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D E C I S I O N
of 14 May 1998

Case Number: T 0724/93 - 3.3.2

Application Number: 90201402.6

Publication Number: 0396206

IPC: B01J 37/20

Language of the proceedings: EN

Title of invention:

Process for the preparation of a sulphided catalyst and use of said catalyst

Applicant:

Akzo Nobel N.V.

Opponent:

-

Headword:

Sulphided catalyst/AKZO

Relevant legal provisions:

EPC Art. 123(2), 54

Keyword:

"Novelty (yes) "

Decisions cited:

-

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 0724/93 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 14 May 1998

Appellant: Akzo Nobel N.V.
Velperweg 76
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Representative: Schalkwijk, Pieter Cornelis
Akzo Nobel N.V.
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Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 13 April 1993
refusing European patent application
No. 90 201 402.6 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: P. A. M. Lançon
Members: M. M. Eberhard
R. E. Teschemacher

Summary of Facts and Submissions

- I. European patent application No. 90 201 402.6 was refused by a decision of the Examining Division. The decision was based on amended claims 1-4 filed on 22 December 1992.
- II. The grounds for refusal were that claim 1 lacked novelty over the disclosure of either of documents EP-A-0 130 850 (D1) and EP-A-0 153 233 (D2). The Examining Division took the view that since there was a priori no chemical difference between hydrogen sulphide generated in the activation step and hydrogen sulphide added directly to the activation step from an external source, there appeared to be only a difference in the origin of the hydrogen sulphide. This difference was nevertheless of no importance as regards its further activity in the claimed process. In the absence of any clear delimitation between the reactants defined in claim 1 and those used in D1 or D2, claim 1 was not novel.
- III. The Appellant lodged an appeal against this decision and filed a statement of grounds of appeal in due time. Oral proceedings were held on 14 May 1998. At the oral proceedings the Appellant submitted three sets of amended claims 1-3 as the main request, first auxiliary request and second auxiliary request, in replacement of all the previous requests. Claim 1 of the main request reads as follows:

"1. A process for the preparation of a sulphided catalyst which is suitable for use in subsequent catalytic hydrotreatment of a hydrocarbon-containing feed, in which process a catalyst containing a carrier material with deposited thereon one or more catalytically active metals or compounds of metals

selected from iron, cobalt, nickel, tungsten, molybdenum, chromium, vanadium, copper, palladium and platinum is presulphided ex situ and the resulting material is activated by being contacted in situ at elevated temperature with a hydrogen gas-containing stream, characterized in that in the in situ step the catalyst material is contacted at a temperature in the range of 100° to 650°C with hydrogen to which a sulphiding agent has been added, or at a temperature in the range of 100° to 500°C with hydrogen combined with a hydrocarbon-containing feed containing an added sulphiding agent, said sulphiding agent being selected from the group consisting of hydrogen sulphide and compounds that under the prevailing conditions are decomposable into hydrogen sulphide, it being understood that when contacting takes place with a mixture of hydrogen and hydrogen sulphide, said hydrogen sulphide does not originate, or not exclusively originate, from hydrogen sulphide which is generated in the in situ step and then recycled."

IV. The Appellant presented inter alia the following arguments:

Concerning the allowability of the amendments to claim 1 of the main request, the Appellant argued that the anticipation by D1 or D2 was accidental since in these documents hydrogen sulphide was recycled in order to control the temperature in the in situ activation step, whereas in the claimed process hydrogen sulphide was added in the in situ step as sulphiding agent. It was common practice to disclaim accidental anticipation. According to decision T 0433/86, a specific prior art may be excluded from a claim by means of a disclaimer even in the absence of support for the excluded matter in the original application. The disclaimer as formulated in claim 1 was the most accurate disclaimer possible since it excluded

sulphidation in situ by H₂S originating exclusively from recycling as described in D1 or D2. This disclaimer did not make any contribution to the claimed invention and thus did not contravene Article 123(2) EPC.

As regards novelty, the Appellant pointed out that it was acknowledged in the decision under appeal that the subject-matter of claim 1 differed from the process of D1 or D2 by the origin of the hydrogen sulphide. Introducing H₂S from an external source in addition to recycled H₂S rendered the claimed process novel over the process of D1 or D2. Furthermore, using hydrogen sulphide coming from an external source had two advantages: this allowed reduction of the down-time of the unit and control of the amount of H₂S throughout the activation step.

- V. The Appellant requested that the decision under appeal be set aside and that the case be remitted to the Examining Division for further prosecution on the basis of the main request as submitted during the oral proceedings, alternatively on the basis of one of the two auxiliary requests as submitted during the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. *Amendments (main request)*

Claim 1 of the main request is based on the combination of claim 1 as originally filed with features of the original description. In particular, the list of metals and the temperature ranges indicated in claim 1 are disclosed on page 2, lines 27-29, page 4, last line,

and page 5, second paragraph respectively of the original description. It is directly and unambiguously derivable from the second paragraph of page 2, read in conjunction with the sentence bridging pages 1 and 2, that activation of the catalyst material occurs during the in situ step. In the Appellant's view, the additional features of claim 1, ie "it being understood that when contacting takes place with a mixture of hydrogen and hydrogen sulphide, said hydrogen sulphide does not originate, or not exclusively originate, from hydrogen sulphide which is generated in the in situ step and then recycled", are allowable as a disclaimer (see point IV). However, considering that a disclaimer is permissible only in very exceptional circumstances, the Board has examined whether these features are disclosed in the original application.

According to page 1 of the description, in actual practice sulphiding of the catalytically active metals is carried out in situ, ie in the hydrotreatment reactor. The fresh catalyst is contacted at elevated temperature with a hydrogen gas stream mixed with a sulphiding agent or a hydrogen gas stream combined with a spiked feed, said sulphiding agent being either hydrogen sulphide or a compound that under the prevailing conditions is decomposable into hydrogen sulphide (see page 1, lines 13-27). As confirmed by the Appellant at the oral proceedings, when H₂S is the sulfiding agent it has to be supplied to the reactor from an external source. The catalyst being fresh, no H₂S is generated during the reaction.

It is further stated on page 1 that a recent development in this field is the so-called ex situ presulphiding. This method involves an ex situ treatment (impregnation) of the fresh catalyst with a sulphur compound and, in a subsequent step, an in situ treatment with hydrogen at elevated temperature to

activate the catalyst, ie to bring it into the sulphided state (see page 1, last paragraph and page 2, lines 1-2). Reference is made to D1 and D2 to illustrate this second method. Each of these documents further discloses that H_2S is generated in the in situ activation step and that the resulting gas mixture of hydrogen and H_2S can possibly be recycled to the said step (see D1, page 13, line 22 to page 14, line 4; D2, page 12, lines 8-23).

According to page 2 of the original description, it has surprisingly been found that sulphided catalysts of increased activity can be obtained by a preparative process that **combines the two methods described**. In the process of the invention a fresh catalyst is presulphided with a sulphur compound ex situ, in the known manner described in D1 or D2, and in a subsequent step the resulting material is contacted in situ at elevated temperature with a mixture of hydrogen and a sulphiding agent or hydrogen combined with a spiked feed, under the known sulphiding conditions (see page 2, second paragraph). As in the known in situ sulphiding method using H_2S as the sulphiding agent H_2S has to be supplied to the reactor from an external source, it is directly and unambiguously derivable from the combination of the method of D1 (or D2) with the known in situ method that at least part of the hydrogen sulphide added to hydrogen in the second step of the claimed process is supplied from an external source (ie from a source other than the H_2S generated in the activation step and possibly recycled as described in D1 or D2). Therefore, it is implicit from the original description that H_2S does not exclusively originate from a possible recycling of H_2S generated in the activation step. Thus, amended claim 1 meets the requirements of Article 123(2) EPC.

As claims 2 and 3 correspond to original claims 2 and 3, they also fulfil the provisions of Article 123(2) EPC.

3. *Novelty (main request)*

Each of D1 and D2 discloses a process comprising all the features recited in the preamble of claim 1 of the main request. Furthermore, the temperature at which the catalyst material is contacted with hydrogen and recycled hydrogen sulphide in the activation step of D1 or D2 falls within the claimed range of 100 to 650°C (see D1, line 22 to page 14, line 6; D2, page 12, lines 8 to 25). As according to claim 1 the hydrogen sulphide used in the in situ activation step does not exclusively originate from the recycled H₂S generated in the said step, which means that H₂S coming from another source has to be introduced in this step, the claimed process differs from that of D1 or D2 by this external supply of H₂S. It cannot be inferred from the fact that there is a priori no chemical difference between the reactants used in D1/D2 and those defined in claim 1 that the two ways these reactants are brought into the reactor are also identical. Recycling H₂S which is generated in the in situ activation step to the said step, and supplying H₂S from another source to the in situ activation step, clearly represent two different ways of supplying H₂S to the reactor and thus two different process features. Therefore, the subject-matter of claim 1 is new over D1 and D2.

The subject-matter of claim 1 according to the main request also meets the requirement of novelty with respect to the third document cited in the search report.

4. The question of whether the method according to claim 1 of the main request involves an inventive step has not been considered in the course of the examining procedure. The technical problem solved by the claimed process with respect to the closest prior art has not yet been defined. In these circumstances, the Board finds it appropriate, in accordance with Article 111(1) EPC, to remit the case to the Examining Division for further prosecution and thus to allow the Appellant's request for remittal to the first instance for further prosecution.

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 for further prosecution on the basis of claims 1 to 3 according to the main request as submitted during the oral proceedings.

The Registrar:


P. Mastorana

The Chairman:


P. A. M. Lançon

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HEB.

