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Datasheet for the decision of 5 February 2016

Case Number: T 0022/09 - 3.3.07

Application Number: 99906328.2

Publication Number: 1058580

IPC: B01J33/00, B01J23/75, C07C1/04,

B01J37/02

Language of the proceedings: ΕN

Title of invention:

PROCESS FOR PRODUCING FISCHER-TROPSCH CATALYSTS

Patent Proprietor:

Sasol Technology (Proprietary) Limited

Opponent:

Formalities Bureau Limited

Headword:

Fischer-Tropsch Catalysts/ Sasol

Relevant legal provisions:

RPBA Art. 13(3)

EPC Art. 54, 56, 83, 84, 111(1), 123(2)

Keyword:

Late-filed request - admitted (yes) - new sole claim request filed at the oral proceedings

Amendments - added subject-matter (no)

Sufficiency of disclosure - (yes)

Late-filed document - admitted (yes)

Novelty - (yes) - non-medical use

Inventive step - (yes)

Decisions cited:

G 0002/88, G 0007/93, G 0001/03, G 0003/14, T 0420/86, T 0276/96

Catchword:



Beschwerdekammern Boards of Appeal

Chambres de recours

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Case Number: T 0022/09 - 3.3.07

D E C I S I O N
of Technical Board of Appeal 3.3.07
of 5 February 2016

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Decision under appeal: Interlocutory decision of the Opposition

Division of the European Patent Office posted on 29 October 2008 concerning maintenance of the European Patent No. 1058580 in amended form.

Composition of the Board:

Chairman J. Riolo
Members: G. Santavicca

D. T. Keeling

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Summary of Facts and Submissions

- The appeal lies from the decision of the Opposition Division concerning maintenance of European Patent 1 058 580 in amended form, according to (Claims 1 to 9 of) the Main Request filed with letter of 27 August 2008.
- II. Claim 1 according to the Main Request was identical to Claim 1 as granted, and read as follows:
 - "1. A method of pre-treating a catalyst support to be used in a catalyst-forming process involving impregnation of the support in neutral or acidic aqueous conditions, characterized in that it comprises introducing onto and/or into an untreated particulate catalyst support selected from the group consisting of Al_2O_3 , titania (TiO₂) and magnesia (MgO), a modifying component, which is capable, when present in and/or on the catalyst support, of suppressing the solubility of the catalyst support in aqueous acid solutions and/or neutral aqueous solutions, said modifying component comprising Si, Zr, Cu, Zn, Mn, Ba, Co, Ni and/or La and calcining the resultant modifying component containing modified catalyst support to decompose organic groups or form spinel structures with the support, at a temperature from 100°C to 800°C and for a period of from 1 minute to 12 hours, to obtain a protected modified catalyst support, which is less soluble or more inert in the aqueous acid solution and/or neutral aqueous solution than the untreated catalyst support, with the modifying component being present, in the modified catalyst support particles, on the particle surfaces; provided that when the modifying component comprises Si, Co or Zr, the calcination period is from 1 hour to 4 hours, and that when the modifying component comprises Co, the calcination temperature is from 600°C to 800°C."

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III. The patent was opposed in its entirety on the grounds of added subject-matter (Article 100(c) EPC), insufficient disclosure (Article 100(b) EPC) as well as lack of novelty and inventive step (Article 100(a) EPC). The following items of evidence were inter alia relied upon: D3: EP 0 736 326 A1; and D4: B. Beguin et al, "Stabilization of Alumina toward Thermal Sintering by Silicon Addition", Journal of Catalysis, 127, 595-604 (1991);

With letter of 29 August 2008 (one month before the oral proceedings), the opponent filed document US 4,459,372 A (D11), and requested that it be admitted into the proceedings in view of its *prima facie* high relevance.

- IV. In the decision under appeal, the Opposition Division inter alia came to the following conclusions:
 - a) After the deletion of Claim 5 as granted, the ground of opposition under Article 100(c) EPC no longer prejudiced the maintenance of the patent amended according to the Main Request.
 - b) Although the burden of proof lay on the Opponent, its ground of opposition under Article 100(b) EPC had not been substantiated by evidence. Hence, the disclosure of the patent was sufficient.
 - c) Late filed document D11 was not relevant, hence not admitted into the proceedings.
 - d) The claimed subject-matter was novel, in particular over D3 or D11.
 - e) The closest prior art was disclosed by D3, which addressed the problem of the Cobalt-loss of the catalyst.
 - f) However, D3 solved this problem by washing the catalyst before use, to remove the loose cobalt from the catalyst. Furthermore, D3 started from

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boehmite and also used ammonia, which reacted with the free -OH groups present, thus preventing their reaction with the modifying component. Since, however, ammonia was decomposed upon calcination, free -OH were again formed, which increased the dissolution in aqueous acidic/neutral conditions.

- The claimed process was thus not obvious.
- h) The Main Request thus complied with the EPC.
- V. With its statement setting out the grounds of appeal, the Appellant submitted two new items of evidence, inter alia D20, namely EP 0 094 206 A1.
- VI. With its letter of 8 July 2009, the patent proprietor (Respondent) replied to the statement setting out the grounds of appeal and filed two items of evidence.
- VII. In its letter of 3 July 2012, the Appellant enclosed two further items of evidence as follows: D23: GB 1 492 274 A; and,

D24: EP 0 190 883 A2.

- VIII. In response to a communication from the Board in preparation for oral proceedings, dated 27 July 2012: - the Appellant submitted the complete document D19 (with letter dated 26 [sic] July 2012) and Experimental Report D27 (with letter of 9 August 2012); - the Respondent, with letter dated 6 August 2012, inter alia filed Test Report D26.
- IX. In its letter of 27 August 2012, the Respondent inter alia objected to the admissibility of the appeal. With letter of 4 September 2012, evidence from the UK Company Register was filed, in order to show that the company in which name the opposition had been filed had ceased to exist since 2005.

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The Appellant disagreed (letter of 4 September 2012).

- X. Oral proceeding were held on 21 June 2013, the only point at issue being the admissibility of the appeal. The Board decided to refer questions to the Enlarged Board of Appeal (T 22/09, OJ EPO 2013, 582).
- XI. In Decision G 1/13 (OJ EPO 2015, A42), on the questions referred to it by the Board, the Enlarged Board of Appeal indicated that the appeal must be treated as admissible (Point 2 of the order).
- XII. with a communication dated 23 March 2015, the Board invited the parties to restate their requests in order to resume the appeal proceedings. In reaction thereto:
 - a) The Respondent (letter of 21 April 2015) (re)filed amended claims labelled Main Request and Auxiliary Requests 1-4, 4A, 5, 5A, 6, 6A, 7-10, already filed with letter dated 6 August 2012, as well as amended claims labelled Auxiliary Requests 4', 4A', 5', 5A', 6', 6A', 8', 9' and 10', already filed with letter dated 27 August 2012.
 - b) The Appellant (letter of 29 May 2015) maintained that D23 and D24 were prima facie highly relevant and should be admitted into the proceedings and that the patent should be revoked. It also raised objections under Articles 54, 84 and 123, paragraphs 2 and 3, EPC, as well as Rule 80 EPC.
- XIII. In its letter dated 30 July 2015, the Respondent (counter) argued inter alia that:
 - a) D23 and D24 were not prima facie highly relevant, hence not to be admitted into the proceedings;
 - b) still, only D3 qualified as the closest prior art;

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- c) the amended claims of the auxiliary requests complied with Articles 84 and 123, paragraphs 2 and 3, EPC, as well as with Rule 80 EPC.
- The parties were summoned to oral proceedings, which XIV. took place on 5 February 2016. The Board confirmed the decision of the Opposition Division not to admit D11 into the proceedings and decided not to admit D20 but to admit the other late filed documents, e.g. D23 and D24, into the proceedings. The Respondent filed an amended version of Auxiliary Request 1 filed with letter of 21 April 2015, which the Appellant challenged on lack of novelty, over D3, D4, D23 and D24, and inventive step, over D23 and D3. The Board, after having heard the parties, and after deliberation, found that the claimed use was novel and involved an inventive step. The Respondent made the amended version of Auxiliary Request 1 filed at the oral proceedings its Main Request and withdrew all other requests previously submitted.
- XV. The Main Request filed at the oral proceedings contains 6 claims, Claim 1 reading as follows:
 - "1. Use of a modifying component for suppressing the solubility of a catalyst support in aqueous acid solutions and/or neutral aqueous solutions, when present in and/or on the catalyst support, wherein the catalyst support is to be used in a catalyst-forming process involving impregnation of the support in neutral or acidic aqueous conditions and wherein the modifying component comprises Si, Zr, Cu, Zn, Mn, Ba, Co, Ni and/or La and characterized in that the modifying component is introduced onto and/or into an untreated particulate catalyst support selected from the group consisting of Al_2O_3 , titania (TiO_2) and magnesia (MgO), and afterwards

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the resultant modifying component-containing modified catalyst support is calcinated to decompose organic groups or form spinel structures with the support, at a temperature from 100°C to 800°C and for a period of 1 minute to 12 hours, to obtain a protected modified catalyst support, which is less soluble or more inert in the aqueous acid solution and/or neutral aqueous solutions than the untreated catalyst support, with the modifying being present, in the modified catalyst support particles, on the particle surfaces; provided that when the modifying component comprises Si, Co or Zr, the calcinations [sic] period is from 1 hour to 4 hours, and that when the modifying component comprises Co, the calcinations [sic] temperature from 600°C to 800°C ".

Claims 2 to 6 concern embodiments of the use of Claim 1.

- XVI. The Appellant requested that the decision under appeal be set aside and that the patent be revoked.
- XVII. The Respondent requested that the decision under appeal be set aside and that the patent be maintained according to the Main Request filed as the sole claim request at the oral proceedings held on 5 February 2016.
- XVIII. The arguments of the Appellant of relevance for the present decision can be summarised as follows:
 - a) The amended claims of the sole request contravened the requirements of Articles 84 and 123(2) EPC.

 As Claim 1 had been modified (change of category), its clarity might be objected to. The expressions "for suppressing the solubility" and "less soluble or more inert" were contradictory and not clear.

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As to Article 123(2) EPC, the deletion of features as granted, "pre-treating" and "which is capable, when present in and/or on the catalyst support, of suppressing the solubility of the catalyst support in aqueous acid solutions and/or neutral aqueous solutions", extended the initial content.

- b) It was not possible to form spinels with trivalent modifying component precursors and Al₂O₃, nor was it possible to form spinels with TiO₂ and tetravalent modifying component precursors or with MgO and divalent modifying component precursors. The time and temperature conditions defined in Claim 1 at issue were such that calcination might not always give rise to decomposition of organic residues, as shown by D27. As Claim 1 encompassed non-working embodiments, its invention was not sufficiently disclosed.
- c) Documents D11, D20, D23 and D24 should be admitted into the proceedings in view of their *prima facie* high relevance against novelty and inventive step.
- d) The claimed use was not new over D3, D4, D23, D24. D3 illustrated in Examples 18 and 20 the use of Zr salts to pretreat a precipitated alumina labelled "Pural SB alumina". Contrary to the decision under appeal, this led to reduction of the solubility. D4 addressed the problem of thermal stability of γ-alumina (Al₂O₃), disclosed that Si was a good candidate for this stabilization and showed that the stability toward thermal sintering in the presence of water increased with the Si loading. Thus, D4 anticipated the claimed use of silicon to reduce the solubility of Al₂O₃ in aqueous solution.

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D23 (e.g. Example 20) taught the stabilization of γ -alumina used in car mufflers or as supports for catalysts in fluidised beds. The stabilization method included impregnation and calcination. Al₂O₃ treated with Si showed hydrothermal stability and more inertness in aqueous conditions. Hydrothermal stability in aqueous conditions implied solubility stability in aqueous solutions. Thus, the claimed use was not new.

D24 disclosed an alumina catalyst support for withstanding high temperatures, made of high surface area alumina, impregnated with silicone, then calcined to pyrolyze the organic groups and stabilize the alumina. The stabilized support was then treated with an aqueous slurry (washcoat) of a metal catalyst. D24 anticipated the claimed use.

As to inventive step, the closest prior art was e) disclosed by D23, e.g. its Example 20, which concerned heterogeneous catalysis and contained clear indications of treatment of y-alumina with organic silicon compounds in order to improve the hydrothermal stability, which implied stability in aqueous environments. The invoked problem was not solved across the whole breadth of Claim 1, as: the patent in suit did not show that the claimed suppression of solubility was always achieved, as apparent from Figure 11 of the patent in suit, which, for α -alumina, showed that no effect was to be achieved; and, Experimental Report D27 showed that not all the calcination conditions defined in Claim 1 led to complete decomposition of organic groups or formation of spinels. Since the catalysts of D23 were suitable for fluidized beds, they could be used in bubble column reactors as taught in D3. The claimed use was thus obvious.

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- XIX. The (counter) arguments of the Respondent of relevance for the present decision can be summarised as follows:
 - a) The sole claim request maintained at the oral proceedings was filed in reaction to the debate, to overcome the objections raised. Its filing did not surprise the other party nor the Board. Hence, it was admissible.
 - Apart from the change of category (from method to b) use), the features of Claim 1 as granted had been maintained in Claim 1 at issue, which was narrower in scope than Claim 1 as granted, and which did not extend beyond the application as filed. As no lack of clarity arose from the amendments made, no objection under Article 84 EPC might be raised against Claim 1, in line with G 3/14 (OJ EPO 2015, A102). In particular, the feature "suppressing the solubility" was clear. If read in connection with how the use was expressed, it meant that the modifying component was used to exploit its capability of suppressing solubility. This wording was already in the application as filed. The effect "less soluble or more inert" of Claim 1 as granted was still present in Claim 1 at issue. These effects were described (e.g. in Example 11) and depicted in Figure 1, showing that the use of a modifying component on supports, in different loadings, suppressed the solubility and made them more inert. Therefore, the statements in the claims as granted and in the claims at issue were the same, or were equivalent. The claimed use would have been unambiguously gathered from the application as filed by the skilled person.

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- The Appellant had not discharged its onus of proving that the conditions defined in Claim 1 at issue were not always suitable for decomposing organic groups or form spinels, hence for obtaining a modified support as claimed. In fact, these objections were the result of a claim misinterpretation. The skilled person would not consider in isolation the given conditions and the purpose of these conditions. He would disregard non-disclosed conditions based on both low temperature and short time, because Claim 1 required thereby decomposition of organic groups or formation of spinels. In any case, embodiments calcined at low temperature in short time, i.e. without complete decomposition of organic groups or formation of spinel, did not fall under Claim 1. Experimental Report D27, which was about weight loss calculation on calcination's conditions, and which was silent on the reduction in solubility in aqueous environment of the so calcined support, did not corroborate the insufficiency objections.
- d) The Board should confirm the non-admittance of D11 into the proceedings, because the Opposition Division correctly exercised its discretion. D20 was not relevant and should not be admitted. The method of pretreating a catalyst support as illustrated in the examples of D23 and D24 "inevitably" led to suppression/reduction of solubility in aqueous conditions. This was the reason to abandon the Main Request, filed with letter of 21 April 2015, at the oral proceedings. However, D23 and D24 were not relevant against the use of Claim 1 at issue.

e) As established in G 2/88 (OJ EPO 1990, 93), the purpose defined in Claim 1 at issue was a technical functional feature representing a technical effect, namely the suppression of the solubility in aqueous neutral or acidic conditions.

D3 (e.g. Examples 18 and 20) disclosed some of the features defined in Claim 1 at issue. The examples invoked concerned an <u>extruded</u> support (first distinction), calcined at high temperature <u>for 16h</u> (second distinction). D3 did not however disclose the claimed effect (third distinction).

D4 taught the treatment of γ -alumina with Si in order to improve hydrothermal stability. This was apparent from Table 2, in which the pore volume and surface area modifications were tested. D4 did not disclose that the modification with Si to attain hydrothermal stabilization had any influence on the solubility in aqueous acidic or neutral conditions, let alone on its suppression.

D23 and D24 addressed high thermal stability and mechanical resistance, particularly the reduction of phase transition. As apparent from Figure 3, only surface area variations were measured, not the solubility. In Example 20 of D23, the amount of water used was 10 cc, so that it was not possible to equate it to an aqueous solution. Also, the pore volume modification was not an indication of lower solubility in aqueous environment. Hydrothermal stability could not be equated with solubility in acidic/neutral aqueous conditions. Even though the method illustrated in Example 4 of D23 inevitably got a reduced solubility, this effect was not taught in D23 and D24.

Since at least the effect of the claimed use had not been disclosed, the claimed use was new.

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As to inventive step, none of the invoked documents D23 or D24 and D3 dealt with the suppression of solubility in acidic/neutral aqueous conditions. The skilled person was confronted with a situation in which no prior art aimed at the effect defined in Claim 1 at issue. Hence, neither D23 nor D24 might be considered as the closest prior art. If the more specific problem of the Fischer-Tropsch process were considered, i.e. the lowering of the cobalt amount in the wax, then D3 might be the closest prior art. It was true that Al_2O_3 had low solubility in aqueous environment, it was however still soluble to a low extent above zero, which was detrimental for the claimed use, as it eventually led to more cobalt in the product. Thus, the technical problem remained as stated in the patent in suit. According to the examples of the patent in suit, the effect defined in Claim 1 at issue had been effectively achieved, i.e. the problem had been effectively solved across the breadth of Claim 1. The contrary had not been shown by the Appellant. Test Report D26 showed that the use of ammonia disclosed in D3 led, if any, to increased solubility. D23 and D24 did not hint at using a modifying component such as Si in a use as defined in Claim 1 as issue. Therefore, the claimed use was not obvious over the prior art.

Reasons for the Decision

Admissibility of the new Main Request

 The filing of the sole claim request maintained during the oral proceedings was in reaction to the debate between the parties. - 13 - T 0022/09

In particular, it came as a reaction to the opinion of the Board that Claim 1 at issue did not comply with the requirements of Article 123(3) EPC, due to the deletion of the feature that the catalyst support was to be used in a catalyst-forming process involving impregnation in neutral or acidic aqueous conditions. The new claim request is late but addresses and overcomes the finding of the Board, without raising questions which could not have been dealt with during the oral proceedings. The Appellant did not raise any objections against its filing. The Board exercised its discretion (Articles 12(4) and 13(1)(3) RPBA) and decided to admit it into the proceedings, despite its late filing.

Amendments

- 2. Apart from the change of category (from a method of pretreating a catalyst support to the use of a modifying component for achieving a particular effect on that support), the claims of the sole request are based on Claims 1-4 and 6-7 as granted, i.e. contain their features but rearranged to define the use.
- 2.1 Since Claim 1 as granted concerned a method of pretreating a catalyst support, which by virtue of Article 64(2) EPC extended the protection conferred to the pretreated catalyst support directly obtained by the claimed method, and since instead Claim 1 according to the new, sole claim request concerns the use of a modifying component for suppressing the solubility of a catalyst support, i.e. defines the use of a chemical compound to obtain a particular effect on the catalyst support, the protection conferred by the patent as granted has not been extended (Article 123(3) EPC; Case Law, II.E.2.4, e.g. T 420/86 of 12 January 1989 and T 276/96 of 17 June 1997).

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- 2.2 The objections under Article 123(2) EPC raised by the Appellant concern the alleged suppression of the features of Claim 1 as granted "pre-treating" and "which is capable, when present in and/or on the catalyst support, of suppressing the solubility of the catalyst support in aqueous acid solutions and/or neutral aqueous solutions", which alleged suppression extended the content of the application as filed.
- 2.2.1 It is however apparent from the wording of Claim 1 at issue (Point XVII, *supra*) that:
 - (a) the claimed use of the modifying component for suppressing the solubility is to be carried out on a catalyst support, which is then to be used in a catalyst-forming process. Hence, the claimed use implies a pre-treatment of the catalyst support.
 - (b) the purpose defined in Claim 1 at issue "a modifying component for suppressing the solubility of a catalyst support in aqueous acid solutions and/or neutral aqueous solutions, when present in and/or on the catalyst support," is nothing more than the expression of the capability defined in Claim 1 as granted "a modifying component, which is capable, when present in and/or on the catalyst support, of suppressing the solubility of the catalyst support in aqueous acid solutions and/or neutral aqueous solutions", i.e. its exploitation. This is apparent from the features "to obtain a protected modified catalyst support, which is less soluble or more inert in the aqueous acid solution and/or neutral aqueous solutions than the untreated catalyst support, with the modifying component being present, in the modified catalyst support particles, on the particle surfaces". The features

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- of granted Claim 1 alleged to have been deleted, are still defined/implied in Claim 1 at issue.
- (c) Therefore, the objections of the Appellant against the alleged deletions are not convincing.
- 2.2.2 Indeed, the Board is satisfied that the claims at issue are fairly based on the application as filed, namely on Claims 1-5 and 7-8 as initially filed, as well as on the general disclosure in the description as filed, pages 5 (second paragraph) and 6 (second and third paragraphs).
- 2.3 The claims at issue are not objectionable under Article 123 EPC.
- 2.4 The Appellant also raised objections under Article 84
 EPC (lack of clarity) against the features "suppressing
 the solubility" and "less soluble or more inert", which
 were contradictory and not clear ("more inert").
- 2.4.1 According to G 3/14 (order), the claims of a patent may be examined for compliance with the requirements of the Article 84 EPC only when, and then only to the extent that, the amendment introduces a non compliance with Article 84 EPC.
- 2.4.2 Such is evidently not the case here, as the features objected to were already present in Claim 1 as granted, i.e. do not arise out of the amendment carried out (the change of category), and could not have been objected to as such under Article 84 EPC in Claim 1 as granted.
- 2.4.3 Consequently, the claims at issue are not objectionable under Article 84 EPC.
- 2.5 Therefore, the claims at issue are formally allowable.

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Alleged insufficient disclosure

- 3. As to the insufficiency raised during the opposition proceedings, and dealt with in the decision under appeal, the Board has no reason to take another stance.
- 3.1 In respect of the objections that Claim 1 at issue still includes modifications of the support where organic groups are not decomposed or spinels are not formed, the Board notes that the Appellant particularly relied to Experimental Report D27, in order to show that not all the defined operating conditions (time/temperature) for the calcination led to the (complete) decomposition of organic groups or to the formation of spinels.
- 3.2 The objection that Claim 1 defines operating conditions (e.g. 1 minute and 100°C) which do not lead to complete calcination, as shown by D27, is not convincing, already because Claim 1 requires that the calcination is for decomposing organic groups or for forming spinels. Also, Paragraph [0043] of the patent in suit gives guidance on how to assess the right calcination time for decomposing the organic groups. Example 2 of the patent in suit illustrates the formation of a spinel. The skilled person has no reason to envisage a calcination not leading to the stated effects, e.g. 1 minute at 100°C, inter alia because the use thereof would not fall under Claim 1 at issue.
- 3.3 During the discussion of inventive step, the Appellant inter alia contended that the purpose of the claimed use was not achieved across the whole breadth of Claim 1, as Figure 11 of the patent in suit showed that no (suppression-of-dissolution) effect could be achieved for α -Al₂O₃ alumina.

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- 3.3.1 According to settled case law (G 1/03, OJ EPO 2004, 413, Reasons 2.5.2, third paragraph), for claims alleged to encompass non-working embodiments, any invoked lack of reproducibility may become relevant under the requirements of inventive step or sufficiency of disclosure, depending on <a href="https://where.nc.nimes.org/where.n
- 3.3.2 The argument based on Figure 11 of the patent in suit is not convincing already for the reasons given in paragraph [0082] of the patent in suit, namely:
 - that α -Al₂O₃ has a lower dissolution profile than γ and δ -Al₂O₃ (hence, it still has one, albeit low. This is also apparent from Figure 11 itself, as the profile for α -Al₂O₃ is not completely flat on the axis); and,
 - that the catalyst support mainly consists of γ and δ Al₂O₃ (the dissolution profile of which can be lowered).
- 3.4 The alleged insufficiency has not been proven.

Late filed items of evidence

4. Still according to settled case law (G 7/93, OJ EPO 1994, 775, Reasons, 2.6), the discretionary power conferred by Article 114(2) EPC to disregard late filed items of evidence implies that the Opposition Division must have a certain degree of freedom in exercising its power. A Board may overrule the discretion exercised by the Opposition Division only if it is apparent that it has been done according to wrong principles, or without taking into account the right principles, or in an unreasonable way. In the present case, in respect of D11, it is apparent from the decision under appeal

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(Ground on page 4), that the Opposition Division has considered the lack of justification for the late filing as well as the lack of disclosure of relevant features, i.e. it has applied the right approach to admission of late filed documents. As the Board has no reason to overrule the discretion exercised by the Opposition Division, D11 remains non-admitted into the proceedings.

- As to D20, filed with the statement setting out the grounds of appeal, the Board has decided that it should not be admitted into the proceedings, for the reasons already expressed in the Board's communication dated 27 July 2012 (see Point 12.6), namely: The examples of D20 do not mention which alumina (Al₂O₃?) is used; the calcination time in general is not disclosed, whilst the illustrated calcination times are of at least 16 hours; and the sought-for reduction of the solubility of the support is not mentioned. Thus, D20 cannot prejudice the novelty of the claimed use.
- 4.2 Concerning D23 and D24, the relevance of which became even more apparent during the oral proceedings (as the abandonment of Main Request was indisputably due to their relevance to novelty), the Board decided to admit them into the proceedings, in line with the provisional opinion expressed in its communication (Point 12.7).
- 4.3 Also Experimental Reports D26 and D27 were admitted, in so far they were relevant to back up the arguments of the parties on insufficiency and inventive step.

Novelty

5. Documents D3, D4, D23 and D24 were invoked against the novelty of the use defined in Claim 1 at issue.

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- D3 (Claim 1) concerns a process for preparing a FischerTropsch catalyst comprising
 subjecting a slurry composition comprising a
 particulate alumina support, water and an active
 component selected from the group consisting in cobalt
 (Co), iron (Fe) and mixtures thereof, to a subatmospheric pressure environment, thereby to impregnate
 the alumina support with the active component;
 drying the dried impregnated support, thereby to
 obtain the Fischer-Tropsch catalyst.
- 5.1.1 The process of D3 (Claim 12) can include a pretreatment of the particulate alumina support prior to forming the slurry thereof with water and the active component, to modify the average pore diameter and/or to modify the chemical phases, by chemically pretreating the support the support prior to the slurry formation. The chemical pretreatment may involve the treatment with ammonia (page 3, line 21).
- 5.1.2 The pretreated supports of D3 invoked by the Appellant, in particular those of Examples 18 and 20, have been chemically pretreated with ammonia, then extruded and finally calcined for 16 hours.
- 5.1.3 D3 does not disclose the use of a modifying component as defined in Claim 1 at issue, let alone for reducing the solubility of the support in acidic/neutral conditions.
- 5.2 D4 concerns the stabilization of alumina (in particular γ and δ forms) supports toward thermal sintering by silicon addition (title). It teaches (Abstract; page 597, right column, lines 22-24; page 598, left column, lines 20-23; conclusion) that the hydroxyl groups of the alumina react with a Si-containing precursor, to form a layer of silica firmly bonded to the alumina support,

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whereby the stabilization of alumina toward sintering in the presence of water (as shown by ageing tests at 1050°C for 24h under air and 20% vol. water) increases with increasing silicon loadings until 3 wt-% Si. D4 teaches the use of organic Si-precursor. Even if the treatment of D4 led to catalyst supports with reduced solubility in aqueous acidic or neutral conditions, this purpose is not mentioned in D4, so that D4 is not prejudicial to novelty already for this reason.

5.3 D23 concerns a process for surface modifying metal oxides, having catalytic activity in heterogeneous phase reactions, to produce a material having high thermal and mechanical stability (page 1, lines 8-13). The causes of the stability losses mentioned by D23 include higher abrasion, sintering and shrinkage (page 1, lines 50-73). These problems occur particularly in car mufflers and in fluidised beds (page 1, lines 39-43). The material so produced can be used as support material and can be in granules (page 2, lines 4 and 7). The process of D23 can be carried out in liquid phase (page 2, line 13), where the oxide material is impregnated with an organic silicon precursor (page 2, lines 16-33), then dried (paragraph bridging left and right columns of page 2), finally calcined at 300-600°C for 2 to 10 h to inter alia remove organic materials (page 2, lines 83-98). Example 4 illustrates the treatment with organo-silicon carried out on y-alumina to produce a material (Table 1 for the results in terms of stability), which subjected to hydrothermal treatment as illustrated by Example 20 (in which a 15 g sample was treated in an autoclave containing 10 cc water), undergoes no variation. During the oral proceedings it was not in dispute that the γ alumina treated according to Example 4, and showing the properties illustrated in Example 20, might also have a reduced solubility in aqueous conditions. However, D23

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does not disclose that the purpose of using silicon is for reducing the solubility of the support during impregnation in aqueous acidic/neutral conditions used in catalyst-forming processes. Hence, D23 cannot prejudice the novelty of the claimed use at issue.

- D24 acknowledges D23, and discloses (e.g. Example 1) a process for treating a catalyst support of γ -alumina powder with a silicone, then dried and calcined, hence which is similar to the process disclosed by D23. D24 does not disclose either that the purpose of using silicon to modify the treated support is to reduce the solubility in aqueous acidic/neutral conditions during impregnation used in catalyst-forming processes. D24 is not prejudicial to novelty either.
- 5.5 Therefore, the claimed use is novel over this prior art.

Inventive step

The invention

6. The invention at issue relates to the use of a modifying component for suppressing the solubility of a particulate catalyst support to be used in a catalyst-forming process involving impregnation of the support in neutral or acidic aqueous conditions (paragraph [0002]).

The closest prior art

7. At the oral proceedings before the Board, the Appellant invoked D23 as the closest prior art for assessing inventive step, whilst the Respondent took the position that there was no closest prior art document on file dealing with the same objectives and problems as the patent in suit. If one had to be considered, then it was

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D3, which at least addressed the problem of reducing the undesired high cobalt content of the wax product (in this respect, reference was made to paragraph [0038] of the patent in suit). Hence, it has to be established which of D23 and D3 discloses the closest prior art, i.e. represents the most appropriate starting point, for assessing inventive step.

- 7.1 D23 (supra) concerns a process for surface modifying metal oxide materials, having catalytic activity when used in heterogeneous phase reactions, to produce a material having high thermal and mechanical stability (page 1, lines 8-13). D23 does not belong to the technical field of the patent in suit, the production of catalysts for the Fischer-Tropsch process. Furthermore, D23 neither addresses the problem of reducing the solubility of supports in aqueous acidic or neutral conditions, nor the problem of reducing the undesired high cobalt content of the wax product, mentioned in paragraph [0038] of the patent in suit.
- 7.2 D3 (supra) belongs to the same technical field of the patent in suit (catalyst for Fischer-Tropsch process) and was acknowledged in the application as filed, on which the patent was granted (page 3, line 16; page 7, line 29) (see paragraph [0006] of the patent in suit). Moreover, D3 addresses the problem of reducing the undesired high cobalt content of the wax product mentioned in paragraph [0038] of the patent in suit.
- 7.3 In view of the indisputed similarity between the patent in suit and D3 in terms of the cobalt-in-wax problem addressed, D3 rather than D23 is the closest prior art.
- 7.4 The relevant disclosure of D3 is summarised in Point 5.1 to 5.1.3 supra.

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The technical problem according to the Respondent

8. At the oral proceedings before the Board, the Respondent maintained the technical problem formulated in the patent in suit (paragraphs [0036] and [0038]), i.e. the providing of a catalyst support, with which catalysts for Fischer-Tropsch synthesis could be made, which prevented contamination of the wax product with undesired high cobalt content.

The solution

- 9. The patent in suit as amended proposes to solve this problem by providing a use as defined in Claim 1 at issue, which is particularly characterized in that:

 a modifying component comprising Si, Zr, Cu, Zn, Mn, Ba, Co, Ni and/or La is used for suppressing the solubility of a catalyst support in aqueous acid solutions and/or neutral aqueous solutions, when present in and/or on the catalyst support, wherein the catalyst support is to be used in a catalyst-forming process involving impregnation of the support in neutral or acidic aqueous conditions;
 - the modifying component is introduced onto and/or into an untreated particulate catalyst support selected from the group consisting of Al_2O_3 , titania (TiO_2) and magnesia (MgO);
 - the resultant modifying component-containing modified catalyst support is calcined to decompose organic groups or form spinel structures with the support, at a temperature from 100°C to 800°C and for a period of 1 minute to 12 hours;
 - to obtain a protected modified catalyst support, which is less soluble or more inert in the aqueous acid

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solution and/or neutral aqueous solutions than the untreated catalyst support;

- with the modifying being present, in the modified catalyst support particles, on the particle surfaces.

Evidence that the solution effectively solves the problem

- 10. The patent in suit comprises Examples 1 to 11, whereby Examples 1, 2, 3, 7, 9, 10 and 11 are particularly relevant, for the following reasons:
- 10.1 Based on conductivity measurements, used to follow the increase of Al-ions in solution, thus the dissolution profile of treated and non-treated supports, Example 1.2 show that unprotected pure ${\rm Al}_2{\rm O}_3$ dissolves faster than silicon modified ${\rm Al}_2{\rm O}_3$, and that the higher the silica level the better.

Example 7 shows that the introduction of even a relatively small quantity of silicon suppresses the dissolution of Al_2O_3 supports.

Thus, the claimed modification effectively reduces the dissolution profile.

High Resolution Scanning Electron Microscopy (HRSEM) experiments were used in Example 1.3 to determine the presence of an amorphous hydrotalcite-like layer on the cobalt based Al₂O₃ supported catalysts prepared according to Example 60 of D3 (slurry impregnation). The presence of an amorphous layer and of submicron pure boehmite star-like crystals was observed in samples prepared with unmodified Al₂O₃ supports, not however on catalysts prepared on modified Al₂O₃ supports.

Example 1.5 (in particular Table 3 and paragraphs [0053] and [0059], Figure 4) shows that the claimed modification does not affect the intrinsic activity of the catalyst, and that a modified catalyst does not show

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any cobalt in the filtered wax product after 70 days operation (with an unprotected $\mathrm{Al}_2\mathrm{O}_3$ the wax turned grey after 10 days, as mentioned in paragraph [0055]). Example 9 shows that even a relatively small quantity of silicon suppresses the formation of cobalt rich ultrafine particulate material during the slurry phase Fischer-Tropsch process. Hence, the claimed modification effectively reduces the cobalt content in the wax product.

- 10.3 Example 2 shows that a spinel-type protected/modified support has increased resistance to dissolution.
- 10.4 Example 3 shows that titania support modified with silicon had increased resistance to dissolution.
- 10.5 Example 10 shows that zirconium modified particulate Al_2O_3 has increased resistance to dissolution.
- 10.6 It follows from the foregoing analysis that the claimed use of the modifying component effectively solves the technical problem stated in the patent in suit.

Obviousness

- 11. It remains to be decided whether the claimed solution was obvious for the skilled person starting from the closest prior art D3 with the aim of solving the problem posed, having regard to common general knowledge and the teaching of the prior art relied upon by the Appellant.
- 11.1 The main aim of D3 is to produce highly active and selective catalysts without chemical promotion. The optional chemical modification proposed by D3 is with ammonia. As shown by D26 (Figure 1), this modification however leads to an increase of dissolution in aqueous

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acidic/neutral conditions. Moreover, in order to prevent the contamination of the wax product with cobalt, D3 teaches the thorough washing of the catalyst (page 3, lines 6-10; page 16, lines 48-49). Therefore, D3 does not hint at modifying the catalyst support surface with a component as claimed in order to decrease the dissolution of the support in aqueous acidic/neutral conditions used during catalyst-forming processes.

- 11.2 D23 and D24 disclose the use of silicon as the modifying component of the surface of Al_2O_3 supports, however in order to increase their hydrothermal resistance for use in car mufflers or fluidised beds. Apart from the similarity of the treatment steps, neither D23 nor D24 hints at the purpose defined in Claim 1 at issue, which is a technical feature of the claimed use.
- 11.3 Other documents have not been invoked.
- 11.4 Therefore, the skilled person starting from D3 does not find any hint towards the use of a modifying component as claimed in order to reduce the solubility in aqueous acidic/neutral solutions used in catalyst-forming processes, and in this way reduce the undesired cobalt content in the wax product of Fischer-Tropsch processes.
- 11.5 The claimed use is not obvious over the cited prior art.

 The same applies a fortiori to the embodiments thereof defined in dependent claims 2 to 6.

Conclusion

12. The grounds of opposition pleaded by the Appellant do not prejudice the maintenance of the patent in the amended form of the sole (main) claim request filed during the oral proceedings before the Board.

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Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the Opposition Division with the order to maintain the patent on the basis of the claims of the Main Request filed at the oral proceedings on 5 February 2016 and a description to be adapted thereto.

The Registrar:

The Chairman:



S. Fabiani J. Riolo

Decision electronically authenticated