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Datasheet for the decision of 10 December 2015

Case Number: T 1371/12 - 3.3.06

Application Number: 03739388.1

Publication Number: 1476246

IPC: B01D53/86

Language of the proceedings: ΕN

Title of invention:

Process for the selective removal of sulphur compounds from synthesis gas

Patent Proprietor:

Haldor Topsøe A/S

Opponent:

Clariant Produkte (Deutschland) GmbH

Headword:

Selective sulphur compounds removal / HALDOR TOPSØE

Relevant legal provisions:

EPC Art. 52(1), 56, 83, 84, 123(2)

Keyword:

Clarity (yes)
Added matter (no)
Sufficiency of disclosure (yes)
Inventive step (yes) common general knowledge teaching away from claimed invention

Decisions cited:

T 1093/99, T 0985/06

Catchword:



Beschwerdekammern Boards of Appeal

Chambres de recours

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Case Number: T 1371/12 - 3.3.06

D E C I S I O N
of Technical Board of Appeal 3.3.06
of 10 December 2015

Appellant: Clariant Produkte (Deutschland) GmbH

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Decision under appeal: Interlocutory decision of the Opposition

Division of the European Patent Office posted on 30 March 2012 concerning maintenance of the European Patent No. 1476246 in amended form.

Composition of the Board:

Chairman B. Czech
Members: L. Li Voti

C. Heath

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Summary of Facts and Submissions

- The present appeal by the Opponent is from the interlocutory decision of the Opposition Division concerning maintenance of European patent no. 1 476 246 in amended form.
- II. In its notice of opposition the Opponent had sought revocation of the patent on the grounds of Articles 100(a), (b) and (c) EPC citing inter alia document

D1: US 4,521,387 A.

III. The Opposition Division found in its decision that the claims according to the first auxiliary request filed by letter of 13 January 2012 complied with all the requirements of the EPC.

The three claims according to said first auxiliary request read as follows (insertions as compared to the claims as granted made apparent by the Board):

- "1. A process for the selective removal of sulphur compounds from synthesis gas containing at least 5% carbon monoxide, at least 5% hydrogen and at least 0.5% carbon dioxide and containing water being present in a concentration up to saturation, at a pressure of at least 15 bar comprising contacting the synthesis gas at a maximum contact temperature below 100°C with an absorbent comprising Cu/ZnO compounds and activated with a reducing gas."
- "2. Process of claim 1, wherein the sulphur compounds comprise H_2S and COS."
- "3. Process according to any one of the preceding

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claims, wherein the synthesis gas contains ${\rm H}_2{\rm S}$ in an amount effective for suppression of metal dusting of metals in contact with the synthesis gas within a temperature range between 300°C to Boudouard temperature of the synthesis gas."

IV. In its statement of grounds of appeal, the Appellant (Opponent) maintained that claim 1 held allowable by the Opposition Division did not comply with the requirements of Article 123(2) EPC, that the invention as defined in claim 3 was not sufficiently disclosed and that the subject-matter of claims 1 to 3 lacked inventive step in the light of document D1.

Together with a further letter, it filed the following additional document:

D5: "Carbon Formation and Metal Dusting in Advanced Coal Gasification Processes" by J.H.De Van et al., Oak Ridge National Laboratory, February 1997, pages iii and 13 to 15.

In said further letter it complemented its arguments regarding the alleged insufficiency and lack of inventive step and offered a witness to be heard by the Board regarding these issues.

- V. In its reply, the Respondent (Patent Proprietor) rebutted all the objections raised and also offered a witness to be heard by the Board regarding patentability issues.
- VI. The Appellant reacted by filing, as "Annex T", the following document
 - T: Technical Report by Ms J. Reno dated 15 July 2015.

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It also reiterated all its objections and additionally called into question the clarity (Article 84 EPC) of claim 1 held allowble by the Opposition Division.

- VII. The parties were summoned to oral proceedings. In a communication dated 7 August 2015, issued in preparation for the oral proceedings, the Board expressed its provisional opinion concerning some of the pending issues raised. In particular, the Board indicated inter alia why it considered
 - that the claims at issue appeared to comply with the requirements of Article 123(2) EPC,
 - that the invention as defined in claim 3 appeared to be sufficiently disclosed, and
 - that hearing the persons named by the Parties as witnesses did not appear to be expedient or appropriate.
- VIII. In its reply thereto, the Appellant merely complemented its arguments regarding the objections under Article 123(2) EPC and the alleged lack of inventive step.
- IX. The Respondent did not reply in writing to the Board's communication.
- X. Oral proceedings were held on 10 December 2015.

XI. Requests

The Appellant requested that the decision under appeal be set aside and the patent be revoked.

The Respondent requested that the appeal be dismissed or, alternatively, that the decision under appeal be set aside and the patent be maintained based on auxiliary request 2 as filed on 9 February 2012.

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XII. The Appellant's submissions of relevance here can be summarized as follows:

Article 84 EPC

- The wording "synthesis gas containing...and containing water being present in a concentration up to saturation", could be interpreted in different ways and thus rendered the claim unclear. However, at the oral proceedings, the Appellant agreed with the interpretation submitted by the Respondent and adopted by the Board, that this wording required that some water had to be present in the synthesis gas, in a concentration up to saturation.

Article 123(2) EPC

- The feature of claim 1, according to which water is necessarily present in the synthesis gas at a concentration up to saturation, found no basis in the application as filed, at least in its combination with the other features of claim 1 at issue like, for example, a Cu/ZnO comprising absorbent (in general). Similarly, a maximum contact temperature "below 100°C" was only disclosed in the application as filed with respect to specific embodiments of the invention and could not be generalized to all embodiments encompassed by claim 1 at issue. A process pressure of at least 15 bar was also not clearly supported.
- Therefore, the combination of features according to claim 1 found no basis in the application as filed. Hence, claims 1 to 3 at issue did not meet the requirements of Article 123(2) EPC.

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Article 83 EPC

- The patent in suit did not contain any indications concerning an amount of H_2S that would be "effective for suppression of metal dusting...within a temperature range between 300°C to Boudouard temperature of the synthesis gas" (claim 3), let alone concerning a method for the determination of such amount without undue burden. Nor did the patent contain an unambiguous explanation regarding the meaning and determination of the "Boudouard temperature", the latter defining the upper limit of the temperature range defined in claim 3, let alone regarding a way to determine it.
- The embodiment of the invention as defined in claim 3 at issue was thus insufficiently disclosed.

Inventive step

- The process of example 3 of document D1 was the closest prior art for the evaluation of inventive step. This process differed from the one according to claim 1 only in that the synthesis gas treated did not contain at least 0.5% CO_2 and, possibly, water (in fact, some water could be formed during the process), and in that the operative pressure of the process (at least 15 bar according to claim 1) was not mentioned.
- However, in the light of the description of D1, it would have been obvious to the skilled person to also apply the process of example 3 to the treatment of any type of synthesis gas, for example to one produced by steam reforming and containing also water and CO_2 .
- Moreover, even if the wording "selective removal of sulphur compounds" of claim 1 at issue was interpreted

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as referring to a process wherein the amount of sulphur compounds in the synthesis gas was reduced to a level of less than 5ppb by volume and wherein less than 1000 ppm by volume of by-products not originally contained in the initial synthesis gas, e.g. methanol, were formed, it would still be obvious to the skilled person, in the light of the teaching of document D1, to apply the process of example 3 also to a synthesis gas of the type defined in claim 1 at issue, in order to effectively reduce its sulphur compounds content.

- In fact, even though it was known that a Cu/ZnO absorbent catalyses the water shift reaction in a synthesis gas containing CO and water, with production of some methanol, the skilled person would not be dissuaded from trying the process of document D1, since this document actually suggested that the disclosed process could be applied to a synthesis gas containing water and that synthesis gases could be used for methanol synthesis.
- Furthermore, the examples of the patent in suit did not show convincingly that a selective removal of sulphur compounds down to amounts of less than 5 ppb, with a formation of less than 1000 ppm by-products, may be achieved for a period of time sufficiently long to be of industrial interest. Therefore, no surprising unexpected effect was achieved by means of the claimed process.
- Moreover, the lower limits for the operative process pressure and the ${\rm CO}_2$ content in claim 1 at issue were arbitrary and could thus not support the presence of an inventive step.
- The claimed subject-matter thus lacked inventive step.

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XIII. The Respondent submitted in essence that:

- Claim 1 at issue was clear and concerned the treatment of a synthesis gas containing water.
- The amendments made to the claims complied with the requirements of Article 123(2) EPC.
- The process as defined in claim 3 was sufficiently disclosed.
- The technical problem dealt with and solved by the claimed invention, taking example 3 of D1 as closest prior art, consisted in the provision of a process for the selective removal of sulphur compounds (within the meaning of the patent in suit) from a synthesis gas containing at least 5% carbon monoxide, at least 5% hydrogen and at least 0.5% carbon dioxide and containing water being present in a concentration up to saturation.
- Examples 4 and 5 of the patent in suit showed that the claimed process led to the intended selective removal of sulphur compounds, i.e. a removal process wherein the relative amounts of the synthesis gas components other than impurities, steam and inert gas, were not changed to any substantial degree, and wherein the concentration of sulphur compounds in the treated synthesis gas was reduced to below 5 ppb.
- Document D1 did not suggest the application of the process disclosed therein to a synthesis gas containing water. To the contrary, since it was common knowledge that Cu/ZnO absorbents, like that used in example 3 of document D1, catalyse the water shift reaction in a gas containing CO and water, with formation of methanol, the skilled person would have been dissuaded from applying

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the process of D1 to a synthesis gas of the type defined in claim 1 at issue for achieving a selective removal of sulphur compounds as intended in the patent in suit.

- Therefore, the skilled person would not have expected that a selective removal of sulphur compounds from the synthesis gas could be achieved by contacting the synthesis gas of claim 1 at issue with a Cu/ZnO comprising absorbent activated with a reducing gas, at a pressure of at least 15 bar and at a maximum contact temperature below 100°C.
- The claimed subject-matter thus involved an inventive step.

Reasons for the Decision

Respondent's main request (claims 1 to 3 held allowable by the Opposition Division)

- 1. Clarity (Article 84 EPC) claim 1
- 1.1 The Board holds that the particular wording "synthesis gas containing ... and containing water being present in a concentration up to saturation" in claim 1 can be understood to mean that at least some water (steam) must be present in the synthesis gas treated, which water is contained in a concentration up to saturation.

This understanding is technically sensible and fully consistent with the description of the patent in suit (see e.g. paragraphs [0001], [0002], [0014], [0036], [0037] and [0040]).

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At the oral proceedings, the Appellant expressly agreed to this reading (see point XII, *supra*).

- 1.2 The Board is thus satisfied that claim 1 complies with the clarity requirement of Article 84 EPC.
- 2. Compliance with the requirements of Article 123(2) EPC claims 1 to 3
- 2.1 Claim 1 at issue (wording under III, *supra*) differs from claim 1 of the application as filed (in this respect, reference is made hereinafter to the published international application WO 03/068370 A1) only in that
 - A the word "optionally" before "containing water ..." has been deleted and the wording "being present" has been inserted between "containing water" and "in a concentration ...";
 - B a comma has been inserted before the wording "at a pressure of at least 15 bar"; and
 - C the wording "a maximum contact temperature of $100\,^{\circ}$ C" has been amended into "a maximum contact temperature below $100\,^{\circ}$ C" (emphasis added by the Board).

For the Board, all these amendments find sufficient support in the application as filed.

2.1.1 Amendment A

Claim 1 of the application as filed, containing the expression "synthesis gas containing...and optionally containing water...", is clearly directed to both i) a process wherein the synthesis gas to be treated does not contain water, as well as to

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ii) the alternative process wherein at least some water is contained in the synthesis gas.

For the Board, the insertion of the wording "being present" between "containing water" and "in a concentration..." only clarifies beyond any doubt that water must be present. Therefore, its insertion contributes, together with the deletion of the word "optionally", to limit the claim to process alternative ii), according to which some water must be present in the synthesis gas.

Therefore, amendment A is clearly supported by the wording of claim 1 of the application as filed taken alone.

2.1.2 Amendment B

The Appellant argued that since in claim 1 of the application as filed there was no comma before the wording "at a pressure of at least 15 bar", the pressure value represented the partial pressure of the water optionally contained in the synthesis gas, and not the operative pressure of the claimed process as in the amended claim 1 with the comma inserted before the pressure figures.

For the Board, the skilled person, taking also into account the whole content of the application as originally filed, would doubtlessly understand that the pressure values indicated in original claim 1 designate the operative pressure of the claimed process. In fact, the description of the application as filed (page 3, lines 24 to 26, and page 8, line 6) indicates explicitly a range of 15 to 80 bars for the operative pressure of the process, and operative pressure values within this

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range are used in examples 4 and 5 (see page 7, lines 11 and 15, as well as page 16, lines 2 and 22, and page 18, line 17). Moreover, the description is silent about any partial pressure value of water or of any other synthesis gas component.

Therefore, for the Board, the pressure indicated in claim 1 of the application as filed is the operative pressure of the process, and the insertion of a comma before the pressure-defining feature in claim 1 at issue does not amount to any change in meaning, i.e. to any modification of the subject-matter so defined, compared to claim 1 of the application as filed.

2.1.3 Amendment C

Claim 1 of the application as filed recites (emphasis added) a "maximum contact temperature of $100\,^{\circ}\text{C"}$, whereby the temperature of $100\,^{\circ}\text{C}$, as well as temperature values below $100\,^{\circ}\text{C}$, are encompassed.

It is specifically indicated in the application as filed at page 8, lines 3 to 4, where reference is made to a typical synthesis gas from downstream reformers, that (emphasis added) "the synthesis gas is typically saturated with steam at temperatures below 100°C". Since claim 1 at issue is limited to the use of a synthesis gas containing water (steam) up to saturation, the maximum operative contact temperature has thus to be one at which steam saturation occurs, for example a temperature below 100°C as indicated in the above mentioned passage of the description.

Therefore, for the Board, the skilled person, taking into account the whole disclosure of the original application, understands that the specific disclosure

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mentioned above is a feature generally applicable to the claimed process and not restricted to the use of a specific synthesis gas.

In fact, also other passages of the description recite explicitly a maximum contact temperature "below 100°C" (see, for example, the passage on page 8, lines 25 to 30, concerning the use of a specific catalyst Cu/ZnO/Al₂O₃ or the passage from page 11, line 32 to page 12, line 4, concerning the selective removal of sulphur compounds after a step wherein metal dusting is suppressed). Moreover, also example 4 of the invention is carried out at a maximum contact temperature below 100°C, namely at 40°C (see page 16, line 22, in combination with page 16, line 1, of the application as filed).

Therefore, for the Board, the skilled person would derive undoubtedly from the overall disclosure of the application as filed that the maximum contact temperature for the claimed process can be 100°C or "below 100°C ".

- 2.1.4 The combination of features of claim 1 at issue is thus fairly supported by the application as filed.
- 2.2 The wordings of claims 2 and 3 are identical to those of claims 2 and 3 of the application as filed.
- 2.3 In the cases underlying decisions T 985/06 of 16 July 2008 and T 1093/99 of 22 May 2001, cited by the Appellant in its statement of grounds, the amendment of a specific numerical upper limit (T 985/06, Reasons, 2.1) or lower limit (T 1093/99, Reasons, 2.2) of a range to an upper limit "below" or a lower limit "greater than" the original specific numerical limits were found

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to contravene the requirements of Article 123(2) EPC in the absence of a literal basis for "below" and "greater than" .

However, the rationale of these decisions is not applicable to the present case, where values "below $100\,^{\circ}\text{C"}$ are disclosed verbatim in the description of the application as filed.

- 2.4 The Board thus concludes that amended claims are fairly based on the content of the application as filed and thus comply with the requirements of Article 123(2) EPC.
- 3. Sufficiency of disclosure (Article 83 EPC) claim 3
- 3.1 Claim 3 concerns a process "wherein the synthesis gas contains H_2S in an amount effective for suppression of metal dusting of metals in contact with the synthesis gas within a temperature range between 300°C to Boudouard temperature of the synthesis gas."
- 3.2 The Appellant disputed in writing sufficiency of disclosure with regard to claim 3, arguing that the description of the patent in suit did not contain any teaching concerning the amount of H₂S "effective for suppression of metal dusting...within a temperature range between 300°C to Boudouard temperature of the synthesis gas", let alone concerning a method for the determination of such amount without undue burden. Nor did the patent contain an unambiguous explanation regarding the meaning and determination of the "Boudouard temperature", the latter defining the upper limit of the temperature range defined in claim 3, let alone regarding a way to determine it.
- 3.3 The Appellant did not reply to the provisional opinion

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of the Board expressed in the Board's communication (see point VII above) and it did not present additional arguments concerning the alleged insufficiency at the oral proceedings. Therefore, the Board has no reason to diverge from its provisional opinion that the invention as defined in claim 3 is insufficiently disclosed for the following reasons.

- 3.3.1 Firstly, process claim 3 does not require, as a process step, the actual suppression of metal dusting at a given elevated temperature. It only requires that that the synthesis gas treated must contain a certain amount of H_2S "effective for suppression of metal dusting ... within a temperature range between 300°C to Boudouard temperature of the synthesis gas".
- 3.3.2 Therefore, the only point to be addressed with regard to sufficiency is whether the skilled person would have been able, at the priority date of the patent in suit, on the basis of the description of the patent and common general knowledge, to determine relative amounts of $\rm H_2S$ meeting the stated conditions.

The other issues raised by the Appellant, e.g. concerning the exact meaning of the term "Boudouard temperature", concern rather the clarity of (the exact boundaries of) the claim, but do not imply that the claimed invention is insufficiently disclosed.

3.4 The description of the patent in suit discloses the addition of 1 ppm H_2S to the synthesis gas in example 1 (page 5, line 49) and of 3.2 ppm H_2S in example 4 (page 7, line 9 in combination with page 6, line 50). Moreover, it is undisputed that it was well known that H_2S can be added to a synthesis gas for suppressing metal dusting. In this respect, reference is made to the

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patent in suit (page 5, line 15), as well as to the information content of document D5, representing common general knowledge in the field of metal dusting suppression (page 15, lines 8 and 9 below figure 6). D5, filed by the Appellant (see IV, supra), was relied upon by both parties and therefore admitted and considered by the Board (Article 114(2) EPC). Document D5 indicates in fact that amounts of 20 to 200 ppm H₂S "can effectively eliminate metal dusting" (page 15, second paragraph below figure 6) and that water vapour has also a "beneficial effect" in this respect (page 15, penultimate sentence).

Therefore, the Board is convinced that in the light of the teaching of the patent in suit and common general knowledge, the skilled person disposed of sufficient information to be able to identify an amount of $\rm H_2S$ suitable to provide at least some suppression (i.e. not necessarily full elimination) of the dusting of metals in contact within the synthesis gas, at temperatures of 300°C or more, i.e. up to the "Boudouard temperature". From the patent in suit (paragraph [0049]), it can be gathered that the latter is the temperature below which there "is affinity for carbon precipitation", i.e. dusting precipitation, and that it is "typically in the range up to $1000\,^{\circ}\mathrm{C}$ ".

3.5 Hence, in the Board's judgement, the invention as defined in claim 3 is disclosed in the patent in suit in manner sufficiently clear and complete to be carried out by the person skilled in the art considering the information and guidance given in the description and taking into account common general knowledge (Article 83 EPC).

- 4. Inventive step
- 4.1 The invention
- 4.1.1 The patent in suit (see paragraphs [0001] and [0040]) concerns a process for the selective removal of sulphur compounds, in particular H_2S , from synthesis gas rich in carbon monoxide and further containing hydrogen, carbon dioxide and water (steam).
- 4.1.2 The expression "selective removal" is expressly defined in the description (paragraph [0002]) as (emphasis added) "a removal process being basically neutral with respect to the reactants contained in the synthesis gas, i.e. contents of other than impurities, steam and inert gas, is not changed to any substantial degree [sic]. In addition, formation of components not already contained in the synthesis gas must be avoided. In praxis sidereactions must be suppressed to a level lower than 1000 vol ppm levels."

Moreover, in paragraphs [0007] and [0012] it is specifically indicated in this connection that (emphasis added) "[a]s the catalysts in the downstream synthesis section are susceptible to sulphur poisoning, sulphur must be removed to the low ppb level (<5ppb)" and "[w]hen operating the removal process in praxis, selective removal of sulphur requires reduction of sulphur concentration to below 5 ppb in the treated synthesis gas."

4.1.3 It was not in dispute at the oral proceedings that taking into account these explicit indications in the description, the feature "selective removal of sulphur compounds" had to be understood in the sense that the process according to claim 1 must result in a sulphur

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compounds level, in the treated synthesis gas, of below 5 ppb by volume, and in a level of less than 1000 ppm by volume of by-products not already contained in the initial synthesis gas.

- 4.2 Closest prior art
- 4.2.1 Both parties considered document D1 and, in particular, the process of example 3 of D1, to represent the closest prior art.

Considering the similarities between the patent in suit and D1 in terms of the finality and the features (infra) of the processes concerned, the Board has no reason to take another stance and holds that the process of example 3 of D1 is indeed the most appropriate starting point for the assessment of inventive step.

- 4.2.2 More particularly , example 3 of D1 describes a process for removing sulphur compounds from a synthesis gas down to a concentration below the detection level (see in particular column 5, lines 13 to 14). The synthesis gas treated consists of about 48.5 vol % CO and 51.5 vol% $\rm H_2$. In a first step, it is mixed with a small amount of oxygen and contacted at 100°C with active carbon in order to remove metal carbonyl compounds, and in a second step the gas leaving active carbon is passed without further treatment over a Cu/ZnO catalyst previously activated with a reducing gas (D3, column 3, lines 32 to 34: " $\rm H_2/N_2$ mixture").
- 4.2.3 In the absence of any corroborating evidence, the Board does not accept the argument of the Appellant, that small amounts of water would be inevitably formed during the process of example 3, and that the presence of water

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was thus not a distinguishing feature of claim 1 at issue.

As regards the temperature prevailing during the sulphur compounds removal step of example 3, the Appellant acknowledged in writing and during oral proceedings that the temperature of the gas being treated will drop down at least slightly from the intial 100 °C during the passage of the synthesis gas through the active carbon layer and that, therefore, the temperature in the sulphur removal step will thus necessarily be below 100°C. The Respondent did not contest the Appellant's statement. In the following, the Board thus accepts its validity for the sake of argument and in the Appellant's favour.

4.3 Technical problem

For the Board, the technical problem consists in the provision of a process for the "selective removal of sulphur compounds" (within the meaning of the patent in suit; see 4.1.3, *supra*) from a synthesis gas containing at least 5% carbon monoxide, at least 5% hydrogen and at least 0.5% carbon dioxide and containing water, wherein water is present in a concentration up to saturation.

4.4 The solution

As the solution to this technical problem the patent in suit, in its amended version held allowable by the Opposition Division, proposes the process according to claim 1 at issue, which is characterised in particular in that a synthesis gas having such a composition is "contacted", "at a pressure of at least 15 bar" and "at a maximum contact temperature below 100°C", "with an absorbent comprising Cu/ZnO compounds and activated with

a reducing gas".

4.5 Success of the solution

4.5.1 Example 4 of the patent in suit describes a process wherein a synthesis gas containing, by volume, 16.3% CO (carbon monoxide), 70.48% H₂ (hydrogen), 10.2% CO₂ (carbon dioxide), 0.02% H_2O (water), 3% Ar (argon), 3.2ppm H_2S (hydrogen sulfide) and 70 ppb COS (carbonyl sulfide) is contacted at 40°C and at a pressure of 20 bar with a reduced Cu/ZnO2/Al2O3 absorbent (see paragraph [0077] in combination with paragraphs [0073] and [0074]). As already remarked in the Board's communication, this example shows (paragraph [0078] and figure 4) that desulphurization to the required low level of less than 5 ppb (page 7, lines 6 to 7) is achieved for about 200 hours, whereafter a breakthrough of COS is observed. H2S remains, nevertheless, below the detection limit of 5 ppb for at least up to 300 hours, i.e. for the whole duration of the test.

The Board has no reason to doubt the correctness of the statements contained in this example (page 7, lines 6 and 7: "desulphurisation to the required low ppb level (5ppb) is obtained for about 200 hours, wherefafter a breakthrough of COS is seen"), which statement appears to be confirmed by the data reported in figure 4. Hence, the Board does not accept the Appellant's allegation, re-iterated at the oral proceedings, that a sulphur compounds breakthrough (beyond the upper concentration limit implied by claim 1 at issue, i.e. resulting in levels of more than 5 ppb) would occur well before 200 hours under the conditions of example 4. In fact, the Appellant's allegations are based on a hypothetical curve drawn on the basis of the data points reported in figure 4 (page 5 of the Appellant's letter dated

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30 November 2015). Said curve does not, however, take into account the clear and express indications in example 4 that no breakthrough of COS was observed for about 200 hours.

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Therefore, in the absence of experimental counterevidence, the Board accepts that example 4 shows that no breakthrough of sulphur compounds occurred in the tests described within the first 200 hours.

4.5.2 It is also stated in example 4 (page 7, lines 9 to 12), that the experiment was "conducted with 10 times the normal space velocity and at 5 times the required H_2S level. Therefore, an estimated operation time for an industrial application will be at least 50 times the experimental run time".

Thus, 200 hours in example 4 correspond to 10000 hours of industrial operation below the H_2S detection limit, i.e. a significant time for industrial application.

The Appellant contested the correspondence of the experimental times with industrial operative times made in the example. However, it did not provide evidence showing that such a correspondence was incorrect.

The Board has thus no reason to doubt that the results of example 4 are indeed significant as regards the industrial applicability of the claimed process .

4.5.3 The Appellant also submitted in writing (page 6 of the Appellant's letter dated 30 November 2015) another set of calculations made by transposing the results of the process outlined in example 4 into a hypothetical one carried out with an amount of H_2S being 6 to 60 times greater (20 to 200 ppm compared to 3.2 ppm), i.e. an

amount of H_2S as considered appropriate in D5 for eliminating metal dusting. On the basis of these calculations, the Appellant alleged that with such higher relative amounts of H_2S in the sythesis gas a breakthrough of sulphur compounds would occur much earlier, and that such a process would thus not have industrial applicability. Therefore, a selective removal of sulphur compounds could not be achieved in the Appellant's view throughout the whole breadth of claim 1.

The Board notes that the Appellant's allegations are based on calculations, the foundation of which is not supported by any evidence, and that the Appellant did not file experimental data directly comparable to those presented in the patent in suit. Hence, for the Board, the Appellant's allegations are not proved up to the necessary standard. They are thus disregarded as unconvincing in the following.

4.5.4 In Example 4, it is also clearly indicated that in the synthesis gas used "the potential for methanol synthesis is far more than 1000 ppm" (page 7, line 1). In Example 4 it is not tested whether the used absorbent catalyses the water gas shift reaction under the specified conditions.

With regard to the (undesired) formation of methanol, example 5 describes tests run with a different synthesis gas containing (see paragraph [0035]) 14.1% CO, 70.53% $\rm H_2$, 9.72% CO₂, 2.92% Ar and 2.72% $\rm H_2O$. This gas is contacted with a reduced Cu/ZnO absorbent at a pressure of 20 bar and at temperatures of 148, 125 and 110°C, respectively. According to example 5, "[a]t 110°C conversion was hardly to be observed...By extrapolation of the test results it shows that at 100°C and at a

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typical industrial space velocity of 2500 $\rm Nm^3/m^3$ catalyst/h, the conversion of CO will be only a fraction of a per thousand. In other words, and for practical purposes, the $\rm Cu/ZnO/Al_2O_3$ absorbent does not catalyse the water gas shift reaction at 100°C or below."

The burden of proof lies with the Appellant to show that these statements are not correct. The Appellant merely objected that the method for carrying out the extrapolation addressed to in example 5 is not explained. It did thus not discharge the burden of proof in this respect, and the Board thus sees no reason for doubting the correctness of said statemetns.

4.5.5 Referring to experimental report T, in which a different synthesis gas was tested, the Appellant called into question in writing the results indicated in example 5 of the patent. However, the Appellant did not rely on this test at the oral proceedings.

The Respondent remarked instead that the test did not indicate whether methanol was actually formed as byproduct and in which amount. This fact was not disputed by the Appellant. The Board sees no reason to believe that such an isolated test, wherein a clear indication of the amount of byproducts (methanol) obtained is missing, could throw doubts on the validity of the statement contained in example 5 of the patent in suit, discussed above.

Moreover, as also indicated by the Board during oral proceedings, the objection raised by the Appellant in writing, namely that a process as claimed would not necessarily lead to a sufficient suppression of byproducts formation, appears rather to concern sufficiency of disclosure and not inventive step, as

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claim 1 at issue is indeed limited to processes wherein less than 1000 ppm by-products are formed.

- 4.5.6 Summarising, taking into account in particular the exemples of the patent in suit, the Board is convinced that the technical problem posed (4.3, *supra*) is indeed successfully solved by the process of claim 1.
- 4.6 Obviousness of the solution
- 4.6.1 The process of example 3 (see details under 4.2.2 and 4.2.3, *supra*) differs from the subject-matter of claim 1 at issue in that
 - the synthesis gas treated does not contain at least $0.5\%~{\rm CO}_2$ and does not contain water; and
 - the pressure used in the sulphur removal step is not indicated.
- 4.6.2 The evaluation of inventive step thus boils down to the question whether in the light of the teaching of document D1 and of common general knowledge, it would have been obvious to the skilled person to apply the desulphurisation step of example 3 to a synthesis gas additionally containing at least 0.5% CO₂ and water in a concentration up to saturation, at a pressure of at least 15 bar, in order to obtain a desulphurised gas containing less than 5 ppb sulphur compounds and less than 1000 ppm by-products such as methanol.
- 4.6.3 It is undisputed that document D1 teaches that the gas purification process described therein can be applied to synthesis gas also containing CO_2 (see e.g. column 1, lines 5 to 7, and column 2, lines 7 to 9).

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4.6.4 However, this document fails to give any explicit indication that the synthesis gas to be treated may also contain water (steam). In fact, even though it is undisputed that

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- synthesis gases generated in reformers comprise conventionally also steam (see e.g. paragraph [0013] of the patent in suit) and
- document D1 explicitly refers also to synthesis gases produced by steam reforming (column 1, lines 8 to 9),

water is not mentioned therein as a component of the gas to be treated according to the process disclosed and claimed therein (D1: claim 1).

4.6.5 Water is indeed only mentioned in D1 (column 1, lines 57 to 65), after a discussion of prior art processes intended to remove large amounts of impurities from gas streams (in general), with respect to a process involving the use of molecular sieves for removing small amounts of impurities. The latter process is said to remove, however, also harmless components like water or CO₂.

Therefore, even this passage relates only to prior art processes and does not concern, at least not explicitly, the treatment of a water-containing synthesis gas in the ambit of the process of claim 1 of D1.

4.6.6 Hence, for the Board, document D1 does not contain any explicit suggestion to apply the desulphurisation step of the process of example 3 to a synthesis gas additionally containing water as defined in claim 1 at issue.

4.6.7 Moreover, it is undisputed that Cu/ZnO absorbents were known to catalyze the water shift reaction in a gas containing CO and water, according to the reaction mechanism indicated in the patent in suit (see paragraphs [0026] and [0027]), with the formation of H_2 and CO_2 , which may react further with each other to form methanol as a by-product.

Therefore, the methanol synthesis occurring in such systems can potentially proceed to a level of methanol much higher than 1000 ppm. This is illustrated by the theoretical calculations listed in table 1 of the patent in suit with regard to the behaviour of synthesis gases C, D and E at equilibrium at temperatures of 100°C and below 100°C, and is also stated in paragraph [0076] of the patent in suit.

4.6.8 In the Board's judgement, the skilled person, in the light of common general knowledge regarding the behaviour of water containing synthesis gases at elevated temperatures in the presence of a Cu/ZnO absorbent, would thus have expected that applying the desulphurisation step of the process of example 3 of D1 to a synthesis gas additionally containing water would result in the undesired formation of a substantial amount of by-products, in particular methanol.

Therefore, the Board concludes that the skilled person, faced with the technical problem posed (4.3, supra), imposing a limit (less than 1000 ppm) regarding the maximum concentration of by-products that may be formed (4.1.3, supra), would not, without the benefit of hindsight, have considered solving said problem by treating the water-containing synthesis as defined in claim 1 using the process described in example 3 of D1.

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- 4.6.9 For the Board, even the indication, in document D1, that synthesis gases can be used for methanol synthesis (column 1, lines 12 to 13), which fact undoubtedly belongs to common general knowledge, does not even implicitly incite the skilled person to try applying the desulphurisation step of the process of D1/example 3 also to the desulphurisation of water-containing synthesis gases. Instead, doing so, he would expect the water shift reaction to occur simultaneously with the desulphurisation, with production of an unwanted level of methanol as by-product. Moreover, document D1 merely concerns explicitly the fine purification of gases containing H_2 , CO and/or CO_2 for the removal of sulphur compounds and other impurities (column 2, lines 7 to 10). Also for this reason, D1 does not suggest, in the Board's view, the application of the desulphurisation step of example 3 to a synthesis gas comprising water.
- 4.6.10 Summarising, the Board is convinced that both the teaching of document D1 as such and common general knowledge would actually have dissuaded the skilled person from trying to use the sulphur removal step of example 3 of D1 to desulphurise a water-containing synthesis gas having a composition as defined in claim 1 at issue. Furthermore, the skilled person had no particular reason to expect that subjecting such a synthesis gas to this step at a maximum contact temperature below 100°C and at a pressure of at least 15 bar would lead to the "selective removal" of sulphur compounds within the meaning of the patent in suit (4.1.3, supra).
- 4.6.11 The Board thus concludes that the subject-matter of claim 1 and consequently, the subject-matters of dependent claims 2 and 3, involve an inventive step (Articles 52(1) and 56 EPC).

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5. Conclusion

None of the objections raised by the Appellant prejudices the maintenance of the patent in the version held allowable by the Opposition Division.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



D. Magliano

B. Czech

Decision electronically authenticated