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D E C I S I O N
of 16 December 1993

Case Number: T 0404/90 - 3.4.2

Application Number: 82305783.1

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IPC: B01D 53/36, B01J 21/06

Language of the proceedings: EN

Title of invention:
Process for the reduction of the sulfur content in a gaseous stream

Patentee:
Mobil Oil Corporation

Opponent:
BASF Aktiengesellschaft, Ludwigshafen

Headword:
-

Relevant legal norms:
EPC Art. 56

Keyword:
"Inventive step - yes"

Decisions cited:
T 0261/87, T 0366/89

Catchword:
-



Case Number: T 0494 93 - 3.4.2

D E C I S I O N
of the Technical Board of Appeal 3.4.2
of 16 December 1993

Appellant: Mobil Oil Corporation
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New York 10017 (US)

Representative: Fisher, Adrian John
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London WC1A 2RA (GB)

Respondent: BASF Aktiengesellschaft, Ludwigshafen
(Opponent) -Patentabteilung - C6-
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Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office dated 23 March 1990
revoking European patent No. 0 078 690 pursuant to
Article 102(1) EPC.

Composition of the Board:

Chairman: E. Furrini
Members: C. Black
M.V.E. Lewenton

Summary of Facts and Submissions

- I. The Appellant is Proprietor of European patent No. 0 078 690.
- II. The patent was revoked by a decision of the Opposition Division on the ground that the subject-matter of the claims under consideration lacked an inventive step having regard to the disclosure in documents:
- D2: EP-A-0 038 741 and
D3: The Oil and Gas Journal, 12 March 1979, pages 76 to 80.

Also taken into account in the opposition proceedings was:

- D1: GB-A- 622 324.
- III. The present appeal lies against this decision.
- IV. At the end of oral proceedings, the Appellant requested that the decision under appeal be set aside and the patent maintained on the basis of amended documents handed over at the oral proceedings, of which Claim 1 reads as follows:

"A process for reducing the H₂S content of a gas stream by contacting the gas stream and oxygen at an elevated temperature with a catalyst containing a titanium-oxygen compound as an active ingredient, to convert H₂S directly into elemental sulfur, characterized in that the gas stream is subjected to catalytic hydrogenation/hydrolysis to convert substantially all the sulfur compounds of the gas stream into hydrogen sulfide prior to contacting said gas stream with said titanium

compound-containing catalyst, said gas stream contains less than 10% by volume of water and is at a temperature of 160 to 320°C when contacted with said titanium compound-containing catalyst, and said catalyst contains at least 80% by weight of titanium dioxide as the titanium-oxygen compound and has a specific surface area of 80 - 150 m²g⁻¹ and a pore volume of 0.3 - 0.45 cm³g⁻¹."

The Respondent requested that the appeal be dismissed.

V. The gist of the Appellant's argumentation was that the catalyst disclosed in D2 was stated to be suitable for the second stage of the Claus reaction and there was nothing in D2 to suggest to the skilled person that it might catalyse the direct oxidation of H₂S to S. The Opposition Division was therefore wrong on finding that the subject-matter of Claim 1 was obvious from a combination of the teachings of D2 and D3. D1 was of doubtful relevance in view of its age, because the situation here was analogous to those in Decisions T 261/87 and T 366/89, in each of which an old citation was left out of consideration. In any case on a proper reading, D1 could only be said to disclose as catalyst orthotitanic acid, dehydrated to some extent, but not a catalyst consisting of titanium dioxide or containing at least 80% by weight of titanium dioxide.

VI. The Respondent stressed that the disclosure in D3 differed from the subject-matter of Claim 1 only in the catalyst employed, and that a catalyst corresponding to that required by Claim 1 was known from D2. One application of this catalyst was in the second stage of the Claus reaction, a stated advantage being insensitivity to oxygen in amounts up to 0.5% and therefore not liable to be deactivated by sulphation. The same advantage is claimed in the patent in suit, page 4, lines 45 to 50 for the catalyst required by

Claim 1. It was, therefore, obvious to replace the Selectox catalyst known from D3 by the TiO_2 based catalyst known from D2, the more so because it was already known from D1 that catalysts based on titanium oxides could be used for the direct oxidation of H_2S to S. D1 discloses catalysts derived from orthotitanic acid by partial or complete chemical dehydration and therefore containing 80% more TiO_2 .

Reasons for the Decision

1. The appeal is admissible.
2. The features of Claim 1 now under consideration are derivable from those of Claims 2, 3 and 4 as originally filed. Claim 1 moreover contains all the features of the granted claim, plus additional features, so that its scope has not been extended. Amendments to the description make it wholly consistent with the amended claims. Accordingly, the requirements of Article 123(2) and (3) EPC are met.
3. The Opposition Division in its decision found the claims under consideration to be novel, and this is not at issue between the parties. In any case, novelty will become apparent from the consideration of inventive step.
4. The two-part formulation of Claim 1 is based on D1 and this is not unreasonable because D1 is the only document disclosing a direct, or better once-through, process in which H_2S is removed from a gas stream by catalytic conversion to sulphur using a titanium oxide compound. However, for the evaluation of inventive step, the Board prefers to start from D3, disclosing the Selectox

process, because the problem sought to be solved is that of overcoming disadvantages of known processes for removing H_2S from the tail gas from the Claus process (see pages 2 and 3 of the granted patent specification), and the Selectox process is a prime example of such a process.

5. In paragraph 10 of the decision under appeal, the Opposition Division explained how D3 disclosed all of the features of Claim 1 except for the catalyst used. This applies also in substance to the present Claim 1, and is not disputed by the parties. The Board can ^{also} agree with the statements of the Opposition Division regarding D3. The process according to D3 employs a catalyst identified by the proprietary name Selectox-32, and it is also undisputed that this catalyst is not based on any titanium compound so that its actual composition is unimportant.

6. According to the Appellant, the Selectox process suffers from certain disadvantages. In the first place it is inefficient at low H_2S concentrations and therefore cannot be used to improve further the H_2S removal by repeating the process one or more times. It is also an oxidising catalyst, entailing oxidation beyond S to SO_2 and SO_3 , and also, in view of the exothermic nature of the reaction, temperature runaway. It is for this reason that the reaction is carried out using sub-stoichiometric amounts of oxygen (D3, page 78, Figure 4 and page 79 under "Process Control"). This limits the efficiency of the process and moreover in view of the fluctuating H_2S content of the Claus tail-gas, parallel adjustment of the oxygen supply is required. The Board can accept that the object of the patent in suit is to overcome these disadvantages; in any case it is the constant endeavour of the average skilled person to seek alternatives to or improvements on known processes.

7. Now D2 discloses a catalyst based on titanium dioxide whose specific surface area and pore volume correspond closely to these required by the catalyst required by Claim 1. This catalyst is moreover stated to be suitable for the second stage of the Claus process (see page 2, lines 30 to 36 and page 5, line 25 to page 6, line 9). The Opposition Division, and the Respondent, conclude that it would be obvious to replace the Selectox-32 catalyst in the process disclosed in D3 by the catalyst known from D2 and thus arrive at the subject-matter of Claim 1.

8. However, as stated above, the catalyst disclosed in D2 is stated to be suitable for the second stage of the Claus process, which does not require oxygen, and there is no suggestion that it might be suitable also for the first stage of the Claus process, let alone for the oxidation of H₂S to S in a once-through process. The Board can therefore agree with the Appellant that the teaching of D2 is insufficient to permit the conclusion drawn by the Opposition Division.

9. But even taking the disclosure in D1 into consideration does not make good the said insufficiency. In the first place D1 is a relatively old document (published 1949) and its status as pertinent prior art may be judged in the light of the discussion of similar situations in Decisions T 261/87 and T 366/89. In T 261/87, point 8.2.3 of the Reasons for the Decision, the Board found that a 70 year old disclosure had come close to the invention under consideration, but had not given rise to a trend in the direction of the invention. This is also the case with the patent in suit where none of the prior art referred to pages 2 and 3 of the published document relates to the use of titanium compounds as catalysts for the conversion of H₂S to S. Similarly in T 366/89, in point 4.8 of the Reasons for the Decision,

the Board found that the old prior art had not given rise to a trend and more significantly in point 4.10 concluded that it was not likely that the person skilled in the art would turn back to an old technique relating to the quality of optical surfaces since the quality requirements were considerably lower than at the date of the patent under consideration. The situation in the patent in suit is analogous, the requirements for purity of emission gases having in recent years become much more stringent.

It is true that D1 does disclose a once-through process in which hydrogen sulphide is removed from a gas stream by catalytic oxidation, with air or oxygen, to sulphur, using as catalyst a titanium compound which may be an oxide, hydrated oxide or sulphide of titanium (page 2, lines 105 to 108). The said compound may be orthotitanic acid which may be activated by mere physical drying or by partial or complete chemical dehydration (page 3, lines 4 to 7).

It could be argued that in the case of complete chemical dehydration the product is substantially pure TiO_2 and that this might provide a hint to the skilled person, particularly in combination with the teaching of D2, in the direction of the claimed process. However, it is difficult to reconcile this interpretation of the single reference in D1 to complete chemical dehydration with the teaching of the remainder of the patent. In particular, page 3, lines 69 to 71 states that in general the raising of the catalyst temperature above that of normal operation reduces the catalytic activity. Normal operating temperatures appear from Examples I and II to be $100^\circ C$ or $200^\circ C$, whereas temperatures of around $800^\circ C$ are required for conversion of orthotitanic acid to TiO_2 . Nor would a temperature of $200^\circ C$ be sufficient to achieve a product which would contain at least 80% of

TiO₂ as is demonstrated by the weight loss experiments described in the Patentee's letter dated 15 January 1990 during the opposition proceedings.

Moreover, the efficiency of conversion of H₂S to S as given in Example I is only 52%. In Example II the initial efficiency of conversion is 88% but has fallen to 50% after 24 hours' operation.

Further, a stated advantage of the D2 catalyst is that it is not sensitive to oxygen in amounts up to 0.5% (page 7, lines 13 to 16). The Examples in D1 operate with oxygen contents of 1.2%, so that if lack of sensitivity to oxygen were a criterion for choice of catalyst, there would be no suggestion here to replace the D1 catalyst with the D2 catalyst. However, the Board can agree with the Appellant that while insensitivity to oxygen is important for the second stage of the Claus reaction, it is not a factor influencing the choice of an alternative to the Selectox catalyst, since the Selectox process requires the presence of oxygen.

Accordingly, even if the skilled person were to take D1 into consideration in spite of its age and the contra-indications as set out above, there is no teaching which alone or in combination with that of D2 or D3 would lead him towards the subject-matter of Claim 1.

10. The claimed process moreover meets the object of the invention in that it can take place in the presence of excess oxygen (page 4, lines 49, 50), therefore is more efficient than the Selectox process and also does not require constant adjustment of the oxygen feed. Further, the process is applicable to gas streams having a relatively low H₂S content (page 4, lines 14, 15). Finally, the catalyst employed is stated by the Appellant to be much cheaper than the Selectox catalyst.

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 on the basis of the documents presented at the oral proceedings.

The Registrar:

The Chairman:

P. Martorana

E. Turrini