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D E C I S I O N
of 11 January 1994

Case Number: T 0199/92 - 3.3.1

Application Number: 82109001.6

Publication Number: 0075940

IPC: C07C 85/06

Language of the proceedings: EN

Title of invention:
Process for the manufacture of ethylenediamine

Patentee:
Union Carbide Corporation

Opponent:
BASF Aktiengesellschaft, Ludwigshafen
Berol Kemi AB

Headword:
Ethylenediamine/UNION CARBIDE

Relevant legal norms:
EPC Art. 56, 84, 107, 2nd sentence
EPC R. 55(c)

Keyword:
"Admissibility of Opposition (yes)"
"Notice of Opposition - meaning of 'indication' in Rule 55(c)"
"Notice of Opposition - not fatally defective for missing page"
"Party as of right: Art. 107 EPC"
"Inventive step of a two-step process (confirmed)"
"Combination of process features - non-obvious in view of the technical problem"

Decisions cited:
T 0289/91

Catchword:
-



Case Number: T 0199/92 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 11 January 1994

Appellant:
(Opponent 01)

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Representative:

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Respondent:
(Proprietor of the patent)

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Decision under appeal:

Interlocutory decision of the Opposition Division
of the European Patent Office dated 12 February
1992 concerning maintenance of European patent
No. 0 075 940 in amended form.

Composition of the Board:

Chairman: A. Jahn
Members: P. Krasa
J.A. Stephens-Ofner

Summary of Facts and Submissions

- I. The mention of the grant of patent No. 0 075 940 in respect of European patent application No. 82 109 001.6 filed on 29 September 1982, was published on 18 September 1985 (c.f. Bulletin 85/38) on the basis of four claims.
- II. Two notices of opposition were filed on 11 June 1986 and 16 June 1986, respectively, in which the revocation of the patent in its entirety was requested on the ground that its subject-matter was not inventive. From the second notice of opposition (Opponent II), in which the novelty of the claimed subject-matter was also called into question, a page 5 was missing, which, after a telephone call from the EPO, indicating this deficiency, was submitted on 23 June 1986.

The oppositions were supported, *inter alia*, by the following documents:

- (1) DD-A-149 509,
- (2) DD-A-14 480, and
- (6) Törnquist, *Kemisk Tidskrift*, 1981 (Nr. 8), 37-42.

After expiry of the period allowed for filing notice of opposition, the Opponent II also relied, *inter alia*, on:

- (9) US-A-3 697 598.

By its decision of 12 February 1992, the Opposition Division maintained the patent in amended form with three claims. Claim 1 reads:

"A continuous process for producing ethylenediamine which comprises providing a continuous, homogeneous fluid stream containing ammonia and monoethanolamine wherein the moles of ammonia substantially exceed the moles of alcoholic hydroxyl in said stream, to an amination zone comprising a solid amination catalyst to form an amination product stream and continuously recovering ethylenediamine from said amination product stream, characterized in that (a) the homogeneous fluid stream is the product of the direct reaction of ethylene oxide and ammonia, under supercritical fluid phase conditions in the presence of a small amount of water as the only catalyst and in the absence of hydrogen, and contains additionally diethanolamine and triethanolamine, (b) a stream consisting essentially of monoethanolamine is separated from the amination product stream and fed to the homogeneous fluid stream and (c) an amination feed stream is formed in which at least 70 weight percent of the total ethanolamines is monoethanolamine and based on the total ethanolamines the weight percent of monoethanolamine is at least 5 percent greater than in the homogeneous feed stream."

The Opposition Division held that the subject-matter of the patent in suit was novel. Further, it found that document (1), which was deemed to represent the closest state of the art, solved the same technical problem as the patent in suit, which means to provide a continuous process for the manufacture of ethylene diamine (EDA) from ethylene oxide (EO) and ammonia with low amounts of piperazine. The solution suggested in the patent in suit, a continuous process where ethylene oxide and ammonia are reacted in a supercritical fluid phase in the absence of hydrogen and in the presence of small amounts of water as the only catalyst, was held to be inventive as not being derivable in an obvious manner from the citations.

III. A single appeal was lodged against this decision on 4 March 1992 with payment of the prescribed fee. In his Statement of Grounds of Appeal, filed on 12 June 1992, and during the oral proceedings, held on 11 January 1994, the sole Appellant (Opponent I; Opponent II did not lodge an appeal) argued essentially that the appealed decision was based on a wrong assessment of the state of the art. He submitted that the omission of hydrogen from the reaction of ammonia with EO disclosed in document (1) was self-evident for a large scale operation, as hydrogen did not take part in the said reaction, and would therefore have required additional and economically and technically unreasonable expenditure. The use of supercritical conditions and of small amounts of water in the reaction of ammonia with EO was said to have been disclosed in document (1). Furthermore, the Appellant, relying on document

(10) Ullmanns Encyclopädie der technischen Chemie,
Vol. 8, Fourth Ed., 143-144 (1974),

submitted that the manufacture of ethanolamines in the absence of hydrogen and in the presence of small amounts of water under supercritical conditions was part of the state of the art. As document (1) also discloses the recycling of a product stream, a skilled person would have combined the teaching of document (1) with that of document (6), the latter relating to the industrial manufacture of EDA from EO and ammonia whereby the monoethanolamine (MEA) resulting in the first step and not consumed in the second step of the process was recycled to the said second step. The use of supercritical conditions for the EO-ammonia reaction was, so the Appellant submitted, also disclosed in the state of the art, e.g. not only in document (1), but

also in references (2), (6), (9) and (10). He concluded that, therefore, the claimed process was not inventive, in particular over citations (1) and (6).

Finally, the Appellant submitted that the improved selectivity achieved in the example of the patent in suit, as compared with the process of citation (1), was due to the selection of a particular catalyst used for the MEA amination and not to the features of the present Claim 1. The Appellant argued that such a selection of an appropriate catalyst was not inventive.

- IV. In a communication, dated 6 December 1993, the Board had expressed its doubts as to Opponent II being a party as of right to these proceedings under Article 107, 2nd sentence, EPC in view of the incompleteness of his notice of opposition, and the possible consequential inadmissibility of his opposition, which would mean that he could never satisfy the provisions of Article 107, 2nd sentence, EPC since, his opposition having been inadmissible, he would not be a party to proceedings capable of becoming a party as of right to the appeal.

In the course of oral proceedings, which took place on 11 January 1994, the Respondent submitted that at the time of the receipt of the notice of opposition, he could not readily understand the obviousness attack against Claim 1 as granted. Opponent II (the potential party as of right under Article 107 2nd sentence, EPC), countered by submitting that there was no proof that page 5 had in fact been omitted by him from its notice of opposition and not merely misplaced by the EPO and, furthermore, that the case made against Claim 1 as granted was wholly and readily comprehensible, even without page 5, which merely served as an index or guide to material that was either already known to the

Respondent from the examination proceeding or had been filed in due time. In other words, so he argued, page 5 was superfluous to the understanding of the obviousness attack.

Opponent II did not comment in writing on the substantive issues of this appeal. During oral proceedings, which took place on 11 January 1994, he adopted the Appellant's arguments and submitted in particular that the expression "liquid" in the citations refers also to "supercritical liquid phases", and that, thus, supercritical liquid phases were already disclosed in document (6).

- V. In the course of oral proceedings, the Respondent, after having been made aware by the Chairman of possible deficiencies of the pending amended Claim 1, filed a new set of three claims. In new Claim 1, the passage "under supercritical fluid phase conditions in the presence of a small amount of water as the only catalyst and" was replaced by "wherein a single supercritical fluid phase is maintained with a density of at least 240 kg/m^3 in the presence of 0.5% to 5% by weight water based on the weight of the reaction mixture as the only catalyst and". Furthermore, in the last line "feed stream" was replaced by "fluid stream". The dependent Claims 2 and 3 are identical with those on which the decision under appeal was based.

The Respondent also submitted a description adapted to the new set of claims, and argued essentially that the process according to document (1) was not performed under supercritical conditions and that this citation neither gave a hint to use water as the sole catalyst in the first process step nor to recycling a stream consisting essentially of MEA to the amination zone. Furthermore, the Respondent contested that any of the

citations disclosed an EO-ammonia reaction under supercritical fluid conditions. Thus, in his submission, the particular combination of the features of the present process could not be deduced from the cited documents and in particular not from a combination of references (1) and (6).

VI. The Appellant and Opponent II both requested that the latter should be admitted to the appeal proceedings under Article 107, 2nd sentence, EPC. The Respondent requested that such admission be refused. After deliberation of the Board, the Chairman announced the Board's decision to refuse the Respondent's request. The Appellant further requested that the decision under appeal be set aside and that the European patent No. 0 075 940 be revoked. The Respondent requested that the appeal be dismissed and that the patent be maintained on the basis of claims and description submitted in the course of oral proceedings. At the end of the oral proceedings the Chairman announced the Board's decision to maintain the patent in amended form.

Reasons for the Decision

1. *Procedural Issues*

1.1 This Board, in a different composition, had already decided that the admissibility of an opposition, as an indispensable procedural precondition for examining an appeal from an Opposition Division's decision on its merits, has to be established by the EPO on its own motion (see T 0289/91 of 10 March 1993, No. 2.1 of the Reasons for the Decision, headnote I published in the OJ EPO 12/1993). Therefore, the Board is empowered to raise

the issue of admissibility of an opposition, if it has sound reasons for so doing, as is the case in the present appeal.

1.2 It is accepted jurisprudence that the term "indication" in Rule 55(c) EPC means that a Patentee must be able to understand, without undue burden, the case that is being made against his patent in the notice of opposition. Whilst this requirement does not exclude the possibility that a Patentee may have to undertake a certain amount of interpretation, it does clearly preclude the admissible presentation of an attack against a patent in a manner that leaves a Patentee either at a complete loss to understand it, or imposes an undue mental burden upon him. Clearly, admissibility under Rule 55(c) EPC must therefore depend upon the facts of each case.

1.3 The notice of opposition of Opponent II sets out in the first paragraph on page 4 that feature (a) of Claim 1 as granted was admitted in the patent in suit as being known in the art and paragraphs 1 and 2 on page 7 refer to the recycling of unreacted MEA to the amination step, i.e. to the features (b) and (c) of Claim 1 as granted. In the first paragraph on page 7, reference was made to a document "(4)" mentioning reactors "A" and "B". Given this fact, it was a simple matter by reference to the two documents enclosed with the notice of opposition, to identify this particular document "(4)" and the attached page of its partial translation into English - now citation (6) - as being the supporting evidence for the obviousness attack explicitly raised in the second paragraph of the said page 7.

1.4 Thus, the Board is prepared to accept that in this case, on the balance of probabilities, the Respondent was not presented with a jig-saw puzzle, and that he not only could have readily understood the precise nature of the

obviousness attack that Opponent II had made against the claims as granted, but could also have related the said document (6) to the Appellant's arguments concerning features (a) and (b).

Accordingly, the Board finds the second opponent's notice of opposition to have been in compliance with the requirements of Rule 55(c) EPC, and, thus, the respective opposition to have been admissible, with the consequence that as a party to the opposition proceedings he may become a party as of right under Article 107, 2nd sentence, EPC.

2. *Amendments*

The Board is satisfied that the amended claims are supported by the application documents as originally filed and do not extend the scope of protection conferred by the claims as granted. As this was not contested by the parties, no further comments are necessary in respect to Article 123 EPC as a whole.

The Appellant submitted that Claim 1 is unclear (Article 84 EPC) owing to the absence of a specific temperature range to define "supercritical fluid phase conditions". However, the skilled person is familiar with the term "supercritical" which, thus, together with the density given, sufficiently defines the "fluid phase conditions". Therefore, the present Claim 1 is clear and in compliance with the requirements of Article 84 EPC.

3. *Novelty*

After examination of the cited prior art, the Board has reached the conclusion that the claimed subject-matter is novel. Since the novelty of the present claims was no longer disputed in the appeal proceedings, it is not necessary to give reasons for this finding.

4. *The Technical Problem and its Solution*

4.1 The patent in suit is concerned with the manufacture of EDA, by reacting ammonia with a product mixture comprising MEA, which in turn is obtained from the reaction of EO with ammonia under the reaction conditions as specified in the present Claim 1. According to the patent in suit, similar processes were already known (column 1, line 14 to column 3, line 35), but suffered from the co-production of piperazines (piperazine, aminoethylpiperazine, and hydroxyethylpiperazine; all together referred to in the following as PIP) as by-products (column 2, lines 34 to column 3, line 35). The PIP formation was assumed to be induced by diethanolamine (DEA) and/or triethanolamine (TEA) present in the MEA starting material (column 3, lines 45 to 51), which necessitates a price increasing purification of the latter (column 3, line 60 to column 4, line 12). The PIP formation itself is undesirable because there exists no large enough market for PIP (column 2, lines 21 to 26) .

Consequently, the technical problem addressed in the patent in suit was to reduce the formation of PIP in the manufacture of EDA by the amination of MEA (column 5, lines 12 to 20).

4.2 In contrast to the position taken by the Opposition Division (see the decision under appeal, page 6, 3rd paragraph, and page 9, 1st paragraph), the Board cannot see any need to reformulate the stated technical problem, even if it would consider reference (1) as the most relevant prior art, as suggested by the first instance and all the parties to the proceedings. In these circumstances, the Board is prepared to accept document (1) as the starting point for evaluating inventive step. It follows from the examples of this document that in the obtained product mixture the molar ratio of EDA : PIP is about 1:1 to 1:2. (example 1 = 11,9:10,9; example 2 = 8,3:17,9; example 3 = 9,4:12,2; see the table on page 8). Thus, the process of document (1) suffers from the same drawback as discussed in the patent in suit, i.e. from a (rather high) formation of PIP.

4.3 According to the data given in the example of the patent in suit, which were not contested by the Appellant, the molar ratio of EDA:PIP in the obtained product mixture is 14,4:1, which demonstrates an improved selectivity of the process.

The Appellant argued that this increased EDA/PIP ratio, as compared with that disclosed in document (1), is due to the amination catalyst used in the second step of the process according to the example of the patent in suit, and not to the features of present Claim 1. In other words, the use of a "wrong" catalyst in the second step of a process according to this claim, which does not call for a particular catalyst, would not give the desired result. This amounts in fact to the objection that the existing technical problem is not solved by all embodiments falling within the scope of Claim 1. However, as no supporting evidence was submitted by the

Appellant, the Board is not prepared to accept this argument, as it is based on a mere allegation.

Thus, the Board is satisfied, that the above technical problem was solved by the process of present Claim 1 of the patent in suit.

5. *Inventive Step*

The next issue to be decided is whether or not the claimed process meets the requirement of inventive step.

5.1 According to a preferred embodiment of the process of document (1), 1 mole EO and from 5 to 50 moles (preferably from 8 to 15 moles) ammonia are introduced at the lower part of a pressure reactor and react in a first zone of inert material to form ethanol amines (page 3, lines 18 to 21 in combination with page 4, lines 22 to 24). There is a second zone, also filled with inert material, in which the appropriate temperature is adjusted, which is required for the following reaction zone, filled with a hydrogenation catalyst for the amination reaction (page 3, lines 21 to 26). In view of the high velocity of the reaction of EO with ammonia, the respective reaction zone can be short - about 15% of the reactor's length - whereas the following zone for the temperature adjustment may require up to 60% of the reactor's length (page 4, lines 3 to 10). Hydrogen, which is required for maintaining the catalyst's activity, is introduced into the reactor either at its lower end or immediately below the zone of the hydrogenation catalyst, and the ammonia used is preferably free of water. However, water has no detrimental effect (page 6, lines 8 to 17). The temperature for the reaction of EO with ammonia is from 70°C to 150°C (from 343°K to 423°K), preferably from 110°C to 130°C (from 383°K to 403°K), and the pressure

has to be sufficient to maintain a liquid phase in all the reaction zones; it is preferably from 12 to 25 MPa (page 5, lines 5 to 17). The reaction product may be recycled and, in such a case, is introduced into the reactor at its lower end (the paragraph bridging pages 4 and 5, in combination with page 7, lines 15 to 18).

5.2 A comparison with the process of present Claim 1 shows that the latter differs from that of document (1) at least in the following two aspects:

- the use of a single supercritical fluid phase for the EO-ammonia reaction, and
- and the recycling of a stream consisting essentially of MEA to the amination feed stream.

5.3 The Appellant submitted that according to example 3 of citation (1), temperatures of 128°C (401°K) and of 192°C (465°K) were measured at distances of 300 mm and of 1100 mm, respectively, above the reactor's lower end. The first temperature was only some few degrees below, and the second temperature was well above the critical temperature of the reaction mixture. The applied pressure of 15 MPa being above the critical pressure of the reaction mixture, the Appellant concluded therefrom that the process of example 3 of citation (1) was performed under supercritical conditions, even if this were not said *expressis verbis*. The Respondent contested this statement, which contention, in the absence of any corroborating evidence, is no more than a bare assertion. Taking into account that document (1) teaches that EO and ammonia react in a rather short zone after their introduction into the reactor (see above No. 5.1; in example 3 EO is introduced through a pipe reaching - from the lower end - 100 mm into the reactor at a temperature of 105°C [378°K]; see page 7, lines 19 to 20 in combination with page 8, column 3 of the table), the

Board finds it more credible that at least the major part of the EO-ammonia reaction in the said example 3 is already completed at the 300 mm distance and is, therefore, not performed under supercritical conditions. Therefore, the Board rejects this part of the Appellant's submission.

In the Board's judgement, there is no clear teaching in document (1) that a single supercritical fluid phase should be maintained in the first reaction step, although the disclosed generic temperature and pressure ranges reach, in theory, into the supercritical region. Furthermore, document (1) suggests to select the flow rate at the catalyst according to the desired polyamine (page 5, penultimate paragraph). This points, as far as the solution of the existing technical problem is concerned, to a direction different from the one followed by the patent in suit.

5.4 The Appellant, submitting that very often no clear distinction is made in the state of the art between "liquid state" and "supercritical fluid state", also referred to documents (2), (6), (9) and (10). In his opinion, these citations were evidence that it was already known to manufacture MEA from EO and ammonia under supercritical conditions.

5.4.1 Document (2) relates to the production of EDA and PIP by reacting ammonia either with MEA (examples 1 to 3) or with EO (example 4) in the presence of a hydrogenation catalyst (page 2, lines 44 to 56) without explicitly mentioning supercritical fluid conditions for the reaction. According to Claim 1, the reaction is carried out in the "liquid phase". While in example 4, the only one being relevant in the present context, a temperature of 210°C is applied (page 2, line 92), no pressure is given. This example yields, inter alia, 10 parts by

weight of EDA and 15 parts by weight of PIP (page 3, lines 1 to 2). The EDA/PIP ratio may be regulated by the EO/ammonia ratio; and an increase of the reaction temperature favours the PIP formation (see page 2, lines 32 to 37). This disclosure points away from the present invention. Therefore, even if one would assume that the EO-ammonia reaction in example 4 had been performed under supercritical conditions - in spite of the incomplete information available in this respect - citation (2) would not have lead the skilled person to the process of the patent in suit as a solution of the existing technical problem.

5.4.2 Document (10) discloses that ethanol amines are manufactured on an industrial scale solely by reacting EO with an excess of aqueous ammonia. The ammonia concentration is up to 90%, the pressure up to 150 bar and the temperature up to 150°C (page 143, right hand column, lines 1 to 3 and 24 to 26 after the sub-heading "2. Herstellung"). The Appellant submitted that these reaction conditions also encompassed a reaction in the supercritical fluid phase when the reaction mixture contained 90% ammonia. This was contested by the Respondent, who maintained that with high amounts of water, such as were involved as soon as aqueous ammonia was used, no single supercritical fluid phase could be achieved under the given conditions. Be that as it may, there is no hint in this document that the reaction product resulting from the EO-ammonia reaction could be directly employed in the EDA manufacture. Thus, in the Board's judgement, the skilled person would not have found any incentive for combining the disclosure of citation (10) with that of document (1).

5.4.3 Document (6) is in Swedish and can be taken into consideration only in the form of a partial translation into English, which had been attached to it. This

document discloses the two-step manufacture of EDA by reacting EO with water-free ammonia and further aminating the resulting MEA with ammonia. Unreacted MEA is recycled to the second, the amination step. Apart from EDA, also PIP is produced (see the above mentioned translation, last paragraph in combination with the second paragraph, lines 1 to 4, and the entire third paragraph). In respect to the first step it is said: "This reaction, being catalyzed by ammonia saturated Ion Exchange Resin, is so selective, that Monoethanolamine needs no purification prior to the second step of the process".

This, in the Board's judgment, makes it clear that a sufficiently pure MEA is required in the second step of the process, which, according to this citation, is achieved by the reaction conditions applied in the first step, i. e. the use of water-free ammonia and of a particular catalyst. These conditions are mandatory to maintain the required selectivity of the first step and there is no indication at all in reference (6) that the latter could also be achieved by simply replacing the ion exchange resin by water. No other reaction parameters are disclosed in respect to the EO-ammonia reaction. As far as a reaction temperature of 200°C, a pressure of "a few tens of MPa", and a liquid phase are mentioned, which according to the Appellant indicates supercritical conditions, these parameters refer only to the exchange of the hydroxy group against an amino group in the second step (see the above mentioned translation, second paragraph, lines 7 to 12). Beyond that, PIP is a desired product in the process of document (6) and in this respect is put on the same footing as EDA, as the first paragraph of the said translation shows: "Ethylene Diamine and Piperazine ... [They] are starting materials for among others Chelating agents, Plant protection agents and Veterinary medicines."

Thus, while reference (6) is silent on the mutual amounts of EDA and of PIP, the skilled person could not gain any information from document (6), how to increase the EDA/PIP ratio.

5.4.4 Document (9) relates to a highly selective continuous process for the preparation of monoalkanolamines from ammonia and alkylene oxides in the presence of a cation exchange resin as a catalyst (column 1, lines 5 to 9, and lines 26 to 30). The manufacture of MEA according to this process is disclosed particularly in examples 1 to 14, 17 to 29, and 31 to 40 (tables I, II and III in columns 5, 6 and 7). The reaction temperatures are from 20°C to 250°C, preferably from 75°C to 150°C, and the reaction pressure is from 40 to 200 atmospheres (column 1, lines 58 to 60, and column 3, lines 65 to 67). The Appellant submitted that this process was in fact the same one that was used in the first step of the process of reference (6) and that it encompassed supercritical fluid conditions for the reaction, both of which submissions were contested by the Respondent. The gist of this document is, however, that a cation exchange resin is required as a catalyst for the process. In this respect the situation is similar to that with document (6): The use of a cation exchange resin is mandatory for achieving a high selectivity and there is no indication that a replacement of this catalyst by plain water would lead to the same result.

Furthermore, in examples 17 to 29 of table II the product distribution (MEA, DEA, TEA) is given for three ammonia: EO molar ratios (40:1 in examples 17 to 24; 60:1 in examples 25 to 27; and 80:1 in examples 28 to 29). For the examples 24, 27 and 29, which in view of the temperature and pressure given (140°C and 145 atmospheres; table II in column 6) may describe a supercritical state for the reaction mixture, MEA-

contents of 84.3 wt%, 80.2 wt%, and 88.7 wt% are disclosed, based on the total of MEA, DEA, and TEA. These figures are lower than the respective ones reported for lower reaction temperatures and pressures, e.g. 100°C and 100 atmospheres: about 89 wt%, about 92 wt%, and about 93 wt%, respectively. Therefore, even if assuming that supercritical conditions were implicitly disclosed for the EO-ammonia reaction in document (9), these results would deter the skilled person from applying the high temperature and pressure required for maintaining a single supercritical fluid phase when aiming at a process with high MEA-selectivity for ascertaining in turn a low PIP production in the following amination step.

5.5 Therefore, the Board concludes that none of the citations (1), (2), (6), (9) and (10), either singly or in combination, would have led the skilled person, faced with the existing technical problem, to the combination of process features of Claim 1. It follows that the subject-matter of Claim 1 involves an inventive step.

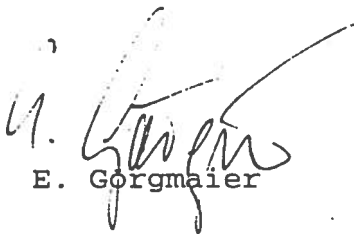
5.6 Dependent Claims 2 and 3 relate to particular embodiments of Claim 1 and derive their patentability from that of Claim 1.

Order

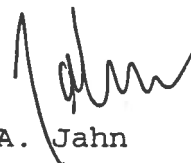
For these reasons, it is decided that:

1. The Respondent's request, not to admit Opponent II as a party as of right, is refused.
2. The Opposition Division's decision is set aside.
3. The case is remitted to the Opposition Division with the order to maintain the patent with the claims and amended description submitted in the course of oral proceedings.

The Registrar:


E. Gorgmaier

The Chairman


A. Jahn