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Datasheet for the decision
of 30 November 2018

Case Number: T 1848/13 - 3.3.02

Application Number: 09173619.9

Publication Number: 2182001

IPC: C07F15/00

Language of the proceedings: EN

Title of invention:
Method for producing ruthenium complex

Patent Proprietor:
Takasago International Corporation

Opponent:
Heraeus Precious Metals GmbH & Co. KG

Headword:

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
Novelty
Inventive step

This datasheet is not part of the Decision.
It can be changed at any time and without notice.

Decisions cited:

Catchword:

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Beschwerdekammern

Boards of Appeal

Chambres de recours

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Case Number: T 1848/13 - 3.3.02

DECISION
of Technical Board of Appeal 3.3.02
of 30 November 2018

Appellant: (Patent Proprietor)	Takasago International Corporation Nissay Aroma Square 17F 5-37-1, Kamata Ohta-ku Tokyo 144-8721 (JP)
Representative:	Beckmann, Claus Kraus & Weisert Patentanwälte PartGmbB Thomas-Wimmer-Ring 15 80539 München (DE)
Respondent: (Opponent)	Heraeus Precious Metals GmbH & Co. KG Heraeusstrasse 12-14 63450 Hanau (DE)
Representative:	Bendele, Tanja RUHR-IP Patentanwälte Brucker Holt 58 45133 Essen (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 1 July 2013 revoking European patent No. 2182001 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	M. O. Müller
Members:	A. Lenzen
	M. Blasi

Summary of Facts and Submissions

I. This decision concerns the appeal filed by the proprietor of European patent No. 2 182 001 against the decision of the opposition division to revoke the patent.

II. In the notice of opposition, the opponent had requested revocation of the patent in its entirety on the grounds that the claimed subject-matter was neither novel nor inventive (Article 100(a) EPC) and that the patent did not disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Article 100(b) EPC).

The documents submitted during the opposition proceedings included:

D1 J. W. Hull, W. L. Gladfelter: "η^4-Bonding in (Arene)ruthenium Complexes of Octamethyl naphthalene", Organometallics, 1984, 3, pages 605-613,

D10 Hünig, Kreitmeier, Märkl, Sauer: "Arbeitsmethoden in der organischen Chemie (mit Einführungspraktikum)", 2006, pages 232, 233, 239 and 250,

D21 Comparative Example (2),

D27 Further additional experiment,

D30 "Laborautoklav bis 3 bar", 2013, and

D31 A. Wolf, "Laborautoklav", 2002.

III. The decision of the opposition division was based on sets of claims of a main request filed with letter dated 26 October 2012 and of first and second auxiliary requests filed with letter dated 11 April 2013.

In as much as it concerns the requests before the board, the decision of the opposition division can be summarised as follows: the subject-matter of the claims of both the first and second auxiliary requests (identical to the sets of claims of the main request and the auxiliary request before the board, respectively) was not inventive in view of the closest prior art D1.

IV. This decision was appealed by the patent proprietor ("appellant" hereinafter).

V. During the appeal proceedings, the opponent ("respondent" hereinafter) filed

D35: "Taschenbuch der Physik", 16th edition, 1999, pages 262-267, and

D38: print-out of the internet page http://www.zirbus.de/autoklaven/hochdruckautoklaven/.

VI. On 30 November 2018, oral proceedings were held before the board.

VII. The appellant requested that the decision under appeal be set aside and that the patent be maintained in amended form based on one of the sets of claims of the "first auxiliary request" and "second auxiliary request" both filed with its letter dated 11 April 2013 (now main request and auxiliary request).

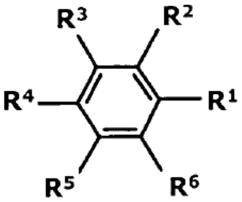
VIII. The respondent requested that the appeal be dismissed.

IX. Claim 1 of the main request reads as follows:

"A method for producing a ruthenium complex represented by the following general formula (3):

$[RuX_2(L^2)]_n$ ... (3)

wherein Ru represents a ruthenium atom, X represents a halogen atom, L^2 represents a benzene derivative represented by the following general formula (4),



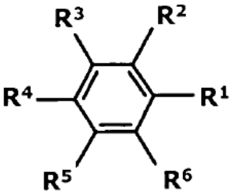
(4)

wherein R^1 to R^6 may be the same or different from each other and each represent a hydrogen atom, a saturated or unsaturated hydrocarbon group, an alkoxy group having 1 to 4 carbon atoms, a hydroxyalkyl group having 1 to 4 carbon atoms, or an alkoxycarbonyl group, and R^1 and R^2 may form together an alkylene chain which may have a substituent or an alkylenedioxy chain which may have a substitutent, and n is 2,

the method comprising heating and stirring, in a hermetically-sealed autoclave, a ruthenium complex represented by the following general formula (1):

$[RuX_2(L^1)]_n$ ... (1)

wherein Ru represents a ruthenium atom, X represents a halogen atom, L^1 represents a benzene derivative represented by the following general formula (4),



(4)

wherein R^1 to R^6 may be the same or different from each other and each represent a hydrogen atom, a saturated

or unsaturated hydrocarbon group, an alkoxy group having 1 to 4 carbon atoms, a hydroxyalkyl group having 1 to 4 carbon atoms, or an alkoxycarbonyl group, and R^1 and R^2 may form together an alkylene chain which may have a substituent or an alkylenedioxy chain which may have a substitutent, and n is 2 and
a benzene derivative represented by the following general formula (2):
L^2 ... (2)
as defined above whose boiling point is lower than that of L^1."

Compared with claim 1 of the main request, claim 1 of the auxiliary request contains the following additional features at the end of the claim: "wherein the reaction temperature is higher than the boiling point of the benzene derivative represented by L^2".

X. The appellant's arguments, in so far as they are relevant to the present decision, can be summarised as follows:

Main request

The claimed method was novel over the disclosure of D1. It differed therefrom in that it was carried out at a high overpressure in an autoclave, i.e. a reaction container suitable for high pressures, and in that this reaction container was hermetically sealed.

The claimed method furthermore involved an inventive step starting from the disclosure of D1 as the closest prior art. The objective technical problem was the provision of a method for producing a ruthenium complex as defined in claim 1 in a higher yield, and there was no suggestion in the prior art to use an autoclave at high pressures to solve this problem.

Auxiliary request

The claimed subject-matter was not only novel but also inventive in view of the disclosure of D1. According to claim 1 of this request, the reaction temperature was significantly higher than the boiling point of L^2, and thus a high overpressure was present during the reaction. This led to a significantly increased yield. For the reasons given already with regard to the main request, the claimed solution was not obvious.

XI. The respondent's arguments, in so far as they are relevant to the present decision, can be summarised as follows:

Main request

The claimed subject-matter lacked novelty over the disclosure of D1. A hermetically sealed autoclave as defined in claim 1 was identical to the Schlenk line used in D1. There was, in particular, no difference as regards the permissible pressure range or the feature of being "hermetically-sealed". Furthermore, both the autoclave as claimed and the Schlenk line of D1 resulted in an infinitesimally small overpressure during the reaction.

Furthermore, the claimed subject-matter was obvious. The appellant had not shown that a higher yield could be obtained over the entire scope claimed. The objective technical problem therefore had to be formulated less ambitiously as the provision of an alternative method for producing a ruthenium complex as defined in claim 1. In view of this problem, the replacement of the Schlenk line of D1 by a hermetically-sealed autoclave did not involve any inventive step.

Auxiliary request

Due to the fact that at least some of the ruthenium complex(es) was(were) dissolved, the reflux temperature in D1 had to be higher than the boiling point of the pure solvent alone. The claimed features "wherein the reaction temperature is higher than the boiling point of the benzene derivative represented by L^2" in claim 1 of the auxiliary request were thus implicitly met already by D1. Hence, for the same reason as given with regard to the main request, the subject-matter of the auxiliary request lacked at least inventive step.

The respondent also objected to both requests under Articles 123(2) and 84 EPC.

Reasons for the Decision

Main request

1. Novelty - Article 54 EPC

1.1 Claim 1 refers to a method for producing a ruthenium complex $[RuX_2(L^2)]_n$, the method comprising heating and stirring, in a hermetically-sealed autoclave, a ruthenium complex $[RuX_2(L^1)]_n$ and a benzene derivative L^2 whose boiling point is lower than that of L^1.

1.2 D1 discloses the conversion of $[RuCl_2(\text{p-cymene})]_2$ to $[RuCl_2(\text{mesitylene})]_2$ in refluxing mesitylene for 24 hours under stirring. The reaction was "carried out under an atmosphere of nitrogen by using normal Schlenk-line techniques" (page 606, first paragraph of the Experimental Section). Chlorine is a halogen atom "X" as required by claim 1. p-Cymene (1-methyl-4-isopropyl benzene) meets the structural definition given for L^1 in claim 1. Mesitylene (1,3,5-trimethyl benzene) corresponds to the structural definition given for L^2 in claim 1. The boiling point of mesitylene is 165 °C and thus lower than that of p-cymene, which is 177 °C. The requirement of claim 1 that the boiling point of L^2 be lower than that of L^1 is thus equally met.

Based thereon, only the remaining features of claim 1, (i) that the reaction is carried out in an autoclave (ii) that is "hermetically-sealed" and (iii) the pressure resulting therefrom, could possibly distinguish the claimed process from that of D1, where a Schlenk-line is used. This issue was the subject of the dispute between the parties.

1.3 The feature of claim 1 that the reaction is carried out in an autoclave

With regard to the term "autoclave", the respondent argued that the patent lacked any definition as to the pressure range at which the autoclave could suitably be operated. It therefore had to be construed as a container withstanding **any** overpressure, i.e. an overpressure which was undefined regarding its upper or lower limit. There was thus no difference between an autoclave as used in the claimed method and a Schlenk line as disclosed in D1 as regards the pressure at which these devices could be operated.

The board does not agree. It is common general knowledge that autoclaves are designed to withstand high pressures. For instance, documents D30, D31 and D38 depict at least one laboratory autoclave and give details as to its specifications, in particular with respect to the pressure range for which it is approved. The pressure ranges are up to 3 bar (D30), up to 3-4 bar (D31) and up to 20 bar (D38), i.e. pressures significantly higher than standard pressure.

The term "autoclave" can thus be circumscribed as a *reaction container suitable for carrying out reactions therein under high pressures*, i.e. under pressures which are significantly higher than standard pressure. Although this circumscription still leaves some ambiguity as to the limits of *high* pressure, in the board's view, as confirmed by the above documents, this ambiguity does not reach thus far that normal glass ware, such as the Schlenk-line used in D1, would be considered suitable for pressures for which autoclaves are made. The method of claim 1 thus differs from the one disclosed in D1 in that it is carried out in a reaction container that is suitable to be used at high pressure. This is a first distinguishing feature.

1.4 The feature of claim 1 that the autoclave is "hermetically-sealed"

This feature stipulates an autoclave which prevents any exchange of material between the autoclave and its surroundings during the entire reaction period. Contrary thereto, the Schlenk-line of D1 must allow any excessive gas phase, resulting from an expansion during the heating up period, to escape, otherwise the reaction apparatus might explode. It cannot therefore be hermetically-sealed. The fact that the reaction according to claim 1 is carried out in an autoclave that is hermetically sealed is thus a second distinguishing feature.

1.5 The pressure at which the reaction is carried out

1.5.1 Document D1 does not disclose whether some sort of pressure valve was attached to the Schlenk-line. Further, even if that had been the case, document D1 still lacked a disclosure as to its nature. For example, document D10 (page 232, first and second paragraphs under "10.3.3 Überdruckventile") refers to bubble counters as pressure relief valves and states that they allow excess pressure to be relieved from the apparatus, i.e. they allow the pressure to go back to ambient pressure. In view of this, it cannot be clearly and unambiguously derived from D1 that the reaction described therein had to have been conducted under a pressure higher than ambient pressure.

On the other hand, the autoclave is hermetically sealed according to claim 1. Heating of its content will therefore lead to at least an infinitesimally small overpressure inside of it. The presence of an infinitesimally small overpressure as compared with atmospheric pressure in D1 is thus a third distinguishing feature.

1.5.2 The appellant argued that a high rather than an infinitesimally small overpressure had to build up inside the autoclave due to the fact that it was hermetically sealed according to claim 1. The distinguishing feature was thus a high rather than an infinitesimally small overpressure. The board does not agree:

As shown by the respondent in its reply to the statement of grounds of appeal (letter dated 3 April 2014, points 2.4-2.4.1.6 on pages 15-17), and as reiterated by the respondent during the oral proceedings, the volume of the autoclave and the amount of the reaction material have a significant influence on the pressure inside the autoclave. For example, heating up 25 mL of mesitylene in an autoclave with a volume of V = 50 mL up to 166 °C results in an internal pressure of 1.5 bar. Reducing the amount of mesitylene to 12 mL lowers the internal pressure to 1.4 bar. Further reducing the amount of mesitylene would thus equally further reduce the internal pressure down to an infinitesimally small overpressure. This was not contested by the appellant.

Because claim 1 does not specify the volume of the autoclave or the amount of the reaction material inside of it, it cannot be accepted that all the methods of claim 1 result in a high overpressure inside the autoclave.

1.6 The appellant further argued that autoclaves were generally used under high pressure. As the method of claim 1 made use of an autoclave, the method implicitly had to be carried out under high pressure. For this reason too, the claimed method differed from that disclosed in document D1 in that it was carried out under a high rather than an infinitesimally small overpressure.

As set out above, the board acknowledges that the suitability of an autoclave for high pressures imparts properties which distinguish them from apparatuses made e.g. from normal glass ware, such as the Schlenk-line of D1. However, even though suitable for high pressures, autoclaves may also be used very well at much lower pressures, such as at infinitesimally small overpressure for instance. This situation is comparable to a racing car that, although undeniably suitable for being driven at very high speeds, can also be driven very well at lower speeds.

1.7 The method of claim 1 thus differs from that disclosed in document D1 in that it is carried out (i) at an infinitesimally small overpressure, (ii) in an autoclave, i.e. a reaction container suitable for high pressures, (iii) wherein this reaction container is a hermetically sealed reaction container. The method of claim 1, and by the same token that of dependent claims 2 and 3, is thus novel over the disclosure of D1.

2. Inventive step - Article 56 EPC

2.1 Both parties started from the disclosure of D1 as the closest prior art. The board sees no reason to depart from this unanimous opinion of the parties.

2.2 As set out above, the board considers the method of claim 1 to be distinguished from the disclosure of D1 in that it is carried out (i) at an infinitesimally small overpressure, (ii) in an autoclave, i.e. a reaction container suitable for high pressures, (iii) wherein this reaction container is a hermetically sealed reaction container.

2.3 The appellant argued that the effect obtained in view of D1 was an increased yield: whereas a yield of 96% was obtained in example 1 of the patent in suit, document D1 (example with $[RuCl_2(1,3,5\text{-}Me_3C_6H_3)]_2$ at the bottom of the left-hand column on page 606) reported a yield of only 92%. Moreover, when reworking this example of D1 with a paraffin valve connected to the Schlenk-line, the appellant found the product to contain only 52% of the actual target complex (i.e. $[RuCl_2(mesitylene)]_2$) amounting to an overall yield of only 36% (D21). The yield of 92% given in document D1 was erroneous and the actual yield much lower.

Example 1 in the patent in suit reacts the same compounds as those in document D1 "in a hermetically-sealed autoclave" but at an internal temperature of 200 °C. After workup using heptane at -20 °C, a yield of 96% was obtained. The appellant conducted an analogous experiment with a slightly modified workup procedure (hexane at 0 °C) and obtained the same yield (D27). Thus, conducting said reaction "in a hermetically-sealed autoclave" gives the product, irrespective of the workup procedure, in a slightly higher yield when compared with D1 (96% vs. 92%). According to the appellant, the internal temperature of 200 °C in these experiments, which is significantly higher than the reflux temperature of D1, caused the pressure inside the autoclave to be high.

Based on a comparison of example 1 in the patent in suit or D27 with D1 one might possibly acknowledge that at high overpressures, the technical effect linked to the distinguishing features above is a (slight) improvement of yield. Yet, as elaborated on above, not only reactions conducted under a high overpressure but also those conducted under only an infinitesimally small overpressure fall within the subject-matter of claim 1.

In the absence of any experimental data, e.g. in the form of comparative tests, showing that a reaction performed at the same temperature as in D1 and only an infinitesimally small overpressure gives a better yield, the effect of a higher yield cannot be acknowledged to occur over the whole breadth of the subject-matter of claim 1.

In view of the above, and a lack of comparative data close to the reaction conditions of D1, it is also irrelevant whether the product of D1 was pure (as one would assume based e.g. on D1 alone) or not (as argued by the appellant based on D21). Under the assumption that the product obtained in D1 was not pure, comparative data would still be missing showing that a reaction performed at the same temperature as in D1 and only infinitesimally small overpressure leads to improved purity and thus to a higher yield.

2.4 Based on the above, the objective technical problem has to be formulated in less ambitious terms, i.e. as an alternative method for producing a ruthenium complex represented by formula (3) in claim 1.

2.5 It remains to be examined whether the solution to the objective technical problem as defined in claim 1 is obvious.

When seeking to provide an alternative method, the skilled person would contemplate replacing the reaction apparatus disclosed in document D1 by other well-known reaction apparatuses. As set out by the board during the oral proceedings and not contested by the appellant, hermetically-sealed autoclaves belong to such well-known reaction apparatuses. By replacing the reaction apparatus disclosed in document D1 by such an autoclave, a slight overpressure would automatically result upon heating. The skilled person would thereby arrive at the subject-matter of claim 1 without the need to apply inventive skills. Thus, the subject-matter of claim 1 does not involve an inventive step.

Auxiliary request

3. Novelty and inventive step - Articles 54 and 56 EPC

Compared with claim 1 of the main request, claim 1 of the auxiliary request has been limited by the following features:

> "wherein the reaction temperature is higher than the boiling point of the benzene derivative represented by L^2".

In that regard, both parties agreed that

- "the boiling point of the benzene derivative represented by L^2" was the boiling temperature of this benzene derivative L^2 under standard conditions, and

- "the reaction temperature" was the temperature of the mixture comprising the ruthenium complex(es) and the solvent L^2.

As set out above (point 1.2), document D1 discloses refluxing $[RuCl_2(p\text{-cymene})]_2$ in refluxing mesitylene. Not disputed by the appellant, it is a law of nature that solutes, such as $[RuCl_2(p\text{-cymene})]_2$, lead to an increase of the boiling point of the solvent (i.e. mesitylene) containing it. This is evidenced, for instance, by document D35 (point 17.2.2). The reflux and thus reaction temperature in document D1 will therefore be higher than the boiling point of (pure) mesitylene under standard conditions. Hence, the features of claim 1 "wherein the reaction temperature is higher than the boiling point of the benzene derivative represented by L^2" are already met by document D1. The subject-matter of claim 1 of the auxiliary request is thus distinguished from the subject-matter disclosed in document D1 by the same features as the subject-matter of the main request.

The reasoning above with respect to the main request thus applies *mutatis mutandis*. Consequently, the subject-matter of claim 1 of the auxiliary request lacks inventive step in view of the disclosure of D1.

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

N. Maslin M. O. Müller

Decision electronically authenticated