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D E C I S I O N
of 12 February 2003

Case Number: T 0816/99 - 3.3.5

Application Number: 93115162.5

Publication Number: 0589406

IPC: B01D 53/047

Language of the proceedings: EN

Title of invention:

02 VSA process with low 02 capacity adsorbents

Patentee:

AIR PRODUCTS AND CHEMICALS, INC.

Opponent:

Praxair Technology, Inc.

Headword:

VSA process/AIR PRODUCTS

Relevant legal provisions:

EPC Art. 54(3), 56

Keyword:

"Novelty: yes - allegedly inherent properties of prior art product not convincingly established"

"Inventive step: yes (after amendment) - non-obvious alternative"

Decisions cited:

-

Catchword:

-



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Boards of Appeal

Chambres de recours

Case Number: T 0816/99 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 12 February 2003

Appellant: Praxair Technology, Inc.
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Decision under appeal: Interlocutory decision of the Opposition Division
of the European Patent Office posted 1 June 1999
concerning maintenance of European patent
No. 0 589 406 in amended form.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: B. P. Czech
M. B. Günzel

Summary of Facts and Submissions

I. The appeal is from the interlocutory decision of the opposition division to maintain European patent 0 589 406 in amended form. The independent claim 1 underlying that decision reads as follows (the sole amendment to claim 1 carried out before the opposition division being **highlighted**):

"1. The process of selectively adsorbing nitrogen from a gas mixture containing nitrogen and at least one less strongly adsorbed other gas component, comprising; contacting the gas mixture in an adsorption zone with an adsorbent selective for nitrogen, selectively adsorbing nitrogen on the adsorbent and passing the gas mixture less the adsorbed nitrogen out of the zone, wherein the adsorbent zone comprises an adsorbent with an isothermal nitrogen working capacity of **between 0.4 and 0.6** mmol/g and a binary isothermal working selectivity of at least 17 mmol N₂/mmol O₂ but whose nitrogen working capacity at any given working selectivity (S) does not exceed the value: $0.0667 \times (S) - 0.667$, for a gas mixture feed at 23°C and 146.9 kPa and evacuation at 20.3 kPa."

II. Nine printed publications had been cited during the opposition proceedings, including the following:

D1: US-A-4 859 217;

D3: R.T. Yang "Gas separation by Adsorption Processes", Butterworths Series in Chemical Engineering, 1987, pages 251 and 252;

D8: EP-A-0 486 384.

In the contested decision, the opposition division considered *inter alia* D1, D3 and D8 and held that the subject-matter of the claims as amended was novel and inventive.

III. In its statement of grounds of appeal, the appellant *inter alia* submitted that the subject-matter of claim 1 of the amended patent as maintained by the opposition division still lacked novelty and inventive step over D8, and was obvious over D3. In support of its objection, the appellant filed a further document, namely

D10: Coe C.G. et al., "Molecularly Engineered, High-Performance Adsorbent: Self-Bound Low-Silica X Zeolite" in Perspectives in Molecular Sieve Science; ACS Symposium Series 368, Washington, US, 1988, pages 478 to 491

and a test report referring to a graph labelled "Exhibit 2".

IV. With its reply, the respondent filed an amended set of claims as new main request. Claim 1 thereof has the same wording as the claim 1 previously filed (see point I here above), but with the following additional features being appended thereto:

"wherein said adsorbent comprises an active adsorbent phase and an inert diluent, said active adsorbent phase being a zeolite selected from the group consisting of A-zeolite, X-zeolite, Y-zeolite, chabazite, mordenite and mixtures thereof, the zeolite further being ion exchanged with lithium to at least approximately 50%."

It submitted *inter alia*

- that the subject-matter of amended claim 1 was novel over the disclosure of D8;
- that D1 was to be considered as the closest prior art for assessing inventive step; and
- that the process of claim 1 was inventive in particular over the disclosures of D1, D3, D8 and combinations thereof.

V. The parties were summoned to oral proceedings.

VI. In a further letter, the appellant argued that the subject-matter of amended claim 1 still lacked an inventive step in view of, *inter alia*, a combination of D1 with D3 or of D3 with D1.

Additionally, the appellant filed a further document, namely

D11: Baksh M.S. et al., "Lithium Type X Zeolite as a Superior Sorbent for Air Separation", Separation Science and Technology, 27(3), March 1992, pages 277 to 294,

together with a declaration of Mr Latus, a test report, and a plot of some data of D1. In its view, the material submitted, when viewed in conjunction with other prior art on file, showed that PSA air separation with the lithium exchanged zeolites referred to in D11 was nothing else than the process of claim 1.

VII. With letter dated 15 January 2003, the respondent filed five amended sets of claims as first to fifth auxiliary requests. Claim 1 according to the second auxiliary request has the same wording as claim 1 according to

the new main request (see point IV here above), but with the following additional features relating to the zeolite adsorbent being appended thereto:

"and is ion exchanged with a second ion to approximately 5% to 50%."

In this letter and in a further letter of 22 January 2003, the respondent contested the relevance of D11 and argued that D11 and the experimental results presented by the appellant should be disregarded due to the belatedness of their filing. It questioned the exact nature of the zeolite material actually used in D11 and contested the conclusions drawn by the appellant from the data disclosed in D11 and from its own experimental data. With another letter dated 3 February 2003, the respondent filed further experimental data "in support" of its auxiliary requests.

VIII. With telefax dated 7 February 2003, the appellant filed a further document, namely

D12: EP-A-0 548 755.

On the basis of D12, it objected to the novelty of the claimed subject-matter.

IX. With its letter dated 11 February 2003, the respondent requested that D12 be disregarded for being filed belatedly and for not being *prima facie* relevant.

X. Oral proceedings took place on 12 February 2003.

In the course of the oral proceedings, the respondent

- dropped its first auxiliary request on file and replaced it by the second auxiliary request on file;

- expressly confirmed that the term diluent as used in claim 1 did not necessarily designate a binder; and
- stated that there was no material on file showing that zeolithes exchanged with lithium and a further cation belonged to the state of the art according to Article 54(2) EPC.

The appellant

- maintained its novelty objections based on D11 and D12;
- stated that D12 was the result of a search conducted to find prior art more relevant for the claims according to the auxiliary requests;
- referred to US-A-5 152 813 claiming the same priority as D12, ie of application US 811,404 of 20 December 1991, to support its assumption that the priority of D12 was valid;
- referred to page 2, lines 33 to 36 of D12 when questioned by the Board about the meaning of the term "LSX" as used in D12;
- considered the subject-matter of the claims according to new first auxiliary request to lack inventive step in view of the fact that the zeolites relied upon belonged to the prior art; and,
- when questioned accordingly by the Board, had no further objection to these claims.

XI. The parties' oral and written submissions, as far as they are relevant for the present decision, can be summarised as follows:

Concerning D12, the appellant essentially argued that since the Li,Ca-LSX-zeolites disclosed in Example 8 thereof had the same composition (85% lithium and 15% calcium exchanged) and the same nitrogen working capacity (far right column and second footnote of Table IV-A of D12) as the ones of Table III of the contested patent, they must inherently have the same properties, in particular the same O₂ working capacity and the same working selectivity of about 25. Moreover, they were prepared in the same manner and were both of the LSX-type, ie they both had a Si:Al ratio of exactly 1. Dilution did not affect the selectivity of the material, and the nitrogen working capacity of the adsorbent of Example 8 comprising 51% binder (0,54 mmol/g) could be obtained by linear interpolation from the data for such zeolites as given in Table III of the patent. Therefore, the disclosure in D12 of an air separation process making use of the adsorbent of Example 8 comprising 51% inert binder was novelty-destroying for the subject-matter of the respective claims 1 of all the requests on file for the designated states DE, FR and GB.

The respondent contested this evaluation of D12 and argued that even though Example 8 of D12 disclosed an Li,Ca-LSX zeolite, this indication of its composition did not necessarily mean that the properties thereof would be identical to the ones of the Li,Ca-LSX zeolite described in the contested patent. In particular, the zeolite disclosed in D12 did not necessarily have the same "history" in terms of preparation, handling and activation. Exhibit 2 provided by the appellant showed that the addition of binder had an impact on selectivity of the zeolite tested. There was not necessarily a linear relationship between the nitrogen working capacity of the undiluted zeolite and the degree of dilution. Since an absolute identity of the diluted zeolite of D12 and the one of the contested

patent in terms of Si:Al ratio, activity, nitrogen working and capacity had not been established, the zeolite diluted with 51% binder as referred to in Example 8 of D12 did not necessarily have a working capacity and a working selectivity meeting the criteria set out in claim 1.

Concerning the obviousness of the subject-matter of the claims according to the main request, the appellant *inter alia* argued that the process of claim 1 lacked the required inventive step in view of a combination of D1 with D3. D1 disclosed the use of very high capacity and very high selectivity zeolite exchanged to 99% with lithium as adsorbents for PSA air separation. The person skilled in the art, confronted with temperature problems occurring when carrying out of the process of D1, would obviously add to the adsorbent an economically optimised amount of a diluent according to the teaching of D3, in order to overcome the said problems. It would inevitably arrive at a process of optimised efficiency wherein the nitrogen working capacity and working selectivity of the composite adsorbent would also lie within the prescribed ranges.

The respondent argued that the skilled person, starting from the process disclosed in D1 as closest prior art, would not consider the addition of diluent, which lowers the working capacity of the adsorbent, as an obvious measure for improving the overall efficiency of the process. Referring *inter alia* to document D1, it argued that the person skilled in the art

- would not consider adding a diluent in view of the trend in the art to produce binder-free adsorbents in order to maximise working capacity and selectivity

- would not consider giving away some of the desirable very high nitrogen working capacity achieved by the use of the specific lithium exchanged adsorbents, and
- would not, therefore, consider the teaching of D3 at all, since the latter related to the use of conventional zeolitic adsorbents of relatively low capacity and selectivity.

Concerning the claims of the first auxiliary request

(i) the appellant argued that since

- zeolites as referred to in claim 1 were known as expressly acknowledged in the contested patent;
- similar zeolites exchanged with two different cations were also known from eg D1, column 2, lines 14 to 17; and
- zeolithes exchanged with lithium and a further cation did not necessarily perform better as shown by Figure 5 of the contested patent, the use of the particular zeolites in the otherwise obvious process of claim 1 according to the main request could not justify the presence of an inventive step; and

(ii) the respondent argued

- that there was no reference on file which disclosed zeolites as referred to in claim 1;
- that the acknowledgement, in the contested patent, of Li,Ca-LSX zeolites as prior art might merely refer to the internal knowledge of the person that drafted the application; and

- that in view of the fact that such zeolites were not known and performed better than known ones their use in a process of the type claimed could not be considered as obvious.

XII. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

As main request, the respondent requested that the decision under appeal be set aside and that the patent be maintained with the claims of the main request filed with letter dated 18 February 2000.

As first auxiliary request the respondent requested that the patent be maintained with the claims of the 2nd auxiliary request filed with letter dated 15 January 2003.

As second to fourth auxiliary requests the respondent requested to maintain the patent with the claims of any of the third to fifth auxiliary requests filed with letter dated 15 January 2003, taken in their numerical order.

Reasons for the Decision

Main request

1. *Novelty*

The Board is convinced that the arguments provided in support of the objections to the novelty of the subject-matter of claim 1 as raised by the appellant on the basis of documents D11 and D12 (concerning the latter, see also point 4 here below) are not conclusive. Nor does the Board consider the disclosure

of any of the other documents cited during the appeal proceedings as novelty-destroying. In particular, D8 does not disclose the use of lithium exchanged zeolites. Since the main request fails for another reason (see point 2 below) this matter need not be further pursued.

2. *Inventive step*

2.1 Closest prior art

2.1.1 As acknowledged by the respondent, document D1 is concerned with a PSA process using highly lithium exchanged X-zeolite and "the same type of active adsorbent is used as in the patent", see page 6, second paragraph of the respondent's letter dated 18 February 2000. The Board therefore concurs with the respondent in that D1 can be considered to represent the closest prior art for the purpose of assessing inventive step.

2.1.2 More particularly, D1 discloses the use of a 99% lithium-exchanged low silica X-zeolite (Si:Al ratio of 1) in powder form labelled "2.0 LiX (99%)" as adsorbent in the PSA separation of nitrogen from gaseous mixtures, in particular from air. See in particular D1, column 1, "Field of the invention", column 2 "Summary of the invention", column 3, line 25 to 61, column 5, lines 2 to 9, the two tables in column 9, column 10, lines 11 to 31 and claims 1 to 5. As pointed out by the appellant in its letter dated 20 December 2002, page 4, last paragraph, the parties agree in that the selectivity of the "2.0 LiX (99%)" powder material disclosed in D1 also has a selectivity of about 25. This is also in accordance with Example 5, Figure 5 and Table III of the patent, where a highly lithium exchanged low silica X-zeolite powder ("LiLSX") is shown to have a binary working selectivity of about 25.

D1 does not disclose the purposive addition of a binder or another type of inert diluent to the "2.0 LiX (99%)" powder sample. Moreover, according to the submission made by the parties during the opposition proceedings, the N₂ working capacity of this material is around 1 mmol/g. Hence D1 does not disclose a composite adsorbent comprising a X-zeolite and an inert diluent and fulfilling the condition specified in claim 1 concerning the N₂ working selectivity.

2.2 Technical problem

According to the patent in suit, the prior art air separation processes using the very high nitrogen capacity materials described eg in D1 have not been able to take full advantage of the very high selectivities of these materials, see page 3, lines 33 to 43. The technical problem to be solved by the claimed process can be considered to consist in (further) improving the overall efficiency, in terms of power consumption and recovery of the non-nitrogen component, of a process for the separation of nitrogen from a gas mixture making use of high capacity and high selectivity lithium exchanged zeolitic adsorbents, such as disclosed in D1, see page 3, lines 43 to 50 of the contested patent.

2.3 The solution

- 2.3.1 The solution proposed by present claim 1 consists in the addition of an inert diluent in order to reduce the N₂ working capacity of the (composite) adsorbent material to values within the specified range while maintaining a very high selectivity of the active (zeolitic) adsorbent, the zeolite used having a

selectivity which is high enough to assure that the relationship between the N₂ working capacity and the binary working selectivity of the (composite) adsorbent as specified in claim 1 is fulfilled.

2.3.2 The Board finds it plausible, and it has not been disputed, that the solution claimed may, at least under certain circumstances, lead to an improved overall efficiency of such a process. In particular, it is plausible that a reduction in efficiency in a commercial PSA or VSA unit caused by heat effects as addressed on page 10, lines 31 to 39 of the patent in suit can be overcome by means of the addition of an optimised amount of an inert diluent.

2.4 Obviousness of the solution

2.4.1 Document D1 clearly states that a good PSA air separation adsorbent should have a high nitrogen working capacity ("delta loadings") **and** a high nitrogen over oxygen selectivity, see column 10, lines 33 to 42. The skilled person would thus consider the "2.0 LiX (99%)" material disclosed in D1 as a very good choice. In putting such a process into practice, the skilled person would obviously take into account the technical knowledge available in the field of PSA air separation using zeolites.

2.4.2 Document D3 is a review-type publication illustrating this knowledge. It generally relates to gas separation by adsorption processes (see title). The pages 251 and 252 filed by the appellant more specifically relate to "PSA: Principles and Processes", as indicated in the page header. The passage from page 251, second full paragraph to page 252, first paragraph addresses thermal effects occurring during an industrial-scale PSA separation of air by means of 5A-zeolite. The said passage has to be read in connection with the

immediately preceding three sentences on page 251. These sentences clearly set out the considerations that led to the concept of adding inert diluents for smoothing temperature variations, the inert additives increasing the bed size and the heat capacity of the bed without decreasing the productivity of the sorbent. Although the said three sentences relate to the separation of another type of gas mixture, the concept set out there is again referred to in the last sentence of the first paragraph on page 252 as a solution to the limitations of the separation due to temperature variations occurring in industrial scale PSA air separation with 5A zeolite, see Figure 7.12 and its legend on page 251 in connection with page 252, lines 6 to 7).

2.4.3 Hence, D3 clearly addresses the problem of heat effects occurring in particular in industrial-scale PSA air separations. Although according to D3 a conventional 5A-zeolite was used as adsorbent, ie a zeolite not having a particularly high capacity and selectivity, a detrimental effect of the observed temperature variations on the efficiency of the separation has been identified. A person skilled in the art, trying to put into practice the process of D1 and optimise its overall efficiency, had good reasons to consider the disclosure of document D3. It would consider the measures suggested by D3, ie the addition of an inert diluent, which according to claim 1 does not necessarily need to be a "binder", as an obvious solution of the stated technical problem, in particular when designing a large scale PSA or VSA process.

2.4.4 In doing so, the skilled person would obviously add the diluent in an amount which corresponds to the inherent optimum value that can be reached, in terms of minimised heat effects and maximised process efficiency, for a given zeolite, as recommended by D3

on page 251, lines 12 to 13. For a given zeolite adsorbent, the optimum amount of a specific diluent correlates with the optimum N₂ working capacity of the composite adsorbent. Hence, the Board holds that the skilled person, applying the teaching of D3 to the process and adsorbent as disclosed in D1 will inevitably arrive at a process according to present claim 1, ie wherein the adsorbent has a N₂ working capacity in the prescribed range.

2.4.5 The further arguments provided by the appellant cannot alter the view of the Board even if it were to be accepted, for the sake of argument, that it was the credo of the skilled workers at the priority date to "increase working capacity to increase selectivity" and that there was a general trend of using adsorbents with ever increasing selectivities **and** capacities, and in particular without the use of inert and hence diluting binders. It appears to be common ground that, in principle, it is desirable for an adsorbent for gas separations to have **both** a high capacity and a high selectivity. See eg D1, column 10, lines 33 to 37, or D8, page 2, lines 18 to 21. However, in the Board's judgment, all these considerations cannot keep the skilled person from taking special measures as disclosed in D3 when confronted with the known problem of heat effects that may occur during real-life, ie more adiabatic PSA separations, in contrast with laboratory-scale separations. Since according to D3 problems based on heat effects may even occur with 5A-zeolithes, ie with zeolites of relatively low capacities, similar or worse problems could actually be expected when using high capacity adsorbents. It is clear to the skilled person that the measure suggested in D3, ie the use of an inert diluent, reduces the working capacity but does not affect the selectivity of the adsorbent. This measure stands for a trade-off between capacity and selectivity, said trade-off being

necessary to take fuller advantage of the very high selectivity and thereby obtain an optimum overall efficiency. The fact that D3 does not address high capacity adsorbents is thus simply not relevant and cannot keep the skilled person from applying its teaching to the process and high selectivity adsorbents of D1.

- 2.5 Therefore, the method of claim 1 according to the main request is found not to be based on an inventive step. Consequently, the main request cannot be allowed.

First auxiliary request

3. *Admissibility of the amendments*

Basis for the present claim 1 can be found in claims 1,4,5,7,9 and 10 as granted (claims 1,4,5,7,9 and 10, and page 5, lines 21 to 22 of the application as filed). The Board is therefore satisfied, and it has not been challenged, that the restricting amendments to the claims satisfy the requirements of Articles 123(2) and (3) EPC.

4. *Novelty*

- 4.1 As will immediately appear from the following, the relevance of document D12 is *prima facie* so high that it potentially represented an obstacle to the maintenance of the patent in suit. Hence the Board decided to take it into consideration despite of its belated submission.

- 4.2 D12 is a patent application of the respondent published before (30 June 1993) the date of filing of the patent in suit (21 September 1993) but after the priority date of the latter (23 September 1992). The date of filing of D12 (15 December 1992) is later than the priority

date of the patent in suit, but the priority date (20 December 1991) of D12 is earlier than the priority date of the patent in suit.

- 4.3 The Board is satisfied, and it has not been challenged, that the priority claimed by the contested patent is valid. See in particular claims 1,4,5,7,9 and 10, page 5, lines 21 to 22 and the examples of the priority application US950093. Hence, the subject-matter disclosed in D12 belongs to the state of the art according to Article 54(3) EPC, as far as it was also disclosed in the application of which D12 claims the priority, ie in the US application 811404 of 1991.

The contents of the US application 811404 as filed have not been made available to the Board up to the taking of the present decision. Hence, it has not been established with absolute certainty whether or not Example 8 of D12, which was cited against the novelty of one of the alternative processes covered by present claim 1, was already contained in the said US application as filed. Assuming for the sake of argument that it was present, the Board holds that D12 is, nevertheless, not detrimental to the novelty of the subject-matter of present claim 1 for the following technical reasons:

- 4.4 The disclosure of D12
- 4.4.1 In its Example 8, D12 *inter alia* discloses the use of an adsorbent comprising an "85% lithium, calcium LSX zeolite" and 51% of an inert binder. Concerning the zeolite, reference is made to sample #1 of Table IV-A on page 10 of D12, where more data are provided concerning the composition and the properties of this particular LSX zeolite. It can be gathered from the column at the right of Table IV-A, from the second footnote of the table, from the third sentence of the

paragraph underneath the table, and from page 9, line 50 that the zeolite of sample #1 is in powder form and exchanged with 85% lithium and with 15% calcium, and has an isothermal nitrogen working capacity of 1.10 mmol/g from 0.2 to 1.2 atm at 23°C. D12 neither expressly mentions the isothermal oxygen working capacity nor the working selectivity of the adsorbent of Example 8 diluted with 51% inert binder. Hence, the question that needs to be answered is whether the indications given in Example 8 and Table IV-A of D1 provide a clear and unambiguous disclosure of a binder-diluted LSX-zeolite adsorbent inherently having all of the properties required by present claim 1.

4.4.2 The method for measuring the isothermal nitrogen capacity of the adsorbent as indicated in the footnote to Table IV-A of D12 is identical with the one used according to the contested patent, see eg page 9, lines 53 to 54. In view of the data given in Table III of the contested patent the Board can also accept, at least for the sake of argument, that the N₂ working capacity of the adsorbents referred to in this table appears to be a linear function of the degree of their dilution. Moreover, the Board can accept that, in principle, the properties of a given adsorbent are determined by its exact composition and structure.

On the other hand, it can be gathered from the appellant's own submission that the addition of a binder may influence the working selectivity of a given exchanged zeolite. According to the graph shown in Exhibit 2 filed with the substantiation of the appeal, the working selectivity of a specific binderless zeolite powder differs by about two units from the one of beads made from the powder and comprising 30% inert binder. The argument of the appellant according to which the test results reported in Exhibit 2 were possibly due to the use of laboratory zeolite samples

which were not loaded in the same perfect manner as the zeolites of Example 8 of D12 cannot affect the conclusion drawn from the graph, since according to the experimental report in section 6.4 of the substantiation of the appeal the powder and the beads comprised the same ion exchanged zeolite material.

Moreover, it appears to be common ground that the Si:Al ratio of an X-zeolite can have a certain impact on its capacity and selectivity, see eg the substantiation of the appeal, section 6.3. However, an exact Si:Al ratio of 1 (in the sense of say "1.00") is neither mentioned in D12 nor in the contested patent. The appellant's allegation that the expression "low silica X-zeolites" or "LSX" was generally used to refer to X-zeolites having a Si:Al ratio of **exactly** 1 cannot be accepted in view of the various diverging statements comprised in eg D12, see page 2, line 29 ("low silicon to alumina ratios of approximately 1 to 1.2") and lines 33 to 34 ("LSX is low silica zeolite with a Si/Al of 1") and page 4, line 49 ("low silica X-zeolite with a Si/al ratio of approximately 1").

Further, as plausibly pointed out by the respondent during the oral proceedings, the exact way (in terms of temperatures, pressures, timings, reagents etc) in which the zeolite was prepared, handled, admixed to the binder, activated etc. may also have an impact on the adsorptive properties of the composite adsorbent.

- 4.4.3 In view of the above and in the absence of any further evidence, the Board is not convinced that the composition and structure of the adsorbent of Example 8 of D12 is sufficiently described in terms of the exact preparation conditions of the zeolite, the method of providing bonded zeolite, and the handling and activation thereof, to establish with certitude that it inevitably will have the adsorptive behaviour

prescribed by present claim 1, ie that its N₂ working capacity and its working selectivity would inherently fall within the boundaries specified by present claim 1. In particular, the appellant has not conclusively shown that the fact that the unbonded powdery zeolites of Example 8 of D12 and the 85/15(Li,Ca)LSX sample of the present patent both have the same N₂ working capacity value of 1.10 mmol/g necessarily implies that, irrespective of some possible but undisclosed variations in the Si:Al ratio and the preparation history of the composite adsorbent, the adsorbent of Example 8 would have the same working selectivity as the composite 85/15 Li,Ca-LSX sample of the present patent.

- 4.4.4 In view of the above mentioned gaps in the disclosure of D12, the Board comes to the conclusion that this document does not provide a clear and unambiguous disclosure of an adsorbent comprising an X-zeolite and an inert diluting binder having inherently and beyond all doubt all of the properties required by present claim 1.
- 4.5 The Board is satisfied that none of the other documents cited in the opposition and appeal proceedings, including D11, discloses a process with all the features of present claim 1. Since this was not in dispute, no reasons need to be given in this respect.
- 4.6 Hence, the subject-matter of present claim 1, and consequently of claims 2 to 11 depending thereon, is novel.
- 5. *Inventive step*
- 5.1 As for the main request, the air separation process disclosed in D1 can be considered to represent the closest prior art.

- 5.2 Starting from D1, the technical problem to be solved by the process as presently claimed can in any case be seen in the provision of an alternative process.
- 5.3 As a solution to this technical problem, present claim 1 prescribes the use of adsorbents comprising an inert diluent and specific zeolites exchanged with lithium and a further cation (exchange with lithium more than 50%, exchange with other cation 5 to 50%), the adsorbents having a relatively high nitrogen/oxygen working selectivity and a working capacity within specific boundaries. It has not been contested, and the data provided by the respondent make it plausible, that the ion exchanged zeolites of the type referred to in present claim 1 have properties which makes them as suitable for adsorptive nitrogen separation as some of the known adsorbents, see eg Table III and Figure 5 of the patent in suit.
- 5.4 The claimed solution cannot be considered to be obvious for the following reasons:
- 5.4.1 As pointed out by the appellant during the oral proceedings, D1 mentions binary exchanged zeolitic adsorbents. However, the zeolites referred to in D1, column 2, lines 13 to 21 are said to be exchanged with calcium and strontium. The quoted passage is silent about an ion exchange with lithium, let alone to at least 50%. The mentioned zeolites are thus different from the ones to be used according to present claim 1. Moreover, the quoted passage of D1 does not refer to properties such as working capacities and selectivities. Hence it cannot suggest the provision of the particular zeolites as defined in present claim 1 and their suitability for the claimed process.
- 5.4.2 Although according to its own statement the appellant has carried out a further search for prior art relating

to adsorbents as referred to in the claims according to the auxiliary requests, the only material subsequently presented was late published D12, which cannot be taken into consideration in the assessment of inventive step.

- 5.4.3 The appellant also argued that some prior art only referred to in the description of the contested patent itself was to be taken into consideration in the evaluation of inventive step. More particularly, in Example 4 of the patent the expression "adsorbents known in the prior art" is repeatedly used to refer to the adsorbents tested, which include "(Li,Ca)LSX-zeolite", ie a zeolite exchanged with lithium and a further cation. See in particular page 9, lines 9, 20 and 21. The Board however takes the view that at least in the present case the expression "known in the prior art" does not necessarily mean that all of the mentioned zeolites, and in particular the "(Li,Ca)LSX-zeolites" indeed belong to the state of the art as defined in Article 54(2) EPC, rather than merely to the respondent's own or "internal" prior art, such as eg D12, unpublished at the priority date of the present patent. Hence, in the absence of further evidence, and considering that the further search conducted by the appellant did not produce any results apart from D12, the Board cannot accept that the said (Li,Ca)LSX-zeolites belonged to the prior art according to Article 54(2) EPC.

Even if, for the sake of argument, the statements in Example 4 of the patent in suit were to be taken as an acknowledgment that the zeolitic adsorbents mentioned there, eg the (Li,Ca)LSX-zeolites, belonged as such to the prior art in the sense of Article 54(2) EPC, these mere statements can still not be considered to represent a disclosure of a VSA or PSA process for separating nitrogen from a gas mixture, let alone with zeolites having a more specific composition, ie being

exchanged with lithium to at least 50% and with a further cation to 5 to 50%, and having specific properties, such as a relatively high nitrogen/oxygen selectivity of about 17 or more. Nor could such a hypothetical prior art suggest the suitability of these more specific zeolites in a specific nitrogen separation process of the type disclosed in D1.

5.4.4 The disclosure provided by the quoted statements in the patent in suit does not, if at all, go beyond the disclosure of (Li,Ca)LSX zeolitic adsorbents as such, without an indication concerning the degrees of the ion exchanges, the working capacities and selectivities or a particular process making use of them. Hence the Board can also not accept the approach to the assessment of inventive step as adopted by the appellant during the oral proceedings, which was based on the consideration of this hypothetical disclosure as closest prior art for the process of claim 1.

5.4.5 Hence, the appellant has not produced evidence convincingly showing that the specific zeolitic adsorbents relied upon according to present claim 1 were known at all from published documents or were known to otherwise belong to the state of the art according to Article 54(2) EPC. Moreover, it could not be derived from the prior art relied upon by the appellant that these particular zeolites would be as suitable as other known ion exchanged zeolites for adsorptive PSA or VSA air separation processes. In view of the above considerations, the Board is also convinced that the other documents cited during the opposition and appeal proceedings are not relevant for the assessment of inventive step in respect of the claims of the present request. Since this was not in dispute, no reasons need to be given in this respect.

5.5 Hence the subject-matter of claim 1, and consequently

of the dependent claims 2 to 11, is found to be based on an inventive step.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the opposition division with the order to maintain the patent with the claims of the "2nd Auxiliary Request" filed with letter dated 15 January 2003 and a description to be adapted.

The Registrar:

The Chairman:

U. Bultmann

R. Spangenberg