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Datasheet for the decision of 17 July 2013

Case Number: T 0537/09 - 3.3.01

Application Number: 01944278.9

Publication Number: 1292587

IPC: C07D 301/10

Language of the proceedings: EN

Title of invention:
Process for operating the epoxidation of ethylene

Patent Proprietor:
Shell Internationale Research Maatschappij B.V.

Opponents:
BASF SE
THE DOW CHEMICAL COMPANY
Scientific Design Company Inc.

Headword:
Epoxidation of ethylene/SHELL

Relevant legal provisions:
EPC Art. 56, 54
RPBA Art. 13(1)

Keyword:
"Novelty - all requests - (yes) - specific increase not mentioned in the prior art"
"Inventive step - all requests - (no) - alleged improved effect (not shown) - obvious alternative"
"Main and auxiliary requests 1-3 - late-filed (yes) - admitted (yes) - not slowing down the procedure"
"Auxiliary request 4 - late-filed (yes) - admitted (no) - filed for tactical reasons"

Decisions cited:
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Catchword:
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Case Number: T 0537/09 - 3.3.01

D E C I S I O N
of the Technical Board of Appeal 3.3.01
of 17 July 2013

Appellant: (Patent Proprietor)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 18 December 2008 revoking European patent No. 1292587 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman: A. Lindner
Members: J.-B. Ousset
C.-P. Brandt

Summary of Facts and Submissions

I. This appeal lies from the decision of the opposition division revoking European patent No. 1 292 587.

II. The opposition division found that the subject-matter of the main request contravened Article 83 EPC, that the subject-matter of the first auxiliary request contravened Article 54 EPC in view of the disclosure of document (1), and that the subject-matter of auxiliary request 2 contravened Article 56 EPC.

III. The following cited documents are considered to be relevant for the present decision:

(1) EP-A-0 266 015
(2) EP-A-0 625 370
(3) EP-A-0 357 292
(4) EP-A-0 352 850
(5) EP-A-0 567 273
(8) GB-A-1 321 095
(18) EP-A-0 352 849
(30) Experimental results submitted by the appellant with its letter of 22 September 2008.
(40) "Calculation of catalyst age for examples 1 and 2 in document (5)", submitted as Annex 1 by the appellant with its letter of 11 July 2013.

IV. The present decision is based on the following requests field by the appellant during oral proceedings:

Claim 1 of the main request reads as follows:

"1. A process for the vapour phase oxidation of ethylene to ethylene oxide, which process comprises reacting a reaction mixture comprising ethylene and oxygen in the presence of a supported highly selective silver-based catalyst comprising a catalytically effective amount of silver, a promoting amount of rhenium or compound thereof, and a promoting amount of at least one further metal or compound thereof, by:

-operating at an initial operation phase wherein fresh catalyst is used, and

-operating at a further operation phase when a cumulative ethylene oxide production exceeds 0.1 kT ethylene oxide per m^3 of catalyst, wherein in said further operation phase the concentration of ethylene in the reaction mixture is raised by from 5 to 30 mol% of the concentration of ethylene used in the initial operation phase."

Claim 1 of the first auxiliary request reads as follows:

"1. A process for the vapour phase oxidation of ethylene to ethylene oxide, which process comprises reacting a reaction mixture comprising ethylene and oxygen in the presence of a supported highly selective silver-based catalyst comprising a catalytically effective amount of silver, a promoting amount of rhenium or compound thereof, and a promoting amount of at least one further metal or compound thereof, by:

-operating at an initial operation phase wherein fresh catalyst is used, and

-operating at a further operation phase when a cumulative ethylene oxide production exceeds 0.3 kT ethylene oxide per m^3 of catalyst, wherein in said further operation phase the concentration of ethylene

in the reaction mixture is raised by from 5 to 30 mol% of the concentration of ethylene used in the initial operation phase."

Claim 1 of the second auxiliary request reads as follows:

"1. A process for the vapour phase oxidation of ethylene to ethylene oxide, which process comprises reacting a reaction mixture comprising ethylene and oxygen in the presence of a supported highly selective silver-based catalyst comprising a catalytically effective amount of silver, a promoting amount of rhenium or compound thereof, and a promoting amount of at least one further metal or compound thereof, by:

-operating at an initial operation phase wherein fresh catalyst is used, and

-operating at a further operation phase when a cumulative ethylene oxide production exceeds 0.5 kT ethylene oxide per m^3 of catalyst, wherein in said further operation phase the concentration of ethylene in the reaction mixture is raised by from 5 to 30 mol% of the concentration of ethylene used in the initial operation phase."

Claim 1 of the third auxiliary request reads as follows:

"1. A process for the vapour phase oxidation of ethylene to ethylene oxide, which process comprises reacting a reaction mixture comprising ethylene and oxygen in the presence of a supported highly selective silver-based catalyst comprising a catalytically effective amount of silver, a promoting amount of

rhenium or compound thereof, and a promoting amount of at least one further metal or compound thereof, by:

-operating at an initial operation phase wherein fresh catalyst is used, and

-operating at a further operation phase when a cumulative ethylene oxide production exceeds 1 kT ethylene oxide per m^3 of catalyst, wherein in said further operation phase the concentration of ethylene in the reaction mixture is raised by from 5 to 30 mol% of the concentration of ethylene used in the initial operation phase."

Claim 1 of the fourth auxiliary request reads as follows:

"1. A process for the vapour phase oxidation of ethylene to ethylene oxide, which process comprises reacting a reaction mixture comprising ethylene and oxygen in the presence of a supported highly selective silver-based catalyst comprising a catalytically effective amount of silver, a promoting amount of rhenium or compound thereof, and a promoting amount of at least one further metal or compound thereof, by:

-operating at an initial operation phase wherein fresh catalyst is used, and

-operating at a further operation phase when a cumulative ethylene oxide production exceeds 0.01 kT ethylene oxide per m^3 of catalyst, wherein in said further operation phase the concentration of ethylene in the reaction mixture is increased."

V. The appellant's arguments, insofar as they are relevant for the present decision, can be summarised as follows:

- The main request and auxiliary requests 1 to 3 should be admitted into the proceedings, since they corresponded respectively to auxiliary requests 2, 4, 6 and 8 submitted in response to the objections of the board.

- The calculation in Annex 1 of the letter of 11 July 2013 should be admitted, since this calculation showed that the catalyst used in document (5) was not as old as the one of the patent in suit.

- Auxiliary request 4 should also be admitted, because it could be used as a basis for further amendments.

- Document (4) represented the closest prior art, because it disclosed a process in which the level of modulator was monitored in order to optimise the selectivity of the reaction and at the same time not to minimise the decline of the activity of the catalyst.

- Document (5) did not mention the same class of catalyst as the one of the patent in suit and the aging of the used catalyst was lower than the one used to achieve 0.1 kT production of ethylene oxide. Flammability problems were mentioned in document (5) wherein the experiments were run in a laboratory. This had to be absolutely avoided in plant scale.

– Document (8) taught that the increase in ethylene concentration led to an increase of the selectivity but also to a decrease in the velocity of the reaction.

– Document (3) also showed that an increase of the concentration of ethylene led to better activity but lowered the selectivity.

VI. The respondents' (opponents 1 to 3) arguments, insofar as they are relevant for the present decision, can be summarised as follows:

– The main request and auxiliary requests 1 to 3 were late-filed and did not represent a response to the board's objections. They had been filed one month before oral proceedings without any reasons for their late-filing, and had been renumbered during oral proceedings. Furthermore, they raised complex issues. They should not be admitted into the proceedings.

– Documents (1), (2), (4) and (18) were novelty-destroying.

– No improvement had been demonstrated by the appellant to show the presence of an inventive step and the mere provision of an alternative process was obvious in view of the disclosures of documents (1) and (5).

VII. The appellant requested that the decision under appeal be set aside and the patent maintained on the basis of

the main request or, alternatively, on the basis of one of auxiliary requests 1 to 4, all filed during oral proceedings on 17 July 2013.

VIII. The respondents requested that the appeal be dismissed.

IX. At the end of the oral proceedings, the decision of the board was announced.

Reasons for the Decision

1. The appeal is admissible.

2. Admissibility of the late-filed requests

Main and auxiliary requests 1 to 3

2.1 The main request corresponds to auxiliary request 2 which was regarded as not patentable by the first instance. Auxiliary requests 1 to 3 correspond respectively to auxiliary requests 4, 6 and 8 submitted by the appellant in response to the board's objections mentioned in the annex to the summons to oral proceedings. The amendments made by the appellant, namely the variation of the amount of ethylene oxide produced and the molar ratio increase of the concentration of ethylene, represent fair attempts to overcome the objections based on Article 123(3) and 56 EPC. Although it is indisputable that these requests were filed late and could have been submitted earlier, they did not create new complex issues necessitating the postponement of the oral proceedings, since the amendments made correspond to claims 2, 3, 4 and 11 as

granted. Considering that the respondents had opposed the patent in suit in its entirety, they cannot be surprised by such amendments.

Therefore, the board decides to admit these requests into the proceedings.

Auxiliary request 4

2.2 The appellant argued that this request should be admitted, since it could be used as a basis for further possible amendments.

The board does not agree. This request, although corresponding to the claims as granted, is considered as late-filed, since it was resubmitted as an auxiliary request during oral proceedings. Moreover, when compared to the preceding requests, the subject-matter of claim 1 of auxiliary request 4 clearly diverges, since the amount of ethylene added in the second operation phase is not limited anymore. This is contrary to the need for procedural economy mentioned in Article 13(1) of the Rules of Procedure of the Boards of Appeal (RPBA) (supplement to EPO Official Journal 1/2013, 38-49). Lastly, the board would like to add that the filing of auxiliary requests should aim at overcoming objections and should not be done for tactical reasons, which can only slow down the proceedings.

For these reasons, the board decides not to admit auxiliary request 4.

3. Admissibility of document (40)

3.1 The appellant argued that the respondents were allowed to present a similar calculation, during oral proceedings before the department of first instance, which was appended to the decision. Moreover, the current calculation should be admitted into the proceedings, since it showed that the catalyst of unspecified composition used in document (5) had an approximate cumulative ethylene oxide of less than 0.002 kT/m3 and was therefore a fresh catalyst, unlike the one used in the patent in suit.

First of all, the board would like to remark that the appellant could also have presented its own calculation before the department of first instance. By not doing so and instead waiting more than four years to submit it just one week before oral proceedings before the board, the appellant tried to introduce new evidence shortly before oral proceedings, without providing any reason for this late-filing. As a consequence, the respondents were deprived of the possibility to adequately react to this new evidence. In view of the current state of the proceedings, admitting this annex would be contrary to the requirement of procedural economy (Article 13(1) RPBA). Furthermore, this calculation was made to assess the age of the unspecified catalyst used in document (5), whereas the respondents' calculation presented before the department of first instance was based on document (1) considered as closest prior art. Thus, document (40) cannot be considered as a response to the respondents' calculation.

3.2 The board therefore does not admit document (40) into the proceedings.

4. Novelty

4.1 The respondents argued that Table 8 in example 10 of document (1) (see page 18) rendered the claimed matter not novel. They further added that on page 15, lines 2 to 4, the composition of the feed gas was slightly changed. Since example 10 described the production of ethylene oxide within a period of 59 days, it was inevitable that this change in the gas feed composition led to an increase in the concentration of ethylene, therefore rendering the claimed subject-matter not novel. A similar reasoning was also put forward by the respondents as to the content of document (2) and (4), according to which variations of the concentration in ethylene automatically took place. The respondent referred to the written submissions of 11 July 2007 and 17 September 2009.

The board does not dispute the fact that these documents relate to the same reaction, namely the formation of ethylene oxide starting from oxygen and ethylene. Moreover, the type of catalyst described in these documents is the same as the one used in claim 1 of the main request. However, the board does not concur with the respondents, because variations in the composition of the feed gas do not necessarily mean an increase in the concentration of ethylene and even if such an increase did take place, it would not necessarily range from 5 to 30% of the concentration of ethylene used in the initial phase. None of the cited

documents (1), (2) and (4) mentions such an increase in the concentration of ethylene.

The respondents also submitted that document (18) was novelty-destroying for the claimed subject-matter.

Similarly to documents (1), (2) and (4), document (18), although disclosing the preparation of ethylene oxide from a mixture of oxygen and ethylene by using a catalyst identical to the catalyst used in the process of claim 1 of the main request, does not disclose the increase in the concentration of ethylene mentioned in claim 1 of the main request.

4.2 The board thus concludes that the subject-matter of claim 1 of the main request is novel vis-à-vis the cited prior art. Since this distinguishing feature is present in all the requests on file, their novelty is also acknowledged (Article 54 EPC).

5. Inventive step

Main request

5.1 Determination of the closest prior art

5.1.1 Document (1) describes the preparation of ethylene oxide from oxygen and ethylene in the presence of a catalyst containing silver, a support, rhenium and at least one further metal (see page 2, lines 26 to 27). Since document (1) describes the preparation of the catalyst (see example 1, parts A to C on page 12 to 14), it can be concluded that a fresh catalyst is used in the process of document (1). The process described in

document (1) aims also at obtaining a catalyst having an optimum selectivity in combination with an improved longevity (activity) (see page 2, lines 22 to 25). The only difference with the subject-matter of claim 1 of the main request is that when the production of ethylene oxide exceeds 0.1 kT, the concentration of ethylene in the reaction mixture is raised by from 5 to 30 mol% of the concentration of ethylene used in the initial phase.

Document (4) describes also the preparation of ethylene oxide from oxygen and ethylene in the presence of a catalyst containing silver, a support, rhenium and at least one further metal (see page 2, lines 31 to 33). The reaction conditions for this catalyst are those known from the prior art (see page 3, lines 12 to 14) and thus those described in document (1) which was published before document (4). The process described therein aims also at finding trade-off conditions between the selectivity and the activity of the catalyst (see page 2, lines 20 to 22). The difference with the subject-matter of claim 1 of the main is thus the same as for document (1).

5.1.2 The appellant contended that document (4) was concerned with aging of catalysts, like the present invention. Document (1) was considered less pertinent, because in a preferred embodiment sulphur was present in the catalyst, which was not the case in the patent in suit (see document (1), page 11, and lines 57 to 58).

The board notes that document (1) also concerns the aging of the catalyst (see page 3, line 1). Even if sulphur can be added in the silver-containing catalyst

of document (1) as a co-promoting agent, the disclosure of document (1) is not limited to this specific embodiment.

5.1.3 In view of that, the board considers that document (1) is the closest prior art, since it aims at obtaining both conditions, namely optimum selectivity in combination with an improved activity.

5.2 Problem

The problem underlying the patent in suit can be seen in the provision of a process to make available ethylene oxide having a high selectivity in combination with a high activity (see page 2, lines 32 to 34 of the patent in suit).

5.3 Proposed solution

5.3.1 The appellant referred to document (30).

Example	Catalyst	Age kT/m3	O2, mol% in feed	C2H4, mol% in feed	S (%EO)	T(°C)
1	I	1.6	9.0	25	75.2	300
2	I	1.6	7.8	40	80.7	289
3	I	1.6	6.5	55	81.7	288
4	II	0.8	8.3	25	89.9	251
5	II	0.8	7.9	30	90.6	249
6	II	0.8	7.5	35	90.9	247

Catalyst I is a catalyst according to the invention having produced 1.6 kT/m3 of ethylene oxide and catalyst II is a catalyst having produced 0.8 kT/m3.

The appellant concluded that this Table showed an increase in selectivity as well as an increase in the activity of the catalyst according to the patent in suit when, at a certain level of aging, it was subjected to an increased ethylene concentration.

When a comparative test is used to show the presence of an improved effect, the comparison should be run in such a way that the alleged effect has its origin in the distinguishing feature. This is here not the case, since the age of the catalyst used in this test is different (1.6 kT/m^3 and 0.8 kT/m^3 as compared to 0.1 kT/m^3 in claim 1 of the main request) (see point 5.1.1 above).

5.3.2 Furthermore, the experimental results summarised in Table 1 of the patent in suit cannot help to show the presence of an improved effect. A comparison is made between a catalyst according to the invention (S-882) and a catalyst which contains neither rhenium nor rhenium co-promoter (see page 6, [0046] of the patent in suit). As mentioned previously, this differing feature is not identical to the distinguishing feature between document (1) and the process of claim 1 (see point 5.1.1 above).

5.4 The board therefore concluded that the problem was not plausibly solved and thus had to be reformulated in the provision of an alternative process to make available ethylene oxide by using a silver-containing catalyst.

Starting from document (1), the person skilled in the art would be aware of the disclosure of document (5) which describes the use of silver-containing catalysts (see page 2, lines 1 to 2) in order to produce ethylene oxide. Furthermore, the invention described in document (5) is applicable to any silver-containing catalyst capable of catalysing the controlled oxidation of ethylene with molecular oxygen to produce ethylene oxide (see page 4, lines 50 to 52) and consequently also to the silver-containing catalyst used in document (1). Additionally, this document discloses that the use of an ethylene ballast in the feed gas not only allows the pixilation reaction to take place but also - contrary to what is implied by related art - lowers the reaction temperature (thus increasing the activity) and provides a slightly higher selectivity (see page 2, lines 3 to 5). Hence, the person skilled in the art has a clear incentive starting from the process of document (1) to increase the concentration in ethylene as described in document (5) to obtain ethylene oxide without any inventive ingenuity. The range mentioned in claim 1, namely 5 to 30%, can therefore only be considered as arbitrary and would be considered by the person skilled in the art aware of the disclosure of document (5).

5.4.1 The appellant argued that the class of catalysts used in document (5) was different from the one of the patent in suit and that the aging of the said catalysts was lower than that of those of the present invention.

It is true that catalysts containing silver and rhenium are not mentioned in document (5). However, the same

document states that the invention described therein is not limited to the use of any specific silver metal-containing catalyst (see page 4, lines 55 to 56). Moreover, any silver metal-containing catalyst capable of catalysing the formation of ethylene oxide can be employed (see page 4, lines 50 to 52). Hence, the appellant's first argument is not conclusive. As to the age of the catalyst, the appellant repeatedly argued that the catalyst used in the "further operation phase" of the process of claim 1 is an aged catalyst. The board considers that this argument is not relevant, since the age of the catalyst is not a feature of the process described in claim 1 of the main request. This process only mentions that after a specific amount of ethylene oxide is produced, the concentration of ethylene in the feed gas must be increased. Whether the catalyst at this stage is aged or not is thus irrelevant. This second argument is also moot.

5.4.2 The appellant further observed that the person skilled in the art would be deterred from considering document (5) in view of the teachings of documents (3) and (8). Document (8) describes an increase in the selectivity of the reaction but a decrease in its velocity, thus a decrease in the activity (see page 2, lines 21 to 25) as well as example 1 on page 4 (see Table)). Document (3) shows (see page 5, "Testing the catalysts") that a higher concentration of ethylene in the feed (43% versus 29%) causes the selectivity to fall slightly whereas the activity increases (see Table 2 on page 6).

First of all, this argument is not convincing, since the problem underlying the patent in suit is the

provision of a mere alternative process and not a process in which both selectivity and activity (i.e. velocity) are increased (see point 5.4 above). Moreover, faced with documents, namely documents (8) and (3) on the one hand and document (5) on the other, disclosing diverging teaching as to the increase of the amount of ethylene in the feed gas, the skilled person would actually try to increase this amount as related in document (5), published after documents (3) and (8), since it states that the use of ethylene as ballast is effective, contrary to what is implied in the related art (see page 2, lines 5 to 7).

5.4.3 The appellant pointed to the flammability problems mentioned in the laboratory experiments of document (5) (see page 4, lines 23 to 27). These should absolutely be avoided in a plant process. This would also deter the person skilled in the art from considering document (5).

The board disagrees. It belongs to the skilled person's general knowledge to reduce or avoid flammability problems in such types of reaction. In addition, document (5) details the measures to be taken to avoid these problems (see page 4, lines 25 to 27, 42 to 46 and 47 to 49). This argument cannot therefore convince the board.

5.5 In view of that, the board concludes that the subject-matter of claim 1 of the main request is not inventive (Article 56 EPC).

Auxiliary requests 1 to 3

5.6 The patentee further contended that in these requests the catalyst used was more aged than the one used in document (1). This gap rendered the claimed matter not obvious vis-à-vis document (1) which used a less aged catalyst.

As to the age of the catalyst, the board refers to its arguments in point 5.5.1 above. Hence, the same inventive-step reasoning as applies to the main request is also applicable to the auxiliary requests, since they differ from the main request only in the amount of cumulative ethylene oxide produced after the further operation phase is initiated. Since no further relevant arguments have been put forward and since no improved or surprising effect can be attributed to this feature, it is considered as arbitrary and can therefore not establish an inventive step.

5.7 As a consequence, none of the requests on file meets the requirements of Article 56 EPC.

Order

For these reasons it is decided that:

1. The appeal is dismissed.

The Registrar: The Chairman:

M. Schalow A. Lindner