen is removed from the column at a point above the sump, while at the top of the column an oxygen-containing nitrogen waste stream is recovered, which is then combusted with fuel to provide the power for the compressor. In this process, the feed air is first compressed to 11 bar, cooled in heat exchangers and then split into "side" and "remaining" streams. The side stream is passed through an expansion turbine to produce refrigeration and then introduced at an intermediate point of the rectification column. The other stream, after passing through a valve, is condensed in a reboiler located in the bottom of the column and then introduced into the top of the column as reflux.

This prior art, as seen above, relates to the production of oxygen and it is a mixture of oxygen and nitrogen which is recovered at the top of the column. Therefore, the person skilled in the art, searching to improve a process for the production of nitrogen gas at high purity and at high recovery like the process known from document E1, would not have his attention drawn towards document E2. Furthermore, in view of the fact that, in the process known from E2, the air portion which reboils the column is not the minor portion and is not introduced at an intermediate point of the column as defined in Claim 1 but is used as reflux, the process according to document E2 on the one hand and the process according to Claim 1 on the other hand cannot be compared with each other. Moreover, the problem underlying the process according to document E2, i.e. to utilize by-products or waste streams to provide energy, is nearly the same as the one underlying the closest prior art known from document E1 and is therefore in complete contrast to that underlying the present invention. No hint is given in this prior art to use the same means for providing refrigeration in a process designed for producing nitrogen. Thus, the person skilled in the art has no reason to consider the process known from document E2.

13. The comments given in respect of document E2 are equally valid with regard to document E3, which briefly describes an identical schematic flow scheme of a single column for separating air into enriched streams of oxygen and nitrogen. Since, here also, an air stream is discharged into the top of the column as reflux liquid, only a mixture of nitrogen and oxygen is removed from the top of said column. Moreover, this prior art essentially deals with an improvement of the crosscurrent heat exchanger for cleaning the feed air upstream of the fractioning column.

14. Summarizing, none of the cited documents provides an incentive to the solution of Claim 1. Thus, the subject-matter of this claim implies an inventive step within the meaning of Article 56 EPC and is patentable within the meaning of Article 52 EPC. Claims 2 to 9 concern particular embodiments of the process according to Claim 1 and thus are not open to objection.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent with the documents submitted at the oral proceedings. In accordance with the request made by the Appellant in his Statement of Grounds, the word "about" on page 4, line 50, of the description should be corrected into "above".

The Registrar: The Chairman:

N. Maslin C. T. Wilson