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D E C I S I O N
of 25 September 2003

Case Number: T 0097/00 - 3.3.1

Application Number: 92202517.6

Publication Number: 0533228

IPC: C07C 1/04

Language of the proceedings: EN

Title of invention:

Process for the activation of a catalyst

Patentee:

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

Opponent:

ExxonMobil Research and Engineering Company

Headword:

Catalyst activation/SHELL

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (no) - obvious solution"

Decisions cited:

T 0355/97

Catchword:

-



Case Number: T 0097/00 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 25 September 2003

Appellant: ExxonMobil Research and Engineering Company
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Representative: Troch, Geneviève
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Respondent: SHELL INTERNATIONALE RESEARCH
(Proprietor of the patent) MAATSCHAPPIJ B.V.
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Decision under appeal: Interlocutory decision of the Opposition
Division of the European Patent Office posted
2 December 1999 concerning maintenance of
European patent No. 0533228 in amended form.

Composition of the Board:

Chairman: A. J. Nuss
Members: P. P. Bracke
S. U. Hoffmann

Summary of Facts and Submissions

- I. The appeal lies from the Opposition Division's interlocutory decision that the set of 16 claims underlying the contested decision met the requirements of the EPC.

Claim 1 of the set of claims underlying the contested decision read:

"1. A process for the activation of a Fischer Tropsch catalyst, which process comprises contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a first stage at a pressure of up to 5 bar, rapidly increasing the pressure to at least 10 bar and contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a second stage at this pressure."

Claims 2 to 14 were dependent on Claim 1 and Claims 15 and 16 concerned a Fischer Tropsch catalyst activated by a process according to any of the preceding claims respectively a process for the preparation of hydrocarbons comprising contacting a mixture of carbon monoxide and hydrogen with a catalyst according to Claim 15.

In particular, the Opposition Division found that the claimed process was not obvious, since it could not be deduced from the cited prior art documents that the claimed process would lead to an increase of the activity, the selectivity and the stability of the catalysts.

II. With telefax of 25 August 2003 the Respondent (Proprietor of the patent) filed three sets of claims according to a first, a second and a third auxiliary request.

Claim 1 according to the first auxiliary request read:

"1. A process for the activation of a Fischer Tropsch catalyst, which process comprises contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a first stage at a pressure of up to 5 bar, rapidly increasing the pressure to at least 10 bar and contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a second stage at this pressure, the catalyst comprising a porous carrier selected from any suitable refractory metal oxide or silicates or a combination thereof."

Claim 1 according to the second auxiliary request read:

"1. A process for the activation of a Fischer Tropsch catalyst, which process comprises contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a first stage at a pressure of up to 5 bar, rapidly increasing the pressure to at least 10 bar and contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a second stage at this pressure, the catalyst comprising silica, alumina, titania or mixtures thereof as carrier."

Claim 1 according to the third auxiliary request read:

"1. A process for the activation of a Fischer Tropsch catalyst, which process comprises contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a first stage at a pressure of up to 5 bar, rapidly increasing the pressure to at least 10 bar and contacting the catalyst with substantially pure hydrogen gas or a mixture of hydrogen with one or more inert gasses in a second stage at this pressure, the catalyst comprising cobalt as a catalytically active metal and silica, alumina, titania or mixtures thereof as carrier."

III. The Appellant (Opponent) contested that the patent in suit met the requirement of sufficiency of disclosure and he contested the novelty of the claimed process. Moreover, the Appellant contested that the alleged technical effect was achieved over the complete claimed area, since processes wherein the hydrogen partial pressure in the second stage is equal or inferior to the hydrogen partial pressure in the first stage are also embraced within the claimed scope and it had not been shown that catalysts activated in that way have an increased reactivity, an improved stability and a higher selectivity to C₅₊ hydrocarbons. Furthermore, the Appellant argued that the claimed process was obviously derivable from document

(4) EP-A-0 168 894,

since the activation of Fischer Tropsch catalysts in two stages wherein the conditions of the first stage are softer than those in the second stage was known from this document.

- IV. The Respondent refuted the objection concerning insufficiency of disclosure and lack of novelty. Moreover, the Respondent submitted that by comparing the activity of the catalysts activated or reactivated as described in examples 2, 3, 4 and 6 with the activity of the catalyst activated as described in example 5 an increase of the activity, the stability and the selectivity of Fischer Tropsch catalysts had been shown. Since the prior art did not suggest to use a two-stage activation process for increasing the activity, stability and selectivity of such catalysts, the claimed process was inventive.
- V. The Appellant requested that the decision under appeal be set aside and that the European patent No. 0 533 228 be revoked.

The Respondent requested that the appeal be dismissed (main request) or that the patent be maintained on the basis of either the first, the second or the third auxiliary request all filed on 25 August 2003.

Reasons for the Decision

1. The appeal is admissible.

2. Since the Board came to the conclusion that neither the main request nor any of the first, the second and the third auxiliary requests meets the requirement of inventive step, it is superfluous to give any reasoning as to whether the requirements of Articles 123(2) and (3) and 83 EPC and the requirement of novelty are met.

3. *Inventive step*

- 3.1 Main request

In accordance with the "problem-solution approach" applied by the Boards of Appeal to assess inventive step on an objective basis, it is in particular necessary to establish the closest state of the art forming the starting point, to determine in the light thereof the technical problem which the invention addresses and successfully solves, and to examine the obviousness of the claimed solution to this problem in view of the state of the art.

- 3.1.1 It was not contested that document (4) represents the closest state of the art.

Document (4) discloses a process for the activation of a supported cobalt containing catalyst suitable for use in the preparation of hydrocarbons from a mixture of carbon monoxide and hydrogen by contacting such catalyst with hydrogen or a hydrogen-containing gas at

a hydrogen partial pressure between 0.001 and 75 bar in which during the activation the hydrogen partial pressure is increased gradually or step-wise from an initial hydrogen partial pressure $(P_{H_2})_i$ to an ultimate hydrogen partial pressure $(P_{H_2})_u$ such that $(P_{H_2})_u > 5 \times (P_{H_2})_i$. Thereby, $(P_{H_2})_i$ is preferably between 0.01 and 10 bar and $(P_{H_2})_u$ lies preferably between 0.1 and 20 bar (page 3, line 20 to page 4, line 15).

- 3.1.2 The Respondent submitted that, starting from document (4), the problem to be solved consisted in providing a process for activating a catalyst having an **increased** activity, an **improved** stability and a **higher** selectivity to C_{5+} hydrocarbons, as described in the patent in suit, column 2, lines 23 to 31.
- 3.1.3 The patent in suit claims to solve this problem by the process defined in Claim 1.
- 3.1.4 The next point to be considered in assessing inventive step is then whether it has been convincingly shown that by the process according to Claim 1 the problem underlying the patent in suit has effectively been solved.

The Appellant did not contest that the activation processes described in examples 2, 3, 4 and the last step in example 6 correspond with the claimed process and that, by comparison of these activation processes with the activation process described in example 5, an increased activity, an improved stability and a higher selectivity had been shown. However, the Appellant contested that with those exemplified processes it had been made plausible that with the complete range of

claimed processes an increased activity, an improved stability and a higher selectivity to C₅₊ hydrocarbons was obtained.

In the activation process described in example 2 and in the reactivation processes described in examples 3, 4 and 6 the catalyst was first treated with a mixture of hydrogen (1% v) in nitrogen; whilst maintaining the water content of the exhaust below a certain value the hydrogen content of the feed gas was increased gradually to 100% v; and once the water content in the exhaust gas was about 1000 ppmv, the gas pressure was increased sharply over a period of 15 minutes to 25 bar and maintained during 24 hours.

However, Claim 1 is not restricted to the activation conditions as described in those examples 2, 3, 4 and 6, but embraces any process wherein the catalyst is contacted at a pressure up to 5 bar in a first stage; the pressure is rapidly increased to at least 10 bar; and the catalyst is contacted in a second stage at this pressure. Consequently, processes wherein the hydrogen partial pressure in the second stage is equal or inferior to the hydrogen partial pressure in the first stage are also embraced within the wording of Claim 1, as long as the total pressure in the first stage is up to 5 bar and the total pressure in the second stage is at least 10 bar.

In the patent in suit it has only been shown that by activating a specific cobalt- and zirconium-containing catalyst in the specific conditions of examples 2, 3, 4 and 6 an increased activity, an improved stability and a higher selectivity to C₅₊ hydrocarbons was obtained

and the Respondent did not provide thereby any proof that such effect was obtained when activating a Fischer Tropsch catalyst under other activation conditions embraced within the wording of Claim 1, for example, by contacting the catalyst in the second stage at a hydrogen partial pressure equal or inferior to the hydrogen partial pressure in the first stage.

3.1.5 As the only evidence, the Respondent referred to examples 1A, 1B and 1C of document

(5) EP-A-0 152 652.

In particular, the Respondent argued that from a comparison of those examples it follows that by activating a Fischer Tropsch catalyst at a reduced partial hydrogen pressure a similar or even improved activity could be obtained as when activating the catalyst with pure hydrogen, since with the catalyst in example 1C, activated at a pressure of 1 bar and a partial hydrogen pressure of 0.1 bar, a CO conversion of 85% v was obtained, whereas such CO conversion with the catalyst 1A and 1B, which were activated with pure hydrogen at a pressure of 1.1 respectively 2, was only 72% v respectively 69% v.

However, since in those examples the catalysts were activated in a single stage at a constant pressure, the catalysts were not activated according to Claim 1. Therefore, those examples cannot provide any relevant evidence that the catalysts activated according to present Claim 1 provide a superior effect. Moreover, a comparison of example 1C with examples 1A and 1B might only possibly be suitable for showing an improved CO

conversion and not, however, an increased activity, an improved stability and a higher selectivity to C₅₊ hydrocarbons.

- 3.1.6 Additionally, the Respondent alleged that it was up to the Appellant to demonstrate that an increased activity, an improved stability and a higher selectivity to C₅₊ hydrocarbons was not obtained over the complete claimed scope.

However, according to the jurisprudence of the Boards of Appeal, each of the Parties to the proceedings carries the separate burden of proof for any fact they allege (see T 355/97 of 5 July 2000, not published in OJ EPO, point 2.5.1 of the reasons). Therefore, in the present case, the burden of proof for showing that the claimed method leads to the alleged and not supported advantageous effects mentioned in the patent in suit, rests upon the Respondent-Patentee. In the absence of any corroborating evidence that said advantageous effects are obtained, the allegation in the patent in suit of increased activity, improved stability and higher selectivity are unsubstantiated and, consequently, such alleged effects are not to be taken into account in assessing inventive step.

- 3.1.7 Therefore the objective, starting from document (4), can only be seen in providing a further method of activating Fischer Tropsch catalysts.

- 3.1.8 The patent in suit claims to solve this problem by the method defined in Claim 1.

That this problem (see point 3.1.7 above) is successfully solved by the method according to Claim 1 was never challenged, neither by the Appellant nor by the Board.

3.1.9 Therefore, it remains to be decided, whether in the light of the teachings of the cited documents a skilled person seeking to solve the above-mentioned problem would have arrived at the process of Claim 1 in an obvious way or not.

3.1.10 From document (4) it may be deduced (see point 3.1.1 above) that Fischer Tropsch catalysts may be activated by contacting them with hydrogen in more than one stage wherein the catalyst is contacted in a subsequent stage at a higher partial hydrogen pressure than in the previous stage (see point 3.1.1 above). It also follows from document (4) that in the subsequent stage the partial hydrogen pressure may be selected from 0.1 to 20 bar, that in the previous stage the partial hydrogen pressure may be selected from 0.01 to 10 bar and that the catalysts may be contacted with pure hydrogen gas or hydrogen-containing gas.

Thus, in order to solve the above stated problem, a skilled person only had to choose a pressure for the first step and for the second step within the limits of the pressure ranges disclosed in document (4). As it was known from document (4) that by working within the pressure limits disclosed therein a Fischer Tropsch catalyst may be activated, a skilled person would have had a reasonable expectation of success by working within those pressure limits.

It is true that document (4) mentions a step-wise increase of the initial to the ultimate partial hydrogen pressure and that there is no explicit mention of a particular period of time for achieving the increase of the pressure.

However, according to the case law of the Boards of Appeal, features which do not contribute to the solution of the problem are not to be considered in assessing inventive step of a combination of features (see Case Law of the Boards of Appeal of the EPO, 4th edition 2001, point I.D.6.5).

As the Appellant never provided any kind of proof that a rapid increase of the pressure from the first stage to the second stage instead of a step-wise increase of the pressure would be significantly different in the sense of having any influence on the activity, the stability or the selectivity of the catalyst, this feature is not to be taken into consideration in assessing inventive step. In any case, the Board has no reason to believe that this is to be regarded as being of any importance for solving the underlying technical problem, all the more so since according to the patent in suit the term "rapidly" is merely intended to express a period of time that is short in comparison to the overall duration of the process (see column 4, lines 21 to 24).

3.1.11 Since from the disclosure of document (4) a skilled person would have carried out the process of Claim 1 with a reasonable expectation of success to activate a Fischer Tropsch catalyst, the method of Claim 1 is an obvious solution to the problem underlying the patent

in suit. Therefore, Claim 1 and, thus, the main request cannot be considered to meet the requirement of inventive step.

3.2 First, second and third auxiliary request

The method of Claim 1 according to the first, second and third auxiliary request differs from the method of Claim 1 according to the main request by the further specification of the metal and/or the carrier of the catalyst.

Since, however, the **process features** of the claimed method are obviously derivable from the disclosure of document (4) and for the further specification of the metal and/or the carrier of the catalyst an effect has not been shown, these features also cannot form the basis for an inventive step.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar:

The Chairman:

N. Maslin

A. Nuss