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D E C I S I O N
of 28 July 2004

Case Number: T 1125/01 - 3.3.5

Application Number: 93103027.4

Publication Number: 0558019

IPC: B01D 53/14

Language of the proceedings: EN

Title of invention:

Method for removing carbon dioxide from combustion exhaust gas

Patentee:

THE KANSAI ELECTRIC POWER CO., INC., et al

Opponent:

ExxonMobil Research and Engineering Company

Headword:

Removing carbon dioxide/KANSAI

Relevant legal provisions:

EPC Art. 54(1), 56

Keyword:

"Novelty - yes"

"Inventive step - yes, non-obvious solution to the problem"

Decisions cited:

-

Catchword:

-



Case Number: T 1125/01 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 28 July 2004

Appellants:

(Proprietors of the
patent)

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Respondent:

(Opponent)

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Decision under appeal:

**Decision of the Opposition Division of the
European Patent Office posted 3 August 2001
revoking European patent No. 0558019 pursuant
to Article 102(1) EPC.**

Composition of the Board:

Chairman: M. M. Eberhard
Members: G. J. Wassenaar
H. Preglau

Summary of Facts and Submissions

- I. European patent No. 0 558 019 was opposed by the respondent (opponent) on the grounds of lack of novelty, lack of inventive step and insufficient disclosure.
- II. The appellants (proprietors) lodged an appeal against the decision of the opposition division to revoke the patent. With the statement of the grounds of appeal three new sets of claims as main request and two auxiliary requests were submitted. A third auxiliary request was filed later on with the letter of 26 May 2003. During oral proceedings, which took place on 28 July 2004, a new main request and one auxiliary request were submitted. The main request comprised two independent claims 1 and 4. Claim 4 read as follows:

"A method for removing carbon dioxide from a combustion exhaust gas which method comprises the step of bringing said combustion exhaust gas under atmospheric pressure into contact with an aqueous solution of 2-ethyl amino ethanol."

The auxiliary request comprised two claims, an independent claim 1 and a dependent claim 2. Claim 1 thereof read as follows:

"A method for removing carbon dioxide from a combustion exhaust gas which method comprises the step of bringing said combustion exhaust gas under atmospheric pressure into contact with an aqueous solution of a hindered amine (exclusive of a amine having two or more amino groups) selected from an aqueous solution of 100 parts by weight of an amine-compound (X) selected from the

group consisting of (A) and 1-25 parts by weight of an amine-compound (Y) selected from the group consisting of (D') piperazine, (E') piperidine, (F') morpholine, (G') glycine, (D) and (B), wherein (A), (B), and (D) are defined as:

(A) a compound having an alcoholic hydroxyl group and a primary amino group, said primary amino group being bonded to a tertiary carbon atom having two unsubstituted alkyl groups;

(B) a compound having one alcoholic hydroxyl group and a secondary amino group in its molecule, said secondary amino group having an unsubstituted alkyl group of 3 or less carbon atoms and a N atom bonded to a group having a chain of 2 or more carbon atoms inclusive of a bonded carbon atom;

(D) a 2-substituted piperidine having a hydroxyl group-substituted alkyl group at the 2-position."

III. The respondent disputed the admissibility of the present auxiliary request because of its late filing. Claim 4 of the main request was attacked on the ground of lack of novelty and claim 1 of the auxiliary request on the ground of lack of inventive step. The respondent's arguments with respect to these claims were supported by the following documents:

D1: GB-A-1 058 304

D3: US-A-4 112 052

D8: Environmental International, Vol. 2,
pages 453-456.

D9: Dictionary of Science and Technology, W & R
Chambers, 1983, pages 470-471.

D11: The Chemical Engineer, October 1966, Review Series
No. 2, pages CE244 to CE281.

- D12: Trans. Instn Chem Engrs, Vol. 45 (1967),
pages T32-T49.
- D13: I&EC Fundamentals, Vol. 8, No. 3, August 1969,
pages 415-423.
- D23: Kirk-Othmer's Encyclopedia of Chemical Technology,
Vol. 3, 1954, pages 128-133.
- D25: US-A-4 336 233.

IV. The respondent's arguments may be summarised as follows:

D1 disclosed all the features of claim 4 of the main request. The subject-matter of this claim therefore lacked novelty over D1.

The process according to claim 1 of the auxiliary request lacked an inventive step. D25 disclosed the removal of carbon dioxide from gases by absorption through an aqueous solution comprising alkanolamines in the presence of piperazine. D25 specifically disclosed that piperazine in catalytic amounts accelerated the absorption of carbon dioxide by conventional physical or chemical solvents. This effect was independent of the gas composition and the pressure used during the absorption process. In this respect reference was made to D11, D12 and D13. The absorption of carbon dioxide from combustion exhaust gas by an aqueous solution of alkanolamines was specifically disclosed by D23 and alkanolamines according to group (A) of claim 1 were known absorption agents as admitted in the patent in suit and also shown by D3. In a process for removing carbon dioxide from combustion exhaust gas by an aqueous solution of alkanolamines according to group (A) it was obvious to increase their absorption rate by

adding a small amount of piperazine. No surprising effect was shown for the claimed combination of amines.

- V. The arguments of the appellants may be summarised as follows:

D1 did not disclose the use of 2-ethyl-aminoethanol (EAE) in combination with a combustion exhaust gas. The subject-matter of claim 4 of the main request was therefore novel. It also involved an inventive step because of the unexpected high efficiency of the claimed process.

D25 did not relate to the treatment of combustion exhaust gas and did not disclose the use of amino alcohols of the group (A) according to claim 1 of the auxiliary request. The accelerating effect of piperazine was described in D25 only for secondary and tertiary amines. It could not be foreseen that the claimed combination of amines would improve the efficiency of the removal of carbon dioxide from a combustion exhaust gas.

- VI. The appellants requested that the decision under appeal be set aside and the patent be maintained either on the basis of the main request or on the basis of the auxiliary request, both requests filed during the oral proceedings.

The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. The novelty of claim 4 of the main request was attacked on the basis of D1. This document discloses a process for the removal of hydrogen sulphide and/or carbon dioxide and/or carbonyl sulphide from a gas by absorption through an aqueous solution of alkanolalkyl amines (claim 1). D1 specifically discloses ethylaminoethanol (EAE) in combination with the absorption of carbon dioxide; see page 2, line 1 and the table on page 4. It is undisputed that this EAE is the 2-ethyl-aminoethanol according to claim 4. Among five types of gases which can be advantageously treated in this way flue gas is mentioned (page 3, lines 54 to 58). The composition of the flue gas is not disclosed but a flue gas is generally the exhaust gas of a boiler furnace wherein a fuel is burned; see D9, under flue gas, and D8, page 454, in particular Tables 1 and 2. The appellant maintained that combustion exhaust gas was not identical to flue gas, but could not provide evidence to show the difference. In the absence of a definition of combustion exhaust gas in the patent in suit, and further taking into account that a boiler combustion gas is used in Example 9 of the patent in suit, the board cannot accept any factual difference between combustion exhaust gas and flue gas. The pressure at which the absorption takes place is not limited in D1 but it is indicated that elevated pressure may be used (page 5, lines 3 to 5 and claim 12). In the only example an aqueous solution of methylaminoethanol (MAE) is used at a pressure of 20 atm. D1 mentions absorption at atmospheric pressure

in comparative experiments to determine the absorption rate of a test gas consisting of substantially pure carbon dioxide (page 4, lines 1 to 5). According to the table on page 4 the rate of absorption of carbon dioxide by EAE at atmospheric pressure is more than 1.5 higher than for monoethanolamine (MEA). A process whereby flue gas is treated by an aqueous solution of EAE at atmospheric pressure is not directly and unambiguously derivable from D1. To arrive at the subject-matter according to claim 4 of the main request the skilled person would have to select EAE from a list of several alkanolalkylamines and the flue gas from a second list of possible gases to be treated, and to choose a treatment at atmospheric pressure for this combination. The subject-matter of claim 4, resulting from the said selection of several features from D1, is therefore novel.

3. Starting from D1 as the closest prior art for the subject-matter according to claim 4 of the main request the problem underlying the claimed method can be seen in providing a process for efficiently removing carbon dioxide from a combustion exhaust gas. According to claim 4 of the main request the appellant proposes to solve that problem by absorbing the gas in an aqueous solution of EAE at atmospheric pressure. It follows from Table 1, Example 3 of the patent in suit that the absorption capacity (expressed in mole CO₂/mole amine) of an aqueous test solution of 30 wt% EAE is higher than that of a 30 wt% solution of MEA, the rate of absorption being maintained at a high level. As acknowledged in the patent in suit (page 2, lines 11 to 18) and confirmed by D8 (page 455), aqueous solutions of MEA have been preferably used in the art for

absorbing carbon dioxide from combustion exhaust gas. The board is therefore satisfied that the process according to claim 4 of the main request actually solves the said problem.

4. An aqueous solution comprising EAE is one of the three solutions mentioned in claim 4 of D1 and thus apparently one of the preferred amine solutions. There is no indication in D1 that EAE would not be suitable, or would be less suitable for the removal of carbon dioxide from flue gas, one of the gaseous mixtures which according to D1 can be advantageously treated by the processes proposed therein (page 3, lines 54 to 58). Furthermore the process of D1 does not require the use of elevated pressure and the absorption rates reported in the table on page 4 show that carbon dioxide is absorbed at atmospheric pressure by EAE at a higher rate than MEA, using substantially pure carbon dioxide. Therefore the skilled person confronted with the problem stated above would have contemplated trying EAE not only at elevated pressure but also at pressures down to the atmospheric pressure. It is within the competence of the skilled person to determine by routine experimentation the most appropriate pressure for performing the absorption in the case of combustion exhaust gas. Moreover, it is known in the art to perform the scrubbing of carbon dioxide from combustion exhaust gas by aqueous solutions of alkanolamines at atmospheric pressure; see D8, page 455, lower part of left hand column. For these reasons the board holds that it was obvious to a skilled person trying to solve the above-mentioned problem to absorb carbon dioxide from the combustion exhaust gas with an aqueous solution of EAE at atmospheric pressure.

5. The appellants' argument that solutions of EAE had a higher absorption capacity calculated as mole of carbon dioxide per mole of amine than MEA and that this surprising effect demonstrated an inventive step cannot be accepted. This argument is not relevant starting from D1 as the closest prior art, since D1 discloses already EAE as one of the preferred alkanolamines. The improvement in absorption capacity by EAE compared with MEA, is automatically obtained by the obvious choice of one of the amines which are taught to be preferred in D1 and is not an indication for an inventive step. Because the method according to claim 4 of the main request lacks an inventive step the main request is not allowable.

6. The auxiliary request was filed at the beginning of the oral proceedings and replaced three earlier auxiliary requests on file. Claim 1 of the auxiliary request corresponds to claims 1 and 2 of the earlier main request filed with the grounds of appeal, with the only difference that diethanolamine was deleted from claim 1 and three further amines were deleted from dependent claim 2. These amendments could be regarded as being induced by the respondent's arguments in its letter dated 25 June 2004. The claims of the present auxiliary request could, therefore, not be considered as a surprise to the respondent. The replacement of the earlier three auxiliary requests with the one auxiliary request filed directly at the beginning of the oral proceedings, in fact, simplified these proceedings for all parties. The board, therefore, admitted the present auxiliary request.

7. Claim 1 of the auxiliary request is based on claim 3 as originally filed with the further requirement that the amine is a hindered amine (exclusive of an amine having two or more amino groups). It further differs from original claim 3 by the deletion of some of the selected compounds or group of compounds from the listed amine compounds (X) and (Y) and by the incorporation of group (D) as compound (Y). The feature of the hindered amine (exclusive of an amine having two or more amino groups) is disclosed in the application as filed on page 7, lines 2 to 8. Present group (D) is disclosed in the original application on page 10, lines 16 to 19. Claim 1 fulfils the requirements of Articles 123(2) and (3) EPC. Since no objections were raised under these articles for this claim it was not necessary to give further reasons in this respect.

8. The novelty of the method according to claim 1 of the auxiliary request is not disputed. In the board's opinion D23 represents the closest prior art. It discloses the Girbotol amine process whereby carbon dioxide from a flue gas is absorbed at atmospheric pressure by an ethanolamine. D3 and D25 are less appropriate as starting point for an inventive step analysis because they concern the absorption of carbon dioxide from essentially oxygen-free gases, such as natural gases, coke-oven gases and synthesis gases, and do not relate to the treatment of combustion exhaust gases. The latter generally contain a few percent of oxygen, which may react with amines (see patent in suit, page 10, lines 49 to 52; D8, page 454, Table 1 and D23, page 131) and are therefore chemically different from the gases mentioned in D3 and D25. Moreover D25 does not even disclose the use of alkanolamines with a

primary amino group as a main component of the absorbing solution. Primary amines are mentioned only in connection with the prior art (column 1). During oral proceedings the respondent also considered JP-A-8 671 819, acknowledged in the patent in suit, as an appropriate starting point. Since the Japanese document was cited by the respondent for the first time during the oral proceedings without submitting a translation thereof, and in the absence of any indication whether it deals with the treatment of combustion exhaust gas, this document is not suitable to evaluate inventive step.

9. D23 specifically discloses as ethanolamine MEA. Although MEA has a high absorption capacity at low temperatures (25°C) it is a less effective absorbent than for example diethanolamine (DEA) because at higher temperatures (75°C) the adsorption capacity is still relatively high so that its net absorption capacity is lower than that of DEA; see D23, Table II on page 132. This is confirmed by the absorption figures at 40 and 60°C given in Table 2 of the patent in suit. Relevant for the effective removal of carbon dioxide are the net absorption capacity and the absorption rate (D23, page 132, paragraph above Table II, and page 3, lines 30 to 33 of the patent specification).

10. Starting from D23 the problem underlying the invention as claimed by claim 1 of the auxiliary request can be seen in providing a process for more efficiently removing carbon dioxide from a combustion exhaust gas. The appellants propose to solve this problem by using as absorption solution an aqueous mixture of 100 parts by weight of an amine-compound of group (A), ie an

amine-compound having an alcoholic hydroxyl group and a primary amino group, the latter being bonded to a tertiary carbon atom having two unsubstituted alkyl groups, and 1 to 25 parts by weight of a compound (Y) selected from the group consisting of (B), (D), (D'), (E'), (F') and (G') as defined in claim 1.

Representative for a compound of group (A) is 2-amino-2-methyl-1-propanol (AMP). Representative compounds (Y) are piperazine and 2-(methylamino)-ethanol (MAE) from group (B). According to Table 2 of the patent specification aqueous solutions comprising a 30 wt% mixture of AMP + MAE or piperazine have an absorption capacity of 56.22 and 62.50 Nm³ carbon dioxide/ m³ solution at 40°C respectively and 37.41 and 48.16 Nm³ carbon dioxide/ m³ solution at 60°C respectively (Example 6). The differences in absorption capacity at the different temperatures of 18.81 and 14.34 Nm³ carbon dioxide/ m³ solution respectively, are an indication for the net absorption capacity. For the two solutions of 30 wt% MEA and 30 wt% DEA the absorption differences at 40°C and 60°C are only 4.70 and 10.97 Nm³ carbon dioxide/ m³ solution respectively (comp. Examples 2 and 3). Calculating the absorption differences on the basis of mole of carbon dioxide absorbed per mole of amine in the solution gives a slightly different picture. According to Table 3 these differences are 0.23 and 0.15 for AMP + MAE and AMP + piperazine respectively (Example 6) and 0.05 and 0.18 for MEA and DEA respectively (Comp. Examples 2 and 3). On the basis of mole/mole absorption there is thus not always an improvement in net absorption capacity by the claimed combination of compounds over the prior art compound DEA. The net absorption capacity on a mole per mole basis of the combination of AMP + piperazine remains,

however, at a relatively high level (0.15); for MEA the corresponding value is only 0.05. The initial absorption reaction rate at 40°C for said mixtures with AMP (0.95 and 0.97) is only slightly lower than for MEA (1.00) but considerably higher than for DEA (0.64) determined for the same weight amount of amine in the solutions; see Table 4 on page 10 of the patent specification. From the experimental data in the patent in suit it can therefore be concluded that, compared with MEA, the AMP mixtures have a much higher net absorption capacity while maintaining a high absorption rate and, compared with DEA, the AMP mixtures have a much higher reaction rate without substantial reduction of the net absorption capacity. Thus, both compared with MEA and DEA, at least one essential parameter determining the efficiency of the carbon dioxide removal is improved without substantial reduction of the other parameter, by the use of compositions according to claim 1. The board is therefore satisfied that the method according to claim 1 of the auxiliary request actually solves the said problem underlying the invention.

11. D23 itself does not provide any hint to the claimed solution of the problem. Of the documents relied on by the respondent during the appeal proceedings D3 discloses a component of group (A) as an absorbing agent for carbon dioxide. It discloses that sterically hindered amines comprising a primary amino group attached to a tertiary carbon atom have a better net absorption capacity than MEA (column 4, lines 3 to 39). It further discloses that in order to improve their solubility in water the amines preferably also carry one or more water-solubilizing groups, eg a hydroxyl

group (column 4, lines 39 to 48). Specifically disclosed is AMP as one of the most preferred amines (column 7, lines 22 to 25). Although the possibility of using a mixture of amines is mentioned in D3, specific mixtures thereof are not disclosed. As gases to be treated hydrogen mixed with carbon dioxide, natural gas and town gases are mentioned (column 11, lines 30 to 48). Combustion exhaust gas or flue gas is not mentioned. Thus D3 does not suggest the combination of amines as now claimed and certainly not to use such a combination for treating combustion exhaust gas.

12. During oral proceedings the respondent pointed to the passage on page 2, lines 42 to 47, of the patent in suit, according to which JP-A-8 671 819 would disclose AMP as scrubbing agent for carbon dioxide containing gases. As already indicated above (point 8) neither the document itself, nor a translation thereof, were submitted so that it was not clear which type of gas was actually treated in said Japanese application. The information in the patent in suit concerning JP-A-8 671 819 does not go beyond the disclosure of D3.

13. The respondent further relied on D25, which discloses aqueous mixtures of secondary or tertiary alkanolamines in combination with monomethylethanolamine (MMEA) and piperazine for the absorption of carbon dioxide and/or hydrogen sulphide from gases (column 2, lines 14 to 25 and column 7, Table 1). Gases which can be purified by the process disclosed in D25 are natural gases, coke-oven gases, gases from the gasification of coal and, preferably, synthesis gases (column 2, lines 23 to 25). The treatment of combustion exhaust gas is not disclosed nor suggested in D25. MMEA is the same

compound as MAE mentioned in the patent in suit. D25 further discloses that the use of piperazine in catalytic amounts, as an absorption accelerator in aqueous solution, together with conventional physical or chemical solvents or their mixtures, is particularly preferred (column 3, lines 25 to 28). Amongst the chemical solvents alkanolamines are mentioned and it is indicated that "amongst the alkanolamines, the alkylalkanolamines containing secondary and tertiary nitrogen atoms, and their dialkyl and monoalkyl derivatives, should be mentioned particularly" (column 4, lines 3 to 6). In the examples only such secondary and tertiary alkanolamines have been used. From Table 1 it follows that the accelerating effect of piperazine on a secondary alkanolamine (DEA) is already considerably smaller than on tertiary alkanolamines (MDEA) and (TEA). Thus, despite the general remark in D25 (column 3, lines 25 to 28), concerning the accelerating effect of piperazine on conventional physical or chemical solvents, the skilled person would, in view of the results in Table 1, not have expected, that an accelerating effect might be obtained with a different gas mixture, ie combustion exhaust gas, and specific primary amines as defined in claim 1. According to D25 the addition of MAE and piperazine also increases the loading difference Δx (column 7, line 11 to column 8, line 11). This effect is, however, relatively small and has only been demonstrated with respect to the tertiary alkanolamine MDEA (Table 3). Moreover the loading difference is determined by flashing from a carbon dioxide partial pressure of 5 bars to a partial pressure of 0.01 bar at 20°C (column 7, lines 24 to 40 and column 8, Table 3). The loading difference at different pressures is not

directly related with the net absorption capacity determined at the same pressure but at different temperatures. Respondent's argument, based on theoretical considerations as set out in D11 to D13 that the absorption mechanism of alkanolamine solutions for carbon dioxide is substantially independent from the exact nature of the alkanolamine and the nature and pressure of the carbon dioxide containing gas is not supported by the experimental data. It follows from the above discussion of D25 that the accelerating effect of some additives is very much dependent upon the nature of the alkanolamine. The effect of the additives on the net absorption capacity at different temperatures of the solvent and the influence of the oxygen content of the carbon dioxide containing gas are not disclosed in D25, which contains no information suggesting that the washing process would be suitable for removing carbon dioxide from a combustion exhaust gas. It was therefore not obvious to the skilled person seeking to improve the efficiency of the Girbotol process described in D23 to apply the teaching of D25, based on the absorption of essentially oxygen-free gases. But even by doing so, he would not have arrived at a method according to claim 1 of the auxiliary request because none of these documents disclosed the use of compound (A) as absorbent. Only by combining D23 with D3 and D25 could one arrive at the claimed method, but there was no obvious reason why a skilled person would have done this, let alone, that he would have combined their teachings in such a way as to arrive at the claimed subject-matter.

14. D11, D12 and D13 are articles of Professor P.V.Danckwerts et al relating to the absorption of

carbon dioxide into solutions of alkalis and amines. In the introduction of D11 on page CE244 it is indicated that the removal of carbon dioxide from mixtures of gases by liquid absorbents is an important industrial process in several contexts such as the synthetic ammonia industry, the cleaning of natural gases and on a much smaller scale the cleaning of air before liquefaction thereof and the atmospheric control in submarines and space-craft. The respondent drew the conclusion therefrom that a skilled person would apply the teachings for the absorption of carbon dioxide from essentially oxygen-free gases such as disclosed in D25 also for the removal of carbon dioxide from oxygen containing gases such as combustion exhaust gas. In view of the known susceptibility of alkanolamines to oxidation (see D23, page 131, above the reaction equation) the board cannot accept this conclusion. Moreover, as already indicated above, the combined teaching of D25 and D23 would not lead to the method according to claim 1 of the auxiliary request. The respondent also made reference to Table XII of D11 (page CE255) disclosing amines according to group (Y) as defined in claim 1 and which are said to be of possible industrial significance for the absorption of carbon dioxide. Mixtures of such amines with other alkanolamines are, however, not indicated, let alone mixtures with primary alkanolamines according to group (A) of claim 1. It is also not apparent from D11 that the amines in said Table XII would act as an accelerator for group (A) alkanolamines.

15. In D12 a reaction-diffusion pattern in heterogeneous carbon dioxide - amine reaction is drawn (Fig. 1 on page T33). By this reaction a carbamate is formed in

the diffusion zone, which decomposes in the bulk solution producing bicarbonate ions and regenerating the amine, which diffuses back to the reaction zone next to the interface and reacts again with carbon dioxide. This reaction scheme is valid for both primary and secondary amines. The respondent drew the conclusion therefrom that the accelerating effects of additives on secondary amines also apply to primary amines and is independent of the actual composition of the gas to be treated. As already discussed above with respect Table 1 of D25 this conclusion is not supported by factual evidence. The board is unable to derive from D12 any incentive to add piperazine or any other amine of the group (Y) to an absorbing aqueous solution of a group (A) alkanolamine in order to solve the problem stated above.

16. D13 relates to the carbon dioxide absorption into amine-promoted potash solutions. In Table III on page 419 there is a list of promoting amines. This list comprises amines according to group (Y) of claim 1 but also primary alkanolamines such as MEA. From the fact that the listed amines promote the carbon dioxide absorption of a potash solution it cannot be derived that they would also promote the absorption of group (A) alkanolamines. As discussed before with respect to D25, the accelerating effect of particular amines is very much dependent upon the composition of the absorbing solution.
17. The other prior art documents on file do not contain information which, in combination with the teaching of the preceding documents, would render the subject-matter of claim 1 of the auxiliary request obvious.

Since they were no longer relied on during the oral proceedings there was no need to discuss them here.

18. For these reasons the board holds that the method according to claim 1 of the auxiliary request involves an inventive step within the meaning of Article 56 EPC. Claim 1 being allowable, the same applies to dependent claim 2, whose patentability is supported by that of claim 1.

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 claims 1 and 2 of the auxiliary request, submitted during the oral proceedings, with the description (and possibly the drawings) to be adapted.

The Registrar:

The Chairman:

A. Wallrodt

M. M. Eberhard