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**Datasheet for the decision
of 26 June 2012**

Case Number: T 0377/09 - 3.3.07
Application Number: 00201039.5
Publication Number: 1043069
IPC: B01J 37/20, C10G 45/08
Language of the proceedings: EN

Title of invention:

Process for sulphiding a hydrotreating catalyst comprising an organic compound comprising N and carbonyl

Patent Proprietors:

Albemarle Netherlands B.V.

Opponents:

Shell Internationale Research Maatschappij B.V.

Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (no) - all requests - obvious improvement"

Decisions cited:

-

Catchword:

-



Case Number: T 0377/09 - 3.3.07

D E C I S I O N
of the Technical Board of Appeal 3.3.07
of 26 June 2012

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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted 8 December 2008
revoking European patent No. 1043069 pursuant
to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman: J. Riolo
Members: F. Rousseau
P. Schmitz

Summary of Facts and Submissions

I. The appeal by the Patent Proprietors (Appellants) lies from the decision of the opposition division revoking European patent No. EP-B-1 043 069. The decision announced at the oral proceedings on 6 November 2008, was based on a Main Request and an Auxiliary Request submitted during the oral proceedings.

II. Independent claim 1 of the Main Request read as follows:

"1. A process for preparing a sulphided hydrotreating catalyst comprising the steps of:

1. providing a catalyst carrier comprising at least 50 wt.% of alumina and at least one hydrogenation metal component from group VIB or Group VIII, wherein the alumina comprises a transition alumina;
2. subsequently compositing the catalyst carrier with an organic compound comprising at least one covalently bonded nitrogen atom and at least one carbonyl moiety, without subsequent calcining, such that the obtained catalyst has a molar ratio of the organic compound to the total hydrogenation metal content of at least 0.01:1; and
3. Subjecting the obtained catalyst to a sulphidation step."

III. Amended claim 1 of the Auxiliary Request defined that the catalyst carrier contained at least one hydrogenation metal component from group VIB and at least one hydrogenation metal component from Group VIII.

IV. The patent had been opposed on the grounds that its subject-matter extended beyond the content of the application as filed (Article 100(c) EPC), lacked novelty and inventive step (Article 100(a) EPC) and was insufficiently disclosed (Article 100(b) EPC). The opposition was based *inter alia* on documents:

D1: EP-A-0 482 818

D2: WO 95/31280

D6: US-A-3 909 450 and

D8: WO 96/41848.

V. The Opponents further relied on Document D9 (an article in Japanese language: Sekiyu Gakkaishi, Vol. 39, No. 2, 1996, pages 158-165) and a translation thereof in English) which had been submitted one month before the oral proceedings before the opposition division.

VI. According to the written decision posted on 08 December 2008, the amended sets of claims met the requirements of Articles 123(2), (3) and 84 EPC. Late-filed document D9, which was in view of its relevance introduced into the proceedings, was found to anticipate claim 1 of the Main Request. Novelty of claim 1 of the Auxiliary Request was acknowledged, because D9 did not disclose the addition of both metals of group VIB and group VIII to the alumina carrier prior to adding the organic additive comprising at least one covalently bonded N atom and at least one carbonyl moiety, which in D9 was nitrilotriacetic acid. As far as inventive step was concerned, no data were available which established any advantage or effect brought about by the addition of the organic additive subsequent to the incorporation of

a metal of group VIB and a non-noble metal of group VIII. The Opponents had furthermore shown with letter of 03 October 2008 that urea, which fell within the definition of the organic additive given in claim 1, led to a decrease of the relative volume activity for hydrodenitrogenation, giving rise to a serious doubt as to which organic additives according to claim 1 gave the alleged increase of activity. As the subsequent addition of the organic additive to the metals impregnated catalyst composition was a common practice in the art, as reflected by the teachings of D1, D2 and D6, an inventive step was denied for the Auxiliary Request.

VII. With the statement setting out the grounds of appeal dated 19 April 2009, the Appellants submitted 3 sets of claims constituting their Main, First and Second Auxiliary Requests. In comparison to the claims underlying the contested decision, claim 1 of all those requests was restricted *inter alia* by defining that the organic additive was nitrilotriacetic acid (NTA), ethylenediaminetetraacetic acid (EDTA) or diethylenetriaminepentaacetic.

VIII. A response to the statement setting out the grounds of appeal was submitted by the Opponents (Respondents) on 26 August 2009.

IX. In preparation for the oral proceedings, the Board in a facsimile letter sent on 25 May 2012 indicated *inter alia* that D2, which had been proposed by the Respondents as starting point for assessing inventive step, appeared to represent a prior art which was closer to the claimed subject-matter than D9. D9

appeared to relate to a more remote process, as first the hydrogenation metal components used in that document were not provided on the carrier in the form of their oxides, and second all hydrogenation metal components were not compounded with the carrier prior to adding the chelating agent. The question also arose whether, when starting from D2 as closest prior art, the teaching provided in D8 or D1 would lead in an obvious manner to the claimed subject-matter.

- X. The Appellants submitted on 29 May 2012 in replacement of the sets of claims then on file, nine sets of claims constituting their Main and First to Eighth Auxiliary Requests. Claims 1 of these Requests read as follows (compared to claim 1 as granted, the deletions are marked in strikethrough and the additions in bold):

Main Request

"1. A process for preparing a sulphided hydrotreating catalyst comprising the steps of:

1. providing a catalyst carrier comprising at least 50 wt.% of alumina and at least one hydrogenation metal component from group VIB ~~or Group VIII~~ **as stable oxide and at least one hydrogenation non-noble metal component from group VIII as stable oxide, wherein the alumina comprises a transition alumina, which process comprises a calcination step before compositing with the organic compound,**
2. subsequently compositing the catalyst carrier with an organic compound ~~comprising at least one covalently bonded nitrogen atom~~

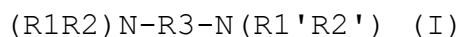
~~and at least one carbonyl moiety~~ **being an aminopolycarboxylic acid**, without subsequent calcining, such that the obtained catalyst has a **molar** ratio of the organic compound to the total hydrogenation metal content of at least 0.01:1, and

3. Subjecting the obtained catalyst to a sulphidation step."

Auxiliary Request 1

The wording of claim 1 of Auxiliary Request 1 differed from that of the Main Request only in that the definition of the aminopolycarboxylic in step 2 was restricted as follows:

"wherein the organic compound is an aminopolycarboxylic acid of formula (I):



wherein R₁, R₂, R₁' and R₂' are independently selected from alkyl, alkenyl, and allyl with up to 10 carbon atoms optionally substituted with one or more groups selected from carbonyl, carboxyl, ester, ether, amino, or amido, and R₃ is an alkylene group with up to 10 carbon atoms which may be interrupted by -O- or -NR₄-, R₄ being selected from the same group as indicated above for R₁, which R₃ alkylene group may be substituted with one or more groups selected from carbonyl, carboxyl, ester, ether, amino, or amido, with the proviso that the organic compound of formula (I) comprises at least one carbonyl moiety and wherein at least two of R₁, R₂, R₁' and R₂' have the formula -R₅-COOX, wherein R₅ is an alkylene group having 1-4 carbon atoms, and X is hydrogen or another

cation, where if X is a multivalent cation, one X can adhere to two or more -R5-COO groups or nitrilotriacetic acid."

Auxiliary Request 2

The wording of claim 1 of Auxiliary Request 2 differed from that of the Main Request only in that the organic compound was defined in step 2 to be nitrilotriacetic acid, ethylenediamine(tetra)acetic acid or diethylenetriaminepentaacetic acid.

Auxiliary Request 3

The wording of claim 1 of Auxiliary Request 3 differed from that of the Main Request only in that the feature "which process comprises a calcination step before compositing with the organic compound" had been replaced by "which catalyst carrier is a conventional freshly prepared hydrotreating catalyst or a used hydrotreating catalyst which has been regenerated".

Auxiliary Request 4

The wording of claim 1 of Auxiliary Request 4 differed from that of the Main Request only in that the feature "which process comprises a calcination step before compositing with the organic compound" had been replaced by "which is a conventional hydrotreating catalyst being either a freshly prepared hydrotreating catalyst or a used hydrotreating catalyst which has been regenerated, wherein the freshly prepared catalyst is prepared -by impregnating a carrier with a solution comprising the hydrogenation metal components in a

first process step and calcining the thus obtained product at a temperature sufficient to convert the hydrogenation metal components into a stable oxide, or is freshly prepared in a process wherein the hydrogenation metal components are mixed wholly or in part with the carrier prior to a shaping step being carried out wherein hydrogenation metals components are deposited on the not yet shaped carrier powder or (hydro)gel, after which the shaping step is carried out, followed by a calcination step applied to the shaped particles."

Auxiliary Requests 5 and 6

Claim 1 to any of Auxiliary Requests 5 and 6 corresponded to claim 1 of Auxiliary Request 3, in which the definition of the organic compound had been restricted to that provided in claim 1 of Auxiliary Requests 1 and 2, respectively.

Auxiliary Requests 7 and 8

Claim 1 of any of Auxiliary Requests 7 and 8 corresponded to claim 1 of Auxiliary Request 4, in which the definition of the organic compound had been restricted to that provided in claim 1 of Auxiliary Requests 1 and 2, respectively.

XI. Oral proceedings before the Board took place on 26 June 2012, at the end of which the decision of the Board was announced.

XII. The Appellants' arguments that are relevant for the present decision can be summarized as follows:

- (a) In the Main and any of Auxiliary Requests 1, 3, 4, 5 and 7, the return to a broader definition of the organic compound in the second step of claim 1, which was fully supported by the content of the application as filed, was adequate in view of the description of the invention made in the patent in suit. Moreover, the return to a broader definition of the organic compound did not raise complex issues, as it was implicit and not disputed by the Opponents / Respondents, that all compounds belonging to the particular group of organic compounds defined in those requests fulfilled the same function, i.e. they served as chelating agents forming complexes with the hydrogenation metals. The other amendments contained in the new requests concerned the steps by which the catalyst carrier to be composited with the chelating agent was obtained. They had a basis in the application as filed, did not induce any ambiguity and allow to overcome the objection raised by the Respondents that the wording "stable oxide" present in the previous requests for defining the state of the hydrogenation metals on the carrier was lacking clarity. Hence, the new requests should be admitted in the proceedings.
- (b) As to the inventive step, D2 constituted the closest prior art. The claimed subject-matter according to any of the requests differed from that of D2 in that the aminopolycarboxylic acid chelating agent was not removed by a calcination step, as was mandatory in D2, which meant that the compound to be sulphided comprised a mole ratio of

chelating agent to hydrogenation metal of at least 0.01:1.

- (c) As already indicated in the patent in suit, the problem solved by the presence of the aminopolycarboxylic acid in the compound to be sulphided was the provision of hydrogenation catalysts having higher activity, which could be obtained in a simpler and cheaper manner. The improved activity resulting from the omission of the calcination step was demonstrated by the comparative test described in paragraphs [0035] to [0041] of the patent in suit, which provided a direct comparison of the claimed process with that of D2 and in which EDTA had been used as chelating agent. It was pointed out in this respect that the hydrogenation metals employed in this test report were in their oxide form, as shown by the fact that the hydrotreating catalyst to be treated with the chelating agent was described to be conventional, and obtained in accordance with the teaching of D2. Moreover, as the process had been restricted to those using aminopolycarboxylic acids as chelating agent, i.e. additives having a similar structure as EDTA, it was credible that the increase of activity for hydrodenitrogenation brought about by the absence of calcination in the presence of EDTA, was also obtained in the presence of any other aminopolycarboxylic acid.
- (d) Starting from D2 and wishing to solve the problem of improving the catalyst activity, the skilled person would not have suppressed the calcination step, which was described in D2 to be mandatory.

Moreover, D2 contained no indication that an increase of activity for hydrodenitrogenation could be obtained by eliminating the calcination step, the improvement of activity obtained in D2 being solely reported in this document to result from an interaction between at least amorphous γ -Alumina, chelating agent and catalytically active components. Moreover, the aging step required by D2 was not a mandatory feature of the present claimed process and even provided as shown in the patent in suit lower activity. Hence, the skilled person, would not have suppressed the mandatory calcination step in D2 in expectation of succeeding to increase the catalyst activity for hydrodenitrogenation.

- (e) As to document D8, that prior art employed a different class of additives, namely glycols, which group of compounds did not embrace aminopolycarboxylic acids, in particular EDTA. Hence, the teaching in respect of D8 to not calcine the carrier treated with glycol compounds, could not be transposed to the closest prior art, even more importantly, because the additives used in D8 were not described to function as chelating agent, i.e. to have the same function as the additives employed in D2.

- (f) The new objection of the Respondents for a lack of inventive step over D8 (see *infra*), based on the argument, that no improvement had been demonstrated to result from the use of an aminopolycarboxylic acid when compared to the groups of compounds having two hydroxyl groups

used in D8, had been submitted for the first time during the oral proceedings. It should, therefore, be rejected as it was impossible for the Appellants to demonstrate at this stage of the proceedings the contrary, e.g. by furnishing adequate experimental evidence.

(g) Consequently, the subject-matter according to the Main Request was inventive.

(h) Concerning the Auxiliary Requests, it was relied, as far as inventive step was concerned, on the same arguments as for the Main Request.

XIII. The arguments of the Respondents which are relevant for the present decision can be summarized as follows:

(a) The insertion in claim 1 of the Main and Auxiliary Requests 1 and 2 of the wording "which process comprises a calcination step before compositing with the organic compound" lead to ambiguity, as it was unclear whether this step concerned the alumina carrier before or after impregnation with the organic compound. Furthermore, it was also unclear whether the feature "a used hydrotreating catalyst which has been regenerated" in claim 1 of Auxiliary Requests 3 to 8 referred to a catalyst comprising the hydrogenation metals in their oxide form. It was also objected that the presence of a hydrogenation metal as "stable oxide" on the carrier, which also was ambiguous, was originally disclosed only in combination with impregnation and calcination steps. In the absence of these process steps, claim 1 was in breach of

Article 123(2) EPC. Further, the combination of the features "providing a catalyst carrier", "which process comprises a calcination step before compositing with the organic compound" and "present as stable oxide" had no basis in the application as filed. Furthermore, claim 1 of any of Auxiliary Requests 3 to 8 also lacked clarity on the grounds that the use of regenerated catalysts was not covered by the wording "compositing". Accordingly, the sets of claims submitted by the Appellants one month before the oral proceedings were prima facie not allowable and should not be admitted. Concerning claim 1 of any of the Main and Auxiliary Requests 1, 3, 4, 5 and 7, the Respondents, however, stated that they did not object to a definition of the organic additive that was broader than in the requests submitted with the statement setting out the grounds of appeal.

- (b) As regards inventive step, two lines of argumentation starting from either D2 or D8 were presented, the line of argumentation starting from D8 being submitted for the first time at the oral proceedings before the Board.

- (c) As regards D2, Example 4 of that document described a process in which a γ -Alumina support comprising molybdenum trioxide and nickel oxide deposited thereon was subjected to pore volume saturation using various aminopolycarboxylic acids as chelating agent, such as nitrilotriacetic acid and ethylenediaminetetraacetic acid (EDTA), the document describing that an interaction took place

between at least the amorphous γ -Alumina, the chelating agent and the catalytically active components. D2 taught that EDTA and the like could improve the dispersion of Ni/Co-Mo metals on the alumina or re-disperse the metals if the chelating agent was added to a calcined or regenerated catalyst.

- (d) The difference between D2 and the claimed invention was the omission of the calcination step between the addition of the aminopolycarboxylic acid and the sulphidation step. The comparison made in the experimental part of the patent in suit was not suitable to prove any increase of activity resulting from the absence of a calcination step, because the catalysts tested after treatment with EDTA and with or without a subsequent calcination step, had not been indicated to comprise the hydrogenation metals in their oxide form. Should the hydrogenation metals in the catalyst carrier tested nevertheless be considered to be in their oxide form, an increase of activity brought about by the calcination step would be acknowledged. The same effect would also be credible in the presence of various aminopolycarboxylic acids.
- (e) D8 however suggested in relation to additives comprising at least two hydroxyl groups and 2-10 carbon atoms, to which EDTA belonged, the omission of a calcination step in order to improve catalytic activity. Reference was in particular made to Example 1 of D8 using as in D4 ethyleneglycol as chelating agent. Thus, even if

the omission of a calcination step were considered to solve the problem of providing an increased activity, this step would have been obvious in view of the teaching of D8. An inventive step should, therefore, be denied. The same held true for Auxiliary Requests 1 to 8.

(f) Starting from the process of D8 as closest prior art, the selection of EDTA, which was a chelating agent encompassed by the group of additives employed in D8, was, in the absence of any evidence for a beneficial effect resulting from this choice, an obvious alternative in view of D2.

XIV. The Appellants (Patent Proprietors) requested that the decision under appeal be set aside and that the patent be maintained on the basis of the Main Request or any of Auxiliary Requests 1 to 8 submitted with letter received on 29 May 2012.

XV. The Respondents (Opponents) requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

Admissibility of the Requests

2. The Respondents requested that the new requests be not admitted into the procedure, because the introduction of the features intended to define that the metals of groups VIB and VIII are in the form of their oxide was

prima facie not allowable in view of the requirements of Article 123(2) EPC and Article 84 EPC. They did not, however, object to a definition of the organic compound, which was broader than in the claims submitted with the statement setting out the grounds of appeal. Using the discretionary power conferred by Article 13(1) RPBA, the Board decided to allow the new claims Requests to be introduced in the proceedings, as the amendments submitted did not shift the technical focus of the case to new issues with respect to inventive step and the objections raised by the Respondents in response to the amendments submitted could reasonably be expected to be dealt with at the oral proceedings.

Main Request - Inventive step

Closest prior art

3. The patent in suit relates to a process for preparing hydrotreating catalysts and their use for hydrodesulphurisation and hydrodenitrogenation of hydrocarbon feeds (see paragraphs [0001] and [0016], claims 1 and 11). It aims in particular at improving the activity of those catalysts.
- 3.1 Document D2 cited by the Respondents as suitable starting point for assessing inventive step also relates to a process for preparing a hydrotreating catalyst having improved activity. The hydrotreating catalysts obtained in Example 4 are prepared in a first step by wet impregnation with a solution comprising oxides of molybdenum and nickel, as well as phosphoric acid of a catalyst carrier comprising γ -alumina, said impregnated support being dried and calcined at 427°C

for one hour. The calcined support is then subjected to pore volume saturation with aqueous solutions of seven different chelating agents, namely ethylenediamine-N,N-diacetic acid, nitrilotriacetic acid, N-tris(2-aminoethyl)amine, ethylenediamine, ethyleneglycol, triethylenetetramine and diammonium-ethylenediaminetetraacetic acid. After aging at 75°C for 3 days and drying, the various samples are subjected to calcination at 454°C. The obtained catalysts are then sulphided *in situ* in a hydrodenitrogenation process.

3.2 It was not disputed that the subject-matter of present claim 1 acquires novelty over the processes described in Example 4 of D2, which use three different aminopolycarboxylic acids as chelating agent, only by the fact that in present claim 1 the aminopolycarboxylic acid chelating agent is not exposed to calcination prior to the sulphidation step. It was moreover not contested, that the definition of the first step of the claimed process leading to the catalyst carrier to be treated with the organic additive, although being objected to lack clarity, at least encompasses the preparation of the catalyst carrier disclosed in Example 4 of D2.

3.3 Accordingly, the processes disclosed in Example 4 of D2, as far as they concern the use of an aminopolycarboxylic acid, which have the same purpose as the present invention and require a single modification compared to the claimed process, represent in agreement with the parties a suitable starting point for assessing inventive step.

3.4 The Respondents submitted at the oral proceedings before the Board and for the first time in the present opposition and appeal proceedings that D8 could equally be considered as the closest prior art. D8 also aims at the same purpose of the present invention and differs from the presently claimed subject-matter only in that the additive employed for treating the carrier comprising the metal oxides are not disclosed to be aminopolycarboxylic acids (*see infra*). However, in view of the negative conclusion in respect of inventive step when starting from D2, it was not necessary to decide whether the additional new line of argumentation starting from D8, which does not come closer to the present invention than D2, should be allowed into the proceedings.

Problem solved and solution

4. Having regard to the processes for preparing a sulphided hydrotreating catalyst disclosed in Example 4 of D2, the Appellants submitted that the technical problem solved by the subject-matter of claim 1 as amended, was as indicated in paragraphs [0008] and [0037] of the patent in suit the provision of a process for preparing a sulphided hydrotreating catalyst which has a higher activity in hydrotreating than the catalysts of D2, and which process is at the same time simpler and cheaper. As a solution to this problem the patent in suit proposes the omission of a calcination step after treatment of the catalyst carrier with the chelating agent.

4.1 The Appellants in order to prove their contention relied on the experimental comparison presented in

paragraphs [0035] to [0041] of the patent in suit. In these paragraphs, a "Catalyst 1" according to the invention is described to be prepared by pore volume impregnation with a diammonium EDTA solution of a conventional hydrotreating catalyst containing specific amounts of molybdenum, nickel and phosphorous on a γ -alumina carrier. After aging and drying, the catalyst obtained is sulphided and tested for hydrodenitrogenation activity. Another catalyst is prepared in Comparative Example 1 in the same way as "Catalyst 1" except that after aging, the catalyst is calcined. The catalyst of Comparative Example 1, which is indicated to be prepared in accordance with the teaching of D2, is also tested for hydrodenitrogenation activity under the same conditions. It exhibits an activity which is about 3% below that of the catalyst according to the invention.

4.2 The Respondents disputed the suitability of the comparison offered in the patent in suit for demonstrating the alleged improved activity over D2, as in their opinion it was unknown, despite the declaration made by the Appellants at the oral proceedings, whether the comparison offered in the specification related to the treatment of hydrogenation metal components in the form of their oxides and therefore to the claimed subject-matter in its amended form.

4.3 The catalyst treated with diammonium EDTA is described in the experimental part of the patent in suit to be a conventional hydrotreating catalyst. This already indicates, in line with the process described in paragraph [0020] of the specification relating to

alternative (a), that the hydrogenation metal components are present in their oxide form. Moreover, in view of the order of the compounding steps employed in Comparative Example 1 and the reference to D2 in that comparative example, it can only be concluded that the method used in Comparative Example 1 is the method of Example 4 of D2, which was acknowledged by the Respondents. The fact that the hydrogenation metal components on the γ -Alumina carrier are present in Example 4 of D2 in their oxide form before treatment with the chelating agents, confirms therefore that is also the case for example 1 and comparative example 1 of the patent in suit. Hence, the Respondents' doubts on whether the hydrogenation metal components, in the examples and the comparative example of the patent in suit, are present on the carrier in the form of their oxide, are unfounded.

- 4.4 Under these circumstances, the Board is satisfied that the comparison offered in the patent in suit convincingly demonstrates that the omission in D2 of a calcination, between the treatment with diammonium EDTA and the sulphidation step, brings about higher activity for hydrodenitrogenation. Furthermore, the Respondents did not dispute, provided that example 1 of the patent in suit concerned the treatment of a carrier comprising the hydrogenation metals in their oxide form, that the same technical effect would also be obtained for other aminopolycarboxylic acids. The Board has no reason to take a different view. It is furthermore manifest, which was not disputed by the Respondents, that the process according to D2 is in the absence of a calcination step simplified and rendered less energy consuming, i.e. economically more favourable.

- 4.5 Summing up, the Board accepts that the solution proposed by the patent in suit successfully solves the technical problem defined by the Appellants.

Obviousness

5. Finally, it remains to be decided whether or not the proposed solution to the above technical problem is obvious for the skilled person in view of the state of the art.
6. As already indicated above, the closest state of the art does not only teach the treatment of the catalyst carrier with three specific aminopolycarboxylic acids, but also with three different polyamines and ethyleneglycol. All these additives are presented in Example 4 as chelating agents, in line with the explanation on page 3, lines 19-21 that they are believed to interact both with the alumina support and the catalytically active metal components. The use of any of these seven chelating agents is shown in Table 4, on page 13 to provide an increased activity of the catalyst for hydrodenitrogenation.
7. Hence, starting from the processes described in Example 4 of D2 which concern the treatment of a hydrodenitrogenation catalyst with various classes of chelating agents, in particular three different aminopolycarboxylic acids and also ethyleneglycol, in order to increase the activity of said catalyst, the person skilled in the art would naturally be aware of document D8, which also aims at increasing the activity of the same type of catalyst and which also recommends

for this purpose, as described in its Example 8, a treatment with ethyleneglycol.

8. More particularly, the catalyst carrier obtained in Example 8 of D8, namely a γ -alumina carrier supporting oxides of molybdenum, nickel and phosphorous, obtained in particular by impregnation and calcination at 500°C, is impregnated to pore volume saturation with various aqueous solutions of ethyleneglycol and dried at 100°C (page 27, Table 9, Examples BA1 to BA3). The samples obtained are following a presulfidation tested for hydrodenitrogenation activity. Compared to untreated samples or samples only treated with water, the catalyst carriers subjected to a treatment with ethyleneglycol are shown to exhibit an increased activity. More importantly, a comparison with an identical process, in which a calcination step was carried out after the treatment with ethyleneglycol and before sulphidation (catalyst BA1(C)), shows that the catalyst which has been calcined before sulphidation has an activity which is above that of the catalyst not treated (as in Example 4 of D2), but which is inferior to that of the uncalcined catalyst treated with ethyleneglycol. The reason for this improvement could be seen, according to page 13, lines 21-27, in the fact that the additive, which remains in the catalyst before sulphidation is carried out, prevents aggregation of the metal compound during the sulphide formation. The improvement for hydrodenitrogenation activity resulting from the absence of calcination is also confirmed in Examples 9 to 14 in relation to further additives belonging to the class of compounds comprising at least two hydroxyl groups and 2-10 carbon atoms, as well as their polyethers.

9. Hence, in view, first of the equivalence of functionality suggested in D2 between aminopolycarboxylic acids and ethyleneglycol, second of the identity of the catalysts treated in D2 and D8 and third of the explanation in D8 for the improvement of activity brought about by the absence of a calcination step after a treatment with ethyleneglycol or further additives taught by this document, the skilled person starting from the catalyst activation method of D2 and wishing to further improve the hydrodenitrogenation activity of the catalysts would have been led with a reasonable expectation of success to omit in Example 4 of D2 the step of calcining the chelating agent. Furthermore, the need for a simpler and economically attractive method, would have provided the skilled person with a further incentive to omit a calcination step. Consequently, the subject-matter of claim 1 of the Main Request, which encompasses embodiments which are obvious modifications of the closest prior art lacks at least to that extent an inventive step.
10. Consequently, the Main Request must be rejected.

Auxiliary Requests

11. The additional modifications contained in the Auxiliary Requests do not introduce additional distinguishing features over the methods disclosed in Example 4 of D2 which relate to the use of aminopolycarboxylic acids. As a result, claim 1 of any of the Auxiliary Requests still encompasses the embodiments of claim 1 of the Main Request which have been shown to be derivable in an obvious manner from the state of the art.

Accordingly, these Auxiliary Requests must also fail on the same ground.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar

The Chairman

S. Fabiani

J. Riolo