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Datasheet for the decision of 12 September 2014

Case Number: T 0227/11 - 3.3.06

Application Number: 96303368.3

Publication Number: 0743093

IPC: B01J27/26, C08G65/10

Language of the proceedings: EN

Title of invention:
Highly active double metal cyanide complex catalysts

Patent Proprietor:
BAYER ANTWERPEN N.V.

Opponents:
01) Shell Internationale Research Maatschappij B.V.
02) The Dow Chemical Company

Headword:
DMC Catalysts / Bayer

Relevant legal provisions:
EPC Art. 52(1), 56

Keyword:
Inventive step - (no) reformulation of the technical problem - obvious combination of features disclosed in one and the same document

Decisions cited:
T 0892/08

Catchword:

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Case Number: T 0227/11 - 3.3.06

DECISION
of Technical Board of Appeal 3.3.06
of 12 September 2014

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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted on 19 November**

2010 revoking European patent No. 0743093 pursuant to Article 101(3)(b) EPC.

Composition of the Board:

Chairman	B. Czech
Members:	G. Santavicca
	S. Fernández de Córdoba

Summary of Facts and Submissions

I. The appeal lies from the decision of the Opposition Division posted on 19 November 2010 revoking European patent No. 0 743 093.

II. Three oppositions had been filed on the grounds of Article 100(a), (b) and (c) EPC 1973. The evidence relied upon in the opposition proceedings includes the following items:

D1: JP 4-145123 A (English translation submitted by Opponent 01)

D8: JP 6-41293 A (English translation submitted by Opponent 02).

Later on, Opponent 03 withdrew its opposition (letter of 9 July 2004).

III. At the oral proceedings on 8 November 2004, the Opposition Division decided to revoke the patent (first decision, posted on 18 November 2004).

IV. This decision was appealed. In decision T 0069/05 of 24 May 2007, the Board entrusted with the appeal case found that the claims according to the then pending third auxiliary request (filed with letter of 29 April 2009) met the requirements of Article 123(2) EPC and were not objectionable under Article 100(b) EPC. Moreover the claimed subject-matter was found to be novel over the cited prior art, in particular D8. The case was thus remitted to the Opposition Division for further prosecution.

V. In the course of the continued opposition proceedings, with its letter dated 26 August 2010, the Appellant/ Patent Proprietor filed a further item of evidence, namely

ER2: Experimental Report by Dr. J. Hofmann entitled "Effect of Order of Addition of Reactant Solutions on Catalyst Activity",

as well as an amended Claim request, comprising a further restricted Claim 1 reading as follows (amendments made to previously pending Claim 1 made apparent by the Board):

"*1. A method of making a double metal cyanide (DMC) complex catalyst which method comprises reacting an aqueous solution of a metal salt and an aqueous solution of a metal cyanide salt, wherein ~~one or both of the reactant solutions~~* ***the metal salt solution*** *contains an organic complexing agent which is a water-soluble aliphatic alcohol selected from the group consisting of ethanol, isopropyl alcohol, n-butyl alcohol, isobutyl alcohol, sec-butyl alcohol, and tert-butyl alcohol, and wherein the metal cyanide salt solution is added to the metal salt solution.*"

VI. In its second decision, i.e. the decision now under appeal, the Opposition Division came *inter alia* to the following conclusions regarding said Claim 1:

a) D8 described the closest prior art.
b) The claimed method differed from the method illustrated in Working Example 1 of D8 in that other organic complexing agents were to be used instead of ethylene glycol dimethyl ether (**glyme** hereinafter).
c) The effects invoked by the Patent Proprietor were improved activity of the catalyst and low

viscosity of the obtained polyols, as shown in ER2. The invoked improvement of the viscosity of the polyols could, however, not be taken into consideration, since it was neither derivable from the application as filed nor had an improvement in this respect been shown in comparison to the method of D8. Moreover,it had not been shown that any improvement in terms of catalyst activity resulted from the use of alcohols other than t-butyl alcohol (**TBA** hereinafter).

d) Thus, the problem actually solved across the whole breadth of Claim 1 was merely the provision of an alternative method for making double metal cyanide (**DMC** hereinafter) catalysts.

e) Since D8 itself taught that TBA was a suitable alternative ligand, the replacement of glyme with TBA in a method according to working Example 1 of D8 was obvious in the light of D8 taken alone, and even more so in view of a combination of D8 with D1.

VII. With its statement setting out the grounds of appeal dated 29 March 2011, the Appellant (Patent Proprietor) filed an amended set of Claims 1 and 2 as its new sole claim request, as well as the following additional items of evidence:

ER3: Experimental Report by Dr. J. Hofmann dated 3 March 2011;

D11: WO 2009/080340 A1; and,

D12: US 3,867,312 A.

Compared to Claim 1 decided upon by the Opposition Division (Point V, *supra*), Claim 1 according to the newly filed claim request is more restricted in terms of the complexing agent to be used, and reads as follows (emphasis added):

"*1. A method of making a double metal cyanide (DMC) complex catalyst ...* ***contains an organic complexing agent which is <u>tert-butyl alcohol</u>****, and wherein the metal cyanide salt solution is added to the metal salt solution.*"

The Appellant held that taking into account all the experimental data on file, including also those presented in

ER1: Experimental Report filed by the Patent Proprietor during substantive examination with letter dated 8 September 2004,

the claimed method was not obvious in the light of D1 and/or D8. In this connection it also referred to documents D11 and D12.

VIII. In its reply, Respondent 1 (Opponent 01) submitted that said new Claim 1 was objectionable under Article 123(2) EPC and that the claimed subject-matter did not involve an inventive step starting from either D8 or D1 as the closest prior art. In this connection it also referred to a further document (D13: US 2005/0101477 A1).

IX. The parties were summoned to oral proceedings.

X. By letter dated 5 March 2014, Respondent 2 (Opponent 02) informed the Board that it did not intend to attend the oral proceedings.

XI. In its letter dated 8 May 2014, Respondent 1 announced that it would not attend the oral proceedings and that it withdrew its request for oral proceedings.

XII. In its response dated 5 August 2014, the Appellant too withdrew its request for oral proceedings and announced that it would not attend the oral proceedings. With said response it filed a new set of Claims 1 and 2 replacing the claims previously on file, together with adapted description pages 1 to 11.

XIII. Oral proceedings were held as scheduled on 12 September 2014, in the (announced) absence of all parties.

XIV. The Appellant (Patent Proprietor) requested in writing that the decision under appeal be set aside and that the patent be maintained on the basis of Claims 1 and 2 and pages 1 to 11 of the description filed with letter dated 5 August 2014, and drawing sheets 1/5 to 5/5 of the patent as granted.

Respondent 1 (Opponent 1) requested in writing that the appeal be dismissed.

Respondent 2 (Opponent 2) did not file a request in these appeal proceedings.

XV. The arguments of the Appellant of relevance here, concerning the issue of inventive step, can be summarised as follows:

a) Unlike the patent in suit, D8 did not address the problem of improving catalyst activity and did not report any improved catalyst activity. Thus D8 was not the closest prior art for assessing inventive step. D1 was more appropriate, as it sought to address the problem that conventional DMC catalysts not always had satisfactory activity.

b) The following could be gathered from the experimental data in the patent in suit, or supplementarily filed in reports ER1, ER2 and ER3:
   i) An improvement in catalyst activity (reduced propoxylation time) was obtained when TBA was the complexing agent and the metal cyanide salt solution was added to the metal solution, more particularly when TBA was present in the metal salt solution or in both solutions.
   ii) Instead, a less active catalyst was obtained when e.g. glyme was used as the complexing agent, in the same way.
   iii) Also, when glyme was the complexing agent, the catalyst activity was not significantly affected by the order of mixing of the solutions.
   iv) Moreover, a polyether polyol having reduced viscosity was obtained when the metal cyanide salt solution was added to the metal salt solution, or when TBA was present in the metal salt solution.
   v) Instead, when glyme was used as complexing agent, the viscosity of the polyether polyol was not significantly affected, irrespective of whether glyme was contained in the metal salt or in the metal cyanide salt solution.

c) The technical problem solved in the light of any of D1 or D8 taken as the closest prior art was the provision of a method for preparing a DMC complex catalyst improved in terms of its catalytic activity when used in the synthesis of polyether polyols and in terms of the lower polyether polyol viscosities so-obtained.

d) Starting from D1 as the closest prior art, the skilled person had no motivation to pre-mix TBA as complexing agent with the metal salt solution and to then add the metal cyanide salt solution to this pre-mix. None of the citations on file pointed to such an approach. Thus, there was no reasonable expectation that by so proceeding a DMC complex catalyst with improved catalytic activity would be obtained.

e) Likewise, starting from D8, there was no specific suggestion that TBA could replace glyme in Working Example 1, let alone that a more active catalyst could be obtained by making this replacement.

f) As regards the combination of D8 with D1, it was to be noted that the latter did not disclose how the use of TBA as the complexing agent affected the activity of the catalyst in the synthesis of polyether polyols. Hence, D1 contained no suggestion that a more active catalyst may be obtained using TBA in place of glyme. D1 (page 4, lines 15-20 and page 14, last paragraph) only taught that the use of TBA gave an improved catalyst life compared with previously known catalysts.

g) Therefore, the claimed method was not obvious.

XVI. The arguments of the Respondents in this respect can be summarised as follows:

a) Like the patent in suit, D8 pertained to the technical field of preparing a polyether compound and a composite metal cyanide complex catalyst therefor. The method disclosed in D8 had most features in common with Claim 1 of the Main Request. D8 disclosed all the features of Claim 1, albeit not in the defined combination, and was the closest prior art for assessing inventive step.

b) In light of the Appellant's claim that the catalysts prepared with TBA as the organic complexing agent had higher activity compared to those prepared with glyme as the organic complexing agent, the technical problem could be seen in the provision of a process for producing DMC catalysts of higher activity.

c) However, the examples of the patent in suit and the experiments illustrated in ER2 and ER3 all comprised the features of homogenizing the mixture while mixing the two solutions and the subsequent treatment of the formed complex with aqueous solution(s) of TBA and homogenization. The comparative experiments described in ER1 comprised no homogenization steps but different orders of addition, and the method of measuring catalyst activity being different, the results reported were not comparable with those used in any other examples. Thus, all these examples showed that the steps of homogenization and/or subsequent addition of the organic ligand TBA in water were necessary in order to achieve the invoked technical effects.

d) Claim 1 at issue did not, however, require homogenization and post-treatment steps of said type. Hence, Claim 1 encompassed embodiments for which it had not been convincingly shown that the desired effects were actually achieved. The stated technical problem was thus not solved across the full scope of Claim 1.

e) As regards the argument relating to the alleged improvement in polyol viscosity, no such improvement was derivable from the application as filed, nor was any such effect proven in comparison to D8. Hence, this argument was to be disregarded.

f) The skilled person starting from Working Example 1 of D8, and looking for a method to provide more active DMC catalysts, would obviously consider the particularly preferred alternative TBA mentioned in its Paragraph [0013] therefor. The claimed method was thus obvious in the light of document D8 taken alone.

Reasons for the Decision

Admissibility of newly cited documents D11 and D12

1. Documents D11 and D12 were filed by the Appellant with its statement setting out the grounds of appeal.

1.1 The late filing of D11 and D12 was not objected to by the Respondents.

1.2 D11 and D12 were filed in reaction to the reasons in the decision under appeal, in which it was held that the skilled person would understand catalyst activity to be correlated to catalyst life. They were thus filed to further corroborate the Appellant's argumentation.

1.3 Hence, the board decided to admit D11 and D12 into the proceedings despite their late filing (Article 114(2) EPC and Article 12(4) RPBA).

Admissibility of the Main Request at issue

2. The amended claims and description pages according to the main request at issue were filed after the issuance of the summons to oral proceedings.

2.1 The claims according to the main request at issue differ from the ones according to the request filed with the statement of grounds only in that in claim 2 a clerical error in the backreference (namely to Claim 2 itself) is rectified.

2.2 Compared to Claim 1 decided upon by the Opposition Division (Point V, *supra*), claim 1 at issue (Point VII, supra) is further limited in that the water-soluble alcohol to be used as complexing agent is TBA, the most preferred complexing agent (see Claim 5 and the Examples of the patent as granted).

2.3 The Respondents did not object to the late filing of said amended claims. For the Board, the filing of these further limited claims constitutes a straightforward reaction to the reasons given in the decision under appeal concerning the lack of inventive step as regards methods not making use of TBA.

2.4 Therefore, the Board decided to admit the new main request at issue despite its late filing (Article 114(2) EPC and 13(3) RPBA).

Allowability of the amendments

3. Since the appellant did not convince the Board that the method of Claim 1 at issue involves an inventive step (*infra*), the objections under Article 123(2) EPC raised by Respondent 1 regarding the allowability of Claim 1 at issue need not be dealt with.

Inventive step - Claim 1

The invention

4. The present invention concerns a method for preparing double metal cyanide (DMC) complex catalysts (see paragraph [0001] and Claim 1).

According to the patent in suit (Paragraph [0001]), the catalysts prepared by the method of the invention are highly active in epoxide polymerisation. More particularly, the method according to the invention is supposed to provide catalysts of improved activity (paragraph [0007]). In this connection, the patent in suit addresses increased polymerisation rate and shortened induction time (Paragraphs [0008], [0031] and [0032], Example 1) achievable in comparison to prior art catalysts. Moreover, the catalysts obtained by the method according to the invention are supposed to permit the synthesis of high molecular weight polyols having relatively low unsaturation (Paragraphs [0001] and [0009]) and low levels of impurities (Paragraph [0010]), whilst being easy to remove from the polyether polyol products (Paragraph [0011]).

The closest prior art

5. In the decision under appeal, the method disclosed in

D8 was held to represent the closest prior art for assessing inventive step. In its statement setting out the grounds of appeal, the Appellant, however, argued that D1 rather than D8 was to be considered as the closest prior art. Hence, it has to be established which of D8 and D1 represents the closest prior art, i.e. the most appropriate starting point for assessing inventive step.

5.1 As regards the objectives and problems respectively addressed in the prior art and the patent in suit:

5.1.1 D8 (see translation), like the patent in suit, concerns a method for producing a polyether compound and a composite metal cyanide complex catalyst (Paragraph [0001]). It addresses the problem of producing metal cyanide complex catalysts for the synthesis of high molecular weight polyether compounds (Paragraph [0005]) suitable e.g. as polyurethane starting materials (Paragraph [0033]). The catalysts so prepared should be easier to remove from the polyether products to be formed than the homogenous catalysts used previously, and should thus be suited for repeated usage (Paragraphs [0006], [0007] and [0050]. Providing catalysts with a high activity is also an issue addressed in D8. In this respect, reference may be made to Working Examples 1 to 3: Tables 1 to 3 report the "PO reaction efficiency %", which, as apparent from Paragraph [0037] of D8, is the conversion rate of fresh and re-used catalysts. Moreover, Paragraph [0020] describes how "to further enhance activity of the catalyst" and Paragraph [0021] describes how to "avoid adversely affecting the catalyst activity".

5.1.2 D1 (Page 2, "*Scope of the Patent Claims*" and "*Industrial field of application*,) also, like the patent in suit, relates to the preparation of DMC catalysts suitable for preparing polyether compounds which are suitable as raw materials for polyurethanes. It addresses the difficulties (pages 3 - 4; "*Means of Resolving These Problems*") encountered when using known catalysts which do not lead to high-molecular-weight polyethers with narrow molecular weight distributions with satisfactory selectivity, and addresses the issue of achieving satisfactory catalyst activity, in particular a "long catalyst life". Although D1 generally refers to "high activity and long life" of the catalysts disclosed (page 14, "Effect of the Invention"), only catalyst life time and selectivity are actually addressed expressly in the examples thereof (Tables I and II).

5.1.3 For the Board, it is apparent from the foregoing analysis that D8 addresses in more detail the main issue addressed in the patent in suit than D1, namely the issue of catalyst activity in the sense of conversion rate rather than catalyst life time. The Board accepts that catalyst life and catalyst activity are not necessarily correlated, as apparent, for instance, from the passages of D11 and D12 invoked by the Appellant in this respect.

5.2 As regards the similarity in terms of features of the methods according to the prior art and Claim 1 at issue, respectively:

5.2.1 The closest embodiment of D8 is the method illustrated in Working Example 1 (Paragraphs [0035] to [0040], according to which saponite powder was dispersed in a mixed solvent comprising water and glyme, and an aqueous solution containing zinc chloride was added thereto. Thereto, an aqueous solution containing cobalt potassium cyanate was then added dropwise over 30 minutes with agitation. The solution temperature was kept at 40°C. Solids were obtained through centrifuging after one hour of agitation. The solids were washed with an aqueous solution of glyme and centrifuged. The solids were again washed but glyme and centrifuged. The solids were dried and ground to yield a catalyst.

5.2.2 The closest embodiment of D1 is a method illustrated in (Reference) Example 1 (Page 10), wherein an aqueous solution of zinc chloride, an aqueous solution of potassium cobalt cyanate and an aqueous solution of tertiary butanol, i.e. TBA, were mixed at room temperature and reacted under agitation to produce a slurry. Filtration gave a white product. This filter cake was washed with an aqueous solution of TBA and filtered again. The filter cake so-obtained was further washed with TBA and filtered. The filter cake was dried and pulverized to give a bimetallic cyanide complex.

5.2.3 The method of Claim 1 at issue thus:

(a) differs from the method illustrated in Working Example 1 of D8 only in that TBA is used instead of glyme as organic complexing agent in the metal salt solution; whereas it

(b) differs from the method illustrated in Example 1 of D1 not only in that the metal salt solution must contain TBA, but also in that the metal cyanide solution is added to the metal solution (in D1/Example 1 no particular order is indicated with regard to the mixing of the two solutions).

5.2.4 The method disclosed in D8/Example 1 thus also has more features in common with the method of Claim 1 at issue than the method disclosed in D1.

5.3 For the Board, following the foregoing analysis, D8/Example 1, rather than D1, is the most appropriate starting point for assessing inventive step.

The technical problem according to the Appellant

6. According to the appellant, the problem to be solved in the light of the closest prior art D8 was the provision of a method for preparing DMC catalysts to be used in the production of polyether polyols, which catalysts are improved in terms of their activity while still producing a polyether polyol having low viscosity.

The solution

7. As a solution to this technical problem, the patent in suit proposes the method for making a double metal cyanide (DMC) complex catalyst according to claim 1 at issue, which method is characterised in that it comprises (emphasis added) "*reacting an aqueous solution of a metal salt and an aqueous solution of a metal cyanide salt, wherein the* ***metal salt solution contains organic complexing agent*** *which is* ***tert-butyl alcohol****, and wherein the* ***metal cyanide salt solution is added to the metal salt solution***".

The alleged success of the solution

8. The Appellant held that the experimental results presented in Examples 3 and 4 (Examples 13 and 14 of the application as filed) of the patent in suit and in the experimental reports ER1, ER2 and ER3 proved that the stated technical problem was solved by methods according to claim 1 at issue.

8.1 It can be gathered from Example 3 (in accordance with Claim 1 at issue) of the patent in suit that a catalyst providing a higher rate of polymerization rate is obtained by incorporating TBA in the metal salt solution and by adding the metal cyanide salt solution to the former, compared to a method wherein TBA is included in the metal cyanide salt solution, and wherein the metal salt solution is added to the metal cyanide solution (i.e. reverse order of addition) as described in Example 4 (comparative).

8.2 Moreover, it can be gathered from ER2 and ER3 that the use of TBA instead of glyme leads to a zinc hexacyanocobaltate TBA complex catalyst of higher activity under the conditions illustrated in these examples. In particular, in ER2, the propoxylation time (31 minutes) needed when using catalyst B (zinc hexacyanocobaltate TBA) was substantially shorter than the propoxylation time (86 minutes) needed when using Catalyst E (zinc hexacyanocobaltate glyme), despite the much higher concentration of the latter (200 ppm vs 25 ppm). Furthermore, it is apparent from ER3 that catalyst M (zinc hexacyanocobaltate TBA; prepared using TBA in both solutions) was even more active (propoxylation time of 19 minutes) than catalyst B.

8.3 However, for the Board, this proof that the claimed solution is not made up of arbitrary features does not necessarily prove that compared to the method of D8/ Example 1 an improvement in terms of catalyst activity is effectively achieved across the whole breadth of Claim 1, in particular considering that the teaching of D8 is neither acknowledged in the application as filed nor in the patent in suit.

8.3.1 In particular, whilst the Examples of the patent in suit and test reports ER1 to ER3 concern a method wherein the specific double metal cyanide catalyst (zinc hexacyanocobaltate) complex (with TBA or glyme or IPA) is made under homogenization (during and after the synthesis) and/or repeated treatments with the organic complexing agent (after the synthesis), Claim 1 at issue does not further define such process conditions. In fact, Claim 1 encompasses the preparation of double metal cyanide catalyst complexes synthesized without homogenization during and/or after synthesis, and/or without treatment with the complexing agent after the synthesis.

8.3.2 The Board, however, notes that the catalyst made according to Working Example 1 of D8 is, after its synthesis, subject to post-treatments comprising contacting it with the organic complexing agent used, i.e. glyme, as described in Paragraph [0036] of D8. As indicated in paragraph [0020] of D8, said post-treatments bring about an enhancement of the activity of the catalyst.

8.3.3 It is thus not possible to safely conclude from the experimental data on file that any DMC catalyst prepared according to a method comprising only the steps expressly prescribed by in Claim 1 at issue will necessarily have an increased activity in epoxide polymerisation compared to a catalyst prepared according to Example 1 of D8. Consequently, the Board is not convinced that all preparation methods falling within the terms of claim 1 (across its full breadth), will necessarily provide catalysts having an improved activity compared to the ones prepared as described in D8/Example 1.

8.4 The application as filed neither mentions (literally or implicitly) nor foreshadows or implies that less viscous (polyether) polyols would advantageously be obtainable upon use of the catalysts prepared according to the method of Claim 1 at issue. Under these circumstances, in accordance with established case law, this alleged improvement is not to be taken into account in the formulation of the technical problem (see e.g. "Case Law of the Boards of Appeal of the EPO", 7th edition, 2013, I.D.4.4.1).

Reformulated technical problem

9. Hence, the technical problem has to be reformulated in a less ambitious manner. Starting from the method described in Example 1 of D8, as a further method for making highly active double metal cyanide complex catalysts suitable for the production of polyether polyol.

The success of the claimed solution

10. Taking into account the experimental data on file (Points 8.1. and 8.2 *supra*), the Board is satisfied that this less ambitious problem is effectively solved across the whole scope of Claim 1 according to the Main Request. This was not disputed by the Respondents.

Obviousness

11. It remains to decide whether the claimed solution was obvious in view of the state of the art.

11.1 Document D8

11.1.1 According to D8 (paragraph [0013]), glyme and TBA are two of the four most preferred organic ligands to be used in the preparation of the DMC catalysts. Although the use of TBA is not exemplified in D8, let alone in connection with a method as described in Working Example 1 thereof, TBA and glyme are nonetheless presented as equally preferred organic complexing agents.

11.1.2 Thus, in the light of D8 taken alone, the use of TBA instead of glyme, presented in D8 as a suitable alternative organic ligand, is one of the few options readily available to the skilled person seeking to solve the less ambitious technical problem of providing a further method for making highly active DMC catalysts complexes. According to case law, choosing one of these options is devoid of any inventive character (see. e.g T 0892/08 of 15 September 2010, Reasons 1.7).

11.1.3 By implementing that option in the context of the preparation method described in D8/Example 1, e.g. by replacing at least to some extent glyme with TBA as the organic complexing agent, the skilled person would come to a method as claimed without any ingenious activity.

11.1.4 Hence, in the Board's judgement, the method according to claim 1 at issue does not involve an inventive step (Articles 52(1) and 56 EPC) in the light of the whole teaching of document D8 taken alone.

Conclusion

12. Therefore, the Appellant's sole request is not allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

Beschwerdekammern des Europäischen Patentamts
Boards of Appeal of the European Patent Office
Chambres de recours de l'Office européen des brevets

D. Magliano

B. Czech

Decision electronically authenticated