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Bezeichnung der Erfindung: The removal of hydrogen sulphide from gas streams
Title of invention:
Titre de l'invention :

Klassifikation / Classification / Classement : B 01 D 53/18

ENTSCHEIDUNG / DECISION

vom / of / du 17 February 1987

Anmelder / Applicant / Demandeur : Imperial Chemical Industries PLC

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Article 56

Kennwort / Keyword / Mot clé : "inventive step" (no)

Leitsatz / Headnote / Sommaire

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Case Number : T 301 /85

D E C I S I O N
of the Technical Board of Appeal 3.4.1
of 17 February 1987

Appellant : Imperial Chemical Industries PLC
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Decision under appeal : Decision of Examining Division 031 of the European Patent Office dated 29 May 1985 refusing European patent application No. 83 300 034.2 pursuant to Article 97(1) EPC

Composition of the Board :

Chairman : K. Lederer
Member : J. Roscoe
Member : O. Bossung

Summary of Facts and Submissions

I. European patent application No. 83 300 034.2 filed on 5 January 1983 (publication No. 84 410) was refused by a decision of the Examining Division 031 dated 29 May 1985. That decision was based on claims 1 to 10 as filed on 10 December 1984 and Claims 11 and 12 as originally filed.

II. Claim 1, the only independent claim, read as follows:

1. A process for removing hydrogen sulphide from a gas stream containing hydrogen sulphide and carbon dioxide by contacting the gas stream with an aqueous solution of an alkanolamine, characterised in that said contacting is carried out in a high surface area rotor rotating at such a speed as to subject said solution to an acceleration, expressed in a direction radial with respect to the axis of rotation, which is greater than the acceleration due to gravity.

III. The reason given for the refusal was that the subject-matter of Claim 1 did not involve an inventive step having regard to
US-A-4 299 801 (hereafter document (2)), and
GB-A-757 149 (hereafter document (3))
or to the document (3) and EP-A-0 002 568 (hereafter document (1)) taken in conjunction with the fact that alkanolamines are known to be effective hydrogen sulphide absorbents.

IV. An appeal was lodged against the decision on 19 June 1985 and the appeal fee duly paid, but no Statement of Grounds was filed until 8 January 1986.

V. On 12 February 1986 the Appellant filed an application for re-establishment of rights and paid the corresponding fee.

VI. By an interlocutory decision dated 27 June 1986 the Applicant was restored in his rights and his Statement of Grounds is to be considered as having been filed within the time limit provided by Article 108 EPC.

VII. With the Statement of Grounds the Appellant submitted a new set of claims numbered 1 to 11 and requested that the impugned decision be cancelled and the application allowed on the basis of this set of claims.

The only independent claim, Claim 1, is identical with the rejected Claim 1 and the Claims 2 to 11 correspond to Claims 3 to 12 valid at the time of rejection save for renumbering and the alteration of appendicies necessitated by the cancellation of the redundant Claim 2.

VIII. The Appellant argues mainly as follows.

(i) The broad problem on which the invention is based is, to use his own words "to improve the efficiency of the plant" and could have been attacked in a variety of ways e.g. by use of a different absorbent so that there could have been an inventive step in reformulating it as a narrow problem viz. such as to recognize the object as "a design of smaller apparatus".

This broad problem is highlighted by five papers, presented at an Offshore Technology Conference, collectively referred to hereafter as document (5), in the submitted copies of which certain passages have been emphasized by pencil markings.

(ii) These papers also confirm that there has been a long-felt want and unsatisfied need for reduction in the size of apparatus.

(iii) The view that the claimed process is lacking in inventive step is irreconcilable with the fact that, although both the alkanolamine process for the absorption of hydrogen sulphide as such and the centrifugal device and its use for absorbing the hydrogen sulphide in water, as described in document (3) has been known for a period of 30 years throughout which the skilled man had been seeking to improve the alkanolamine process, no one had hitherto proposed to apply the teaching of document (3) to that process when used on a stream containing both hydrogen sulphide and carbon dioxide.

(iv) From the absence of reference to centrifugal devices in the much more recent document (2) it would seem that its authors, persons skilled in the field, had either ignored the teaching of document (3) or tried centrifugal devices and found them deficient. It was thus apparent that a skilled man could see no advantage in their use in the alkanolamine process.

The Appellants on the other hand had shown that the use of such a device in the process provides surprising selectivity when using a gas stream containing both H_2S and CO_2.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is therefore admissible.

2. There is no objection to the current version of the claims as far as Article 123(2) EPC is concerned since it is adequately supported by the original disclosure.

3. The subject-matter of Claim 1 is novel.

3.1 Document (2) describes a process having all the features of the preamble of Claim 1 as being commonly employed, and refers to the process (i.e. the contacting) as being carried out in any suitable absorption apparatus such as a packed bed absorber, a spray contact apparatus, a bubble-cap tray absorber or the like and subsequently refers to absorption being effected as described in "The Gas Conditioning Fact Book", Chapter 3, Dow Chemical Co., 1962. There is however no suggestion in the book or in document (1) itself to effect the contacting in a rotor rotating so as to subject the solution to an acceleration.

3.2 The article by R. L. Pearce at pages 139-144 of Proceedings Annual Convention - Gas Processors Association, 1978, Vol. 57, (hereafter document (4)), drawn to the attention of the Examining Division by the Appellant, also discloses a process according to the preamble of Claim 1. Though the use of countercurrent absorbers and of different numbers of trays is discussed no mention is made of rotors.

3.3 Document (3) describes a process for removing H_2S from a gas stream containing H_2S and CO_2 by contacting the gas stream with an absorbing liquid. In this process contacting is carried out in a high surface area rotor (i.e. rotor 3 with filler 10 or plates 11) rotating at such a speed as to subject said liquid to an acceleration, expressed in a direction radial with respect to the axis of rotation, which is greater than the acceleration due to

gravity (cf. page 2, lines 53 to 58). In contrast to the subject-matter of Claim 1 the absorbing liquid is water and not an aqueous solution of alkanol-amine.

3.4 The remaining documents cited in examination and appeal proceedings do not come closer to the subject-matter of Claim 1, and need not be discussed for appreciating novelty.

4. The question now to be considered is whether the process according to Claim 1 involves an inventive step.

4.1 Claim 1 is based on a process for removing hydrogen sulphide from a gas containing both it and carbon dioxide as described in documents (4) and (2).

According to the original description the amine-based sweetening process, especially that using monoethanol-amine, frequently entails large gas throughputs and any process improvement making possible the use of small gas treatment apparatus is desirable, and, particularly where hydrogen sulphide is to be removed by treatment on a production platform at an offshore well, the weight and volume of the treatment plant are critical factors determining the feasibility of well-head treatment and rig design.

The Board is satisfied that the measures set out in the characterizing part of Claim 1, by intensifying the gas treatment, permit the use of much smaller apparatus for a given volume of gas to be treated.

It is additionally stated in the description that carbon dioxide is also absorbed by the alkanolamines and that when this gas is present in the gas to be treated (as in the process as claimed in Claim 1) and the recovered

hydrogen sulphide is to be used in certain chemical processes it is important that the absorption process should be selective for hydrogen sulphide against carbon dioxide. The Board is further satisfied that the claimed process is useful in this respect since it is more selective than the known processes.

Thus the problems faced by the Appellant were to find some way of modifying this particular absorption process so as to reduce the size of the apparatus required for it and to improve the selectivity of the absorption of hydrogen sulphide.

4.2 No contribution to inventive step is to be found in recognition of these problems.

The passage at col. 2, lines 1 to 19 of document (2) and at col. 1, lines 6 to 10 of page 141 of document (4) both point to the need in certain circumstances to increase the relative amount of hydrogen sulphide absorbed from a gas stream containing both it and carbon dioxide.

On the other hand to reduce the size of technical equipment wherever possible is one of the commonest aims in the engineering arts. Thus, contrary to the Appellant's opinion, the Board cannot see that the skilled man would need inventive considerations to narrow the broad problem "to improve the efficiency of the plant" to the more specific problem "to design of smaller apparatus". Consideration of the papers constituting document (5) newly introduced by the Appellant does not lead to a different conclusion. They show that the mounting of gas treatment apparatus on off-shore platforms was under active consideration in the mid-seventies, and it is evident that in such treatment apparatus for the removal of hydrogen sulphide from the gas would be required.

Indeed paper OTC 3641 refers to the use of gas removal units though without identifying the process employed in them. The papers in general suggest that there are serious engineering problems associated with the size of the plant for the total treatment. In the light of this it is concluded that the desirability of reducing the size of all items of equipment, including the gas removal units, was recognized in the art at the priority date.

4.3 In seeking a solution to the problem of size reduction the skilled man would take into consideration prior art documents which are directed to the problem of reducing the physical size of chemical apparatus in which gas treatment processes analogous to the alkanolamine process are performed. The size of the apparatus required for such processes is determined not only be the amount of gas to be treated but also by the efficiency with which the gas is brought into contact with and taken up by the absorbent liquid.

The basic problem of efficiently contacting a gas which is to be absorbed in and/or chemically reacted with a liquid or a component of such a liquid is well known and common to a wide range of processes. Conventional apparatus used to solve this problem of mass transfer include packed bed absorbers, columns of perforated and bubble-cap trays and spray contact apparatus.

4.4 Document (3) is expressly concerned with this problem and proposes the use of a rotor rotated so as to subject a liquid which is being used to treat a gas to a radial acceleration (centrifugal force) greater than that due to gravity.

By its references e.g. at page 1, lines 67-83 to the apparatus hitherto used, namely screening members and bubble caps, and their inadequacies it directly suggests to the skilled man the substitution of the described rotor for such apparatus. At page 2, lines 52-62 of the document a particularly intensive exchange of material between the liquid and gas phases is said to be obtained when the liquid phase is under the action of a centrifugal force amounting to a multiple of the acceleration due to gravity and the length of stay of the gas between the walls is said to be frequently less than 1 second. Such short contact times are stated to be advantageous for certain purposes. In one exemplary scrubbing process using an acceleration of 13 g a scrubbing capacity per unit volume 40 times as great as that obtainable with conventional apparatus was achieved.

It is abundantly clear that the teaching of this document is of quite general application (see **inter alia** page 6, lines 36-42) and there is nothing in it which could lead the skilled man to believe that it would not be effective if applied to the alkanolamine process. Appellant's contention that the description, at page 3, lines 47-87 of the use of two scrubbers and of the complicated recharging process (at page 5, lines 67-95) would discourage the skilled man from applying it to that process cannot be followed. In the quoted passages the use of the scrubbers is an optional expedient to be adopted in certain circumstances e.g. to allow scrubbers of smaller size to be used. The complicated recharging on the other hand is a proposal specific to a particular process in which ammonia and hydrogen sulphide are being scrubbed and is not seen to be relevant to the akanolamine process. It is the general principles enunciated in this document which would

attract the interest of the skilled man concerned with the akanolamine process rather than the details of particular and different applications thereof.

4.5 For these reasons the Board considers it would be prima facie obvious for the skilled man faced with the size problem to apply the teaching of document (3) to the process according to the preamble of Claim 1.

4.6 Faced with the additional problem of increasing the selectivity of the absorption of hydrogen sulphide against carbon dioxide the skilled man would now have to consider whether this process also enables that problem to be solved. This could be determined by carrying out experiments well within his competence and which he would be encouraged to undertake by information contained in documents (2), (3) and (4).

Thus at col. 1, lines 28-40 of document (2) it is pointed out that H_2S reacts almost instantaneously with an aqueous alkanolamine solution to form alkanolamine sulphide or hydrosulphide but that CO_2 takes a significantly finite time to react with the water in the solution to form carbonic acid prior to reacting with the alkanolamine to form alkanolammonium carbonate or bicarbonate. Hence the CO_2 tends to be taken up less readily by the solution so that H_2S can be selectively removed by controlling the time the gas is in contact with the absorbing solution.

In document (4) at page 141, col. 1, line 21 to col. 2, line 5 it is said to be an important attribute of methyldiethanolamine (an alkanolamine) that it selectively removes H_2S from gases containing it and CO_2 and that the selectivity is affected by contact time to such an extent that differential reaction rates between H_2S and CO_2

constitute a significant part of the selectivity. In fact some selectivity can be built into even monoethanolamine or caustic provided short contact times are utilized. A 20-fold increase in H_2S/CO_2 weight ratio in the liquid absorbent by reduction of contact time is claimed when using caustic.

In the light of this information the skilled man would, on reading the above-mentioned statement in document (3) relating to the very short times of contact available with the rotor described therein, and the remark on page 5 line 37 to 52 that rotor contacting is especially useful for selective scrubbing of H_2S, be led to expect use of the rotor to provide the desired enhanced selectivity.

4.7 The skilled man even if looking for a modification of the known process providing both an improvement in its selectivity for hydrogen sulphide absorption and a tangible reduction in size of apparatus, would therefore be led by the above considered prior art to the use of a rotor as claimed in Claim 1.

4.8 The Appellant's opinion that the absence from the recent document (2) of any reference to centrifugal devices shows that a skilled man could see no advantage in the use of such a device in the alkanolamine process is wholly unjustified, since the main concern of the authors of that document was with the regeneration of the absorbing solution, and not with the absorption step as such. There is moreover nothing in the document to suggest that the size of the apparatus used in that step was the subject of interest.

4.9 It is pleaded by the Appellant that the fact that the claimed invention meets a long-felt and hitherto unsatisfied want points to the existence of an inventive

step. In the Board's opinion however neither documents (5), published only in the second half of the seventies, nor any of the other documents in the proceedings shows there to have been a long-felt want for a reduction in the size of apparatus needed for the alkanolamine process. At most document (5) shows that the size of conventional apparatus required for processing gas offshore has recently posed problems. It makes no direct reference to the need to use the alkanolamine process in such processing nor does it suggest that the realisation of a smaller absorber would make a decisive contribution to solution of the general problem.

The available documents also do not establish that the posed problem of selectivity is one that has long resisted the efforts of the skilled man. Apart from this no evidence has been produced by the Appellant to show that the claimed process has been an immediate commercial success or that it has aroused widespread interest in the art as a practical answer to the alleged long-felt want.

5. In view of the above considerations, the Board finds that the subject-matter of Claim 1 does not involve an inventive step and the claim cannot therefore be allowed.

6. Claims 2 to 11 are dependent claims, the allowability of which is conditional on that of Claim 1. Furthermore, in view of the prior art the Board can find no patentable features in these claims.

Order

For these reasons, it is decided that:

The appeal is dismissed

The Registrar The Chairman