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**Datasheet for the decision  
of 15 April 2010**

**Case Number:** T 0730/07 - 3.3.06

**Application Number:** 99935183.6

**Publication Number:** 1098952

**IPC:** C10G 65/08

**Language of the proceedings:** EN

**Title of invention:**  
Hydrogenation process

**Patentee:**  
ENGELHARD CORPORATION

**Opponent:**  
Johnson Matthey PLC

**Headword:**  
Hydrogenation/ENGELHARD

**Relevant legal provisions:**  
EPC Art. 114(2), 56

**Relevant legal provisions (EPC 1973):**  
-

**Keyword:**  
"New evidence - not filed in due time"  
"Inventive step - no"

**Decisions cited:**  
T 0201/92, T 0951/91

**Catchword:**  
-



Case Number: T 0730/07 - 3.3.06

**DECISION**  
of the Technical Board of Appeal 3.3.06  
of 15 April 2010

**Respondent:**  
(Opponent)

Johnson Matthey PLC  
2-4 Cockspur Street  
Trafalgar Square  
London SW1Y 5BQ (GB)

**Representative:**

Gibson, Sara Hillary Margaret  
Intellectual Property Department  
Johnson Matthey Catalysts  
P.O. Box 1  
Belasis Avenue  
Billingham  
Cleveland TS23 1LB (GB)

**Appellant:**  
(Patent Proprietor)

ENGELHARD CORPORATION  
101 Wood Avenue  
Iselin  
New Jersey 08830-0770 (US)

**Representative:**

Oudshoorn, Otto Leonard  
Vereenigde  
Johan de Wittlaan 7  
P.O. Box 87930  
NL-2508 DH Den Haag (HL)

**Decision under appeal:**

Interlocutory decision of the Opposition  
Division of the European Patent Office posted  
21 February 2007 concerning maintenance of  
European patent No. 1098952 in amended form.

**Composition of the Board:**

**Chairman:** P.-P. Bracke  
**Members:** G. Dischinger-Höppler  
J. Van Moer

## Summary of Facts and Submissions

I. This appeal is from the interlocutory decision of the Opposition Division concerning maintenance of the European patent No. 1 098 952 in amended form on the basis of the then pending auxiliary request, the independent Claim 1 reading:

"1. Process for the hydrogenation of a sulfur containing feedstock, having a sulfur content of less than 50 ppm, wherein the feedstock is hydrogenated in the presence of a precious metal catalyst, the precious metal being selected from platinum, palladium, rhodium, ruthenium, iridium, osmium and alloys thereof, such as platinum-palladium, and a nickel catalyst, said process being carried out in such a manner, that the feedstock is contacted initially with the precious metal catalyst followed by contact with the metaloxide and then followed by the nickel catalyst, and wherein the metal oxide has been selected from the oxides of silver, lanthanum, antimony, bismuth, cadmium, lead, tin, vanadium, calcium, strontium, barium, cobalt, copper, tungsten, zinc, molybdenum, manganese and iron."

II. A notice of opposition had been filed against the granted patent, wherein the Opponent sought revocation of the patent on the grounds of, inter alia, Article 100(a) EPC for lack of inventive step (Article 56 EPC). The opposition was based, amongst others, on documents

D5 WO-A-97/03150,

D6 EP-A-0 398 446 and

D7 US-A-5 059 304.

- III. In its decision, the Opposition Division held that the subject-matter claimed in accordance with the auxiliary request fulfilled the requirements of the EPC. The higher ranking main request was not allowed for the reason that its subject-matter (claims as granted) did not meet the requirements of Article 56 EPC in view of the disclosure of documents D5 and D6, in particular if the sulphur is present in the form of thiophenes.
- IV. This decision was appealed by the patent Proprietor, now Appellant, who filed amended sets of claims in a new main request and first auxiliary request under cover of a letter dated 3 July 2007 and experimental evidence under cover of a letter dated 16 December 2009. The claims as maintained by the Opposition Division were also maintained.
- V. Claim 1 of the main request reads:
- "1. Process for the hydrogenation of a sulfur containing feedstock, having a sulfur content of less than 50 ppm, wherein the feedstock is hydrogenated in the presence of a precious metal catalyst, the precious metal being selected from platinum, palladium, rhodium, ruthenium, iridium, osmium and alloys thereof, such as platinum-palladium, and a nickel-catalyst, said process being carried out in such a manner, that the feedstock is contacted initially with the precious metal catalyst followed by contact with the metal oxide and nickel catalyst in combination or first with the metal oxide and then with the nickel catalyst, or that the

feedstock is contacted first with a mixture of precious metal catalyst and metal oxide, followed by contact with the nickel catalyst, and wherein the metal oxide has been selected from the oxides of silver, lanthanum, antimony, bismuth, cadmium, lead, tin, vanadium, calcium, strontium, barium, cobalt, copper, tungsten, zinc, molybdenum, manganese and iron."

Claim 1 of the first auxiliary request differs there from in that the terms "initially" and "nickel catalyst in combination or first with the metal oxide and" have been deleted.

VI. Upon request by the Appellant, oral proceedings before the Board of Appeal had been scheduled under cover of a letter dated 10 September 2009 for 10 February 2010. On 8 February 2010, these proceedings were postponed. They were held on 15 April 2010 in the absence of the Opponent, now Respondent, as announced by letter dated 18 March 2010.

VII. The Appellant, orally and in writing, submitted in essence that document D5 taught to improve the teaching of document D6 by using precious metal followed by nickel instead of a combination of hydrogenation component and metal oxide. Thus, there was no incentive to combine these documents in order to arrive at the claimed embodiments which all allowed operating the process within a widened temperature window and which showed improved properties over documents D5 and D6 as illustrated in the experiments filed with letter dated 16 December 2009. In any case there was no incentive to combine precious metal and metal oxide in a first step, followed by nickel in a second step. Rather, there

existed a prejudice against using metal oxide in the process of document D5. Document D7 was not relevant since its product was comparable with the starting feed of the claimed process.

The experimental evidence was filed in reaction to the contested decision and was not at a disadvantage to the Respondent who could have made the same or even accelerated tests.

VIII. The Respondent, in writing, requested not to admit the experimental evidence into the proceedings for being too late filed and insufficiently described for being verified by the Respondent. Further it was submitted that according to document D6 the technical problem of deactivation of the hydrogenation catalyst by sulphur compounds was solved by providing in addition to the hydrogenation catalyst a metal oxide component either separately or in a mixture. Since document D5 proposed to improve the process of document D6 by contacting the feed with a platinum group metal prior to or simultaneously with the nickel catalyst, the claimed subject-matter was not based on an inventive step. The Respondent also maintained its arguments provided during opposition proceedings.

IX. The Appellant requested that the decision under appeal be set aside and the patent be maintained in amended form on the basis of the new main or first auxiliary request.

The Respondent requested that the appeal be dismissed.

## Reasons for the Decision

### 1. *Admissibility of late filed evidence*

The Appellant filed experimental evidence under cover of a letter dated 16 December 2009, i.e. more than three months after the summons to oral proceedings, less than two month before the date for which oral proceedings have been scheduled originally and about four months before the oral proceedings actually took place.

The Appellant explained that the experiments have been carried out to provide additional support for the claimed subject-matter since it had become clear from the decision of the Opposition Division that experimental support was deemed to be essential. As the Respondent could easily have done the same or even accelerated tests, there was no reason to consider the experimental data a disadvantage to the Respondent. Further, the experiments have been sufficiently described since it was clear to the skilled reader that all experiments have been carried out with the same feedstock, namely Varsol 80, which contained less than 1 ppm of sulphur. In all experiments, the materials used were identical, i.e. the nickel, palladium and zinc oxide materials were taken from the same batch.

The Respondent requested not to admit that evidence for being too late filed and insufficiently described for enabling the Respondent to verify the results by own experimentation in the period allowed before oral proceedings.

- 1.1 New facts and evidence filed for the first time during appeal proceedings may be disregarded by the board by virtue of Article 114(2) EPC, if they have not been submitted in due time.

According to the established case law of the Boards of Appeal, filing new facts and evidence would be considered to be in due time, if the filing was occasioned by an argument or a point raised by another party or in the appealed decision. However, in order not to deprive the other parties from their right to verify the new evidence or to prevent the board from ensuring that the proceedings are conducted expeditiously, such facts and evidence have to be submitted once they were available and once it has become clear that they were relevant (e.g. T 201/92, not published in the OJ EPO, points 3.4 to 3.6 of the reasons; T 951/91, OJ EPO 1995, 202, points 5.5 and 5.15 of the reasons).

- 1.2 The new evidence contains experiments conducted over a period of 656 days in total. Yet, the filing of the evidence has never been announced by the Appellant.

Assuming in the Appellant's favour that the experiments have been just finished about the date of filing them, i.e. on the 15<sup>th</sup> or 16<sup>th</sup> of December 2009, they would have been started on the 28<sup>th</sup> or 29<sup>th</sup> of February 2008, which is more than three months after the Respondent's reply dated 14 November 2007 to the Appellant's statement of grounds of appeal dated 3 July 2007, i.e. seven months after the statement of grounds of appeal, and about one year after the decision of the Opposition Division has been issued. Hence, the experiments might



be regarded as an attempt to rebut the arguments provided in the Respondent's letter of reply, rather than a reaction to the contested decision, even if account is taken of the Appellant's argument that preparations were necessary in order to be able to start the tests.

- 1.3 However, for the following reasons it is apparent that the relevance of the experiments was clear already at a much earlier point in time:

The new evidence contains three comparative examples (Examples 1, 5 and 6) deemed to illustrate the prior art, *inter alia*, according to documents D5 and D6 and three examples (Examples 2 to 4) which are said to be according to the invention as defined in the main request. In all examples the time for the conversion of aromatics to fall from 100% to 90% was measured as an indication of the deactivation of the catalyst by sulphur. The evidence shows a deactivation to 90% conversion for the best performing comparative example (Example 5) after 4750 hours (about 198 days). It is further shown that 750 hours (about 31 days) more are required by the worst performing invention example (Example 2) which is an embodiment of the main request and 3500 hours (about 146 days) more are required for invention example 3 which is an embodiment of both, the main and the first auxiliary request. At that time it was already known that the third invention example 4, the only experiment still running, was the best performing embodiment. Hence, the quality of the performance of the invention examples according to the main and first auxiliary requests in comparison with the comparative examples was already known after 5500

hours (229 days) or 8250 hours (344 days), which is 429, respectively 321 days earlier than the filing date of the new evidence.

It is self-evident that the period of time remaining between the filing of the new evidence and the date for oral proceedings was far too short for the Respondent to verify the experiments. However, the time for doing so would have been sufficient, if the evidence had been filed as soon as possible.

Thus, the Appellant did not submit the new evidence in due time, i.e. once it was available and once it has become clear that it was relevant, but about one year later and in particular at a time at which it was apparent that the Respondent would have no chance to verify the new evidence without a considerable delay of the proceedings.

The Appellant's argument that the Respondent could have performed the same or accelerated experiments is not convincing because there was simply no reason for the Respondent to do so. Moreover, if it was possible to carry out similar tests under accelerated conditions, it would have been the Appellant's duty to do so in order to safeguard the Respondent's procedural rights.

Apart from that, the Board agrees with the Respondent that the experimental data are insufficient with respect to the feedstock and Pd catalysts used to enable the work to be repeated. The Appellant's allegation that the examples have all been carried out with Varsol 80 from Exxon which contains less than 1 ppm of sulphur is in contradiction with the test report

according to which the feed contained 3 ppm of sulphur. Considering further that only in two experiments (experiments 4 and 5) the particular palladium catalyst Pd 0155 S is mentioned, it is not convincing that all catalytic material has been taken from the same batch. Hence, it is doubtful from the outset whether an adjournment of the proceedings could have brought about any new insight helpful for the outcome in the present case.

1.4 Therefore, the Board finds that the submission of the new evidence at such a late stage is unacceptable so that it is justified, in the exercise of the discretion under Article 114(2) EPC, to reject that evidence.

## 2. *Inventive Step*

2.1 The patent in suit is directed to a process for hydrogenating a sulphur containing feedstock, in particular feedstock which had been treated by conventional hydrodesulphurisation (HDS) where the sulphur level left is 50 ppm or less (paragraphs 1 and 4).

2.2 It is explained in the description of the patent in suit that these amounts of sulphur are still too high for nickel catalysts to be used since nickel reacts with the sulphur compounds and will deactivate in the course of the time. Further, it is stated that the proposals in the prior art disclosed in documents D5 and D6 suffer from the disadvantages that they are only suitable either for light feeds which may be hydrogenated at temperatures below 200°C (document D5)

or for feeds having a restricted sulphur content (document D6) (paragraphs 6 to 8 of the patent).

Hence, the technical problem to be solved is stated to consist in the provision of a process for hydrogenation of sulphur containing feedstock having improved tolerance for sulphur in the feedstock, where deactivation of the catalyst is considerably retarded and which process can be operated within a widened temperature window with feedstock where the sulphur content may fluctuate (paragraphs 11 to 13).

According to a first embodiment claimed in the main and first auxiliary requests, it is proposed to solve this technical problem by a process where the feedstock is contacted first with a precious metal catalyst like platinum or palladium, then with metal oxide, where the metal may be - amongst others - calcium, barium, copper, molybdenum, zinc or iron, and finally with a nickel catalyst. This embodiment is identical with the subject-matter of the auxiliary request pending before the Opposition Division which was held allowable in the contested decision (points I and III above). The subject-matter claimed in the new main and first auxiliary requests differs there from only in that it comprises a second embodiment where the metal oxide is combined with the nickel catalyst and a third embodiment where the metal oxide is combined with the precious metal catalyst. The latter two embodiments are contained in both, the main request and the first auxiliary request.

- 2.3 Documents D5 and D6, are both concerned with a general technical problem similar to that of the patent in suit,

namely with a process for hydrogenation of sulphur containing feedstock in the presence of a catalyst system having reduced sensitivity to deactivation by sulphur (document D5, page 2, lines 3 to 8 and page 3, lines 3 to 6; document D6, column 1, lines 36 to 39).

- 2.3.1 Document D5 suggests solving that technical problem by contacting the feedstock with a platinum group metal prior to or simultaneously with a nickel catalyst (Claim 1).

In the examples, it is shown that the process is suitable for feedstock having a sulphur content below 300 ppm, such as 20 ppm and, contrary to what is said in the patent in suit, for temperatures of 50 to 350°C, hence also for temperatures above 200°C (Claim 1; page 8, lines 24 to 30; page 10, line 15 and line 35 to page 11, line 2; figures 1 and 2).

- 2.3.2 Document D6 suggests solving the same technical problem by contacting the feedstock with a catalyst comprising on a support material a mixture of separate particles of nickel, platinum or palladium as a hydrogenation component and of a metal oxide like molybdenum or iron oxide which reacts with the sulphur atoms released during hydrogenation under formation of the corresponding sulphide (Claim 1 and column 5, lines 11 to 23).

This process is also useful for temperatures above 200°C (column 7, line 16 to column 8, line 12; column 8, lines 29 to 36; column 9, lines 10 to 15 and figures) and feedstock having a sulphur content of less than 50 ppm (column 6, lines 2 to 15 and Example I).

- 2.4 Consequently, documents D5 and D6 are equally suitable as a starting point for the assessment of inventive step.

The third embodiment of Claim 1 of the main and first auxiliary request differs from those processes in that feedstock is first contacted with the platinum group metal catalyst in combination with an adsorbent like molybdenum or iron oxide prior to contact with a separate nickel catalyst.

- 2.5 It is apparent from the above that the processes disclosed in documents D5 and D6 are suitable to be operated in the same temperature window as the claimed process (column 4, line 49 of the patent). The Appellant argued that the claimed process was not comparable in this respect with the prior art processes since the temperature window within which the process may be operated was dependent on the composition of the feedstock and the activity of the catalysts used. However, as both, the feedstock and the activity of the catalysts are not defined in the claimed process, no distinction can be made in this regard between the processes disclosed in the cited prior art and the claimed one.

Concerning the aspect of the technical problem stated in the patent which relates to the possibility that the sulphur content in the feedstock may fluctuate, no evidence whatsoever has been provided.

Hence, it is not credible that the claimed process provides advantages over the prior art with respect to

the temperature window within which it can be operated or with respect to feedstock having fluctuating sulphur contents. The latter was not contested by the Appellant.

- 2.6 The Appellant further argued that in view of the prior art disclosed in documents D5 and D6 the technical problem underlying the invention consisted in an improvement with respect to deactivation by sulphur of the catalyst used for hydrogenation of the feed. In his opinion, it was apparent from the experiments provided in the new evidence filed with the letter dated 16 December 2009 that this technical problem has been solved.

However, as these experiments are not to be admitted into the proceedings for the reasons set out above (point 1), this evidence cannot be considered by the Board.

- 2.7 The Board nevertheless finds credible that in view of the processes disclosed in documents D5 and D6, the claimed subject-matter results in a reduction of the deactivation of the nickel catalyst by sulphur in those instances where the sulphur present in the feedstock is not in the form of hydrogen sulphide but in the form of thiophenic sulphur or other types of organic sulphur.

The reason for this finding is that organic sulphur compounds like thiophenes are known to be not easily adsorbed on sulphur sorbents. Therefore, they have to be converted into absorbable hydrogen sulphide under suitable conditions by contact of the feed with hydrogen in the presence of a platinum or palladium catalyst prior to any contact with a sulphur sorbent

and prior to any reforming of the feed in order to prevent harm to the subsequent catalyst beds (document D7, column 4, lines 20 to 35 and 58 to 63). In particular, thiophenic sulphur has been found to have a much larger negative influence on nickel catalysts than mercaptans or hydrogen sulphide (document D5, page 2, lines 16 to 22).

2.8 It remains to be decided whether, in view of the available prior art documents, it was obvious for someone skilled in the art to solve the technical problem of reducing the deactivation of the nickel catalyst by organic sulphur such as thiophenic sulphur present in the feedstock by contacting the feedstock prior to contact with a nickel catalyst with a platinum group metal catalyst in combination with a metal oxide suitable as sulphur adsorbent.

2.8.1 Document D6 teaches that the resistance to deactivation by sulphur compounds of a hydrogenation catalyst like nickel, platinum and/or palladium catalyst can be improved if a sulphur binding metal oxide component is also present, for example the oxide of molybdenum or iron (Claim 1, Example I and column 4, lines 32 to 53).

While this document does not mention any particular process conditions in the case of thiophenes, document D7 explicitly teaches the necessity of converting thiophenes into hydrogen sulphide for better absorbability. For this purpose, document D7 proposes to convert in the presence of a platinum catalyst the thiophenes contained in the feedstock into hydrogen sulphide which is then adsorbed on the metal oxide sorbent, prior to any contact of the feed with the



reforming catalyst. Thereby, the latter is protected from being deactivated by the sulphur compounds (Claim 1, Examples VII to X and column 4, lines 55 to 63).

Hence, documents D6 and D7 both teach that the deactivation of the hydrogenation catalyst by sulphur can be improved if the catalyst is used together with or subsequent to a sulphur adsorbent whereby it is essential according to document D7 that the sulphur compounds are first converted into hydrogen sulphide.

The Board, therefore, concludes that at the priority date of the patent in suit a skilled person had ample reasons to expect a reduction of the deactivation of the nickel catalyst by using in the process of document D6 a platinum catalyst under conditions as taught in document D7 suitable to hydrogenate the organic sulphur compounds and absorb the products prior to the nickel catalyst.

- 2.8.2 Contrary to the Appellant's opinion, document D5 is not simply concerned with an improvement of the process of document D6 with respect to the resistance against deactivation by sulphur compounds of a hydrogenation catalyst like nickel, platinum or palladium catalyst but with the specific object of enhancing the amount of thiophenic sulphur in the feed that can be tolerated by the nickel catalyst before it has to be replaced (page 3, lines 3 to 6). This object is based on the finding that thiophenic sulphur has a much larger negative influence on the nickel catalyst than hydrogen sulphide. In order to meet this specific object, it is suggested in document D5 to contact the entire thiophenic sulphur

containing hydrocarbon feed together with hydrogen with a platinum catalyst prior to or simultaneously with contacting the feed with the nickel catalyst (page 2, line 16 to page 4, line 9).

Document D5 does not teach any removal of the sulphur compounds by absorption. It is even said that no further active components are necessary for increasing the resistance against deactivation (page 8, lines 3 to 8). However, since it is known from document D7 that organic sulphur like thiophenes should be converted into hydrogen sulphide for better absorbability (point 2.7 above), the Board concludes that a skilled person would not only consider hydrogenation of the thiophenic sulphur under suitable conditions but also absorption of the resulting hydrogen sulphide on a suitable sorbent before hydrogenation of the feedstock in the expectation of increasing the life-time of the nickel catalyst before it has to be replaced more than is achieved with the process of document D5 where no adsorbent is used (see also patent in suit, column 2, paragraph 12).

- 2.8.3 In the Appellant's view there was no incentive to combine precious metal and metal oxide in a first step, followed by nickel in a second step as claimed in the third embodiment of Claim 1 of the main and first auxiliary request. On the contrary, there was a prejudice against using metal oxide in the process of document D5 and there was no reason to combine the teaching of this document with technology known from the older documents D6 and D7. In addition, document D7 was not relevant here since it was not concerned with a process as claimed. Instead, the product obtained

according to the process disclosed in document D7 was comparable with the starting feed of the patent in suit.

The Board is not convinced by those arguments for the following reasons:

Firstly, document D6 explicitly teaches to combine the hydrogenation catalyst, be it nickel and/or platinum with the sulphur sorbing metal oxide on one support (Claim 1). It is explained that the resulting small mutual distance between the hydrogenation component and the metal oxide would allow the sulphur atoms once released from the sulphur containing compounds at the surface of the hydrogenation catalyst to migrate to and react with the neighbouring metal oxide (column 5, paragraph 23). Therefore, the combination of precious metal and metal oxide in a one step is already known in the prior art disclosed in document D6 and in the light of document D7 obvious to try in a first step (point 2.8.1).

Further, document D5 which has a priority date in 1995 explicitly refers to the technology disclosed in document D6 which has been published in 1990 and hence gives an incentive to consider those documents in combination. Apart from that, the difference in time between those documents cannot be considered to prevent a skilled person to combine the relevant teaching contained therein. The latter argument applies the more to document D7 which has a publication date in 1991.

According to document D5 (page 8, lines 3 to 6), it is not necessary to use any active components other than nickel and the platinum group metal in order to

increase the resistance against deactivation. However, in the Boards opinion, this teaching is to be read in conjunction with the statement on page 3 (lines 5 to 13) of document D5, where it is indicated that 'resistance of the catalyst' means specifically thiophenic sulphur resistance of a nickel hydrogenation catalyst. Hence, there is certainly no prejudice in document D5 against using in the first step, where the thiophenic sulphur is converted into hydrogen sulphide on the platinum metal catalyst (page 3, line 33 to page 4, line 9), a metal oxide suitable to adsorb the released hydrogen sulphide prior to any contact of the thus treated feed with the nickel catalyst.

As it is further known from document D7 that the absorption of the sulphur containing compounds should precede the catalytic reforming of the feed (column 4, lines 44 to 63), it is in the Boards opinion not true that the disclosure of document D7 is limited to products suitable as a starting feed in the claimed process. On the contrary, a skilled person would be motivated by the teaching of document D7 to carry out the reforming as a second step after conversion of the thiophenes and absorption of the released hydrogen sulphides.

- 2.9 Finally, the Appellant's argument that there was no incentive for the skilled person to combine the disclosure of documents D5 and D6 in order to arrive at the claimed subject-matter must also fail since, as pointed out above (2.7 and 2.8 above), document D7 actually suggests such a combination and gives reasons why to do so.

2.10 For these reasons, the Board finds that the subject-matter of Claim 1 of the Appellant's main and first auxiliary requests does not comply with the requirements of Articles 52(1) and 56 EPC.

## **Order**

**For these reasons it is decided that:**

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P.-P. Bracke