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**Datasheet for the decision
of 22 April 2021**

Case Number: T 0288/16 - 3.3.10

Application Number: 08759665.6

Publication Number: 2155658

IPC: C07C209/78, C07C209/86,
C07C211/50, C07C263/10,
C07C265/14

Language of the proceedings: EN

Title of invention:

PROCESS FOR THE PREPARATION OF DI- AND POLYAMINES OF THE
DIPHENYLMETHANE SERIES

Patent Proprietor:

Huntsman International LLC

Opponents:

THE DOW CHEMICAL COMPANY
Covestro Deutschland AG
BASF SE

Headword:

PROCESS FOR THE PREPARATION OF DI- AND POLYAMINES/ Huntsmann

Relevant legal provisions:

EPC Art. 56
RPBA Art. 12(4)

Keyword:

Inventive step - (no)

Late-filed request - request identical to request not admitted
in first instance proceedings - admitted (no)

Decisions cited:

Catchword:



Beschwerdekammern

Boards of Appeal

Chambres de recours

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Case Number: T 0288/16 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 22 April 2021

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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted on 9 December 2015
revoking European patent No. 2155658 pursuant to
Article 101(3) (b) EPC.**

Composition of the Board:

Chairman P. Gryczka
Members: J.-C. Schmid
 T. Bokor

Summary of Facts and Submissions

- I. The appellant (Proprietor of the patent) lodged an appeal against the decision of the Opposition Division revoking European patent No. 2 155 658.
- II. Notice of opposition had been filed by the respondents (opponents 1, 2 and 3) requesting revocation of the patent-in-suit in its entirety on the grounds of lack of novelty and inventive step (Article 100(a) EPC), insufficient disclosure of the invention (Article 100(b) EPC) and extension of the subject-matter of the patent-in-suit beyond the content of the application as filed (Article 100(c) EPC).

Inter alia, document

(1) US-A-2006/0094897

was cited in the opposition and appeal proceedings.

According to the opposition division, the grounds for opposition under Article 100(b) and (c) EPC did not prejudice the maintenance of the patent as granted. The subject-matter of claim 1 of the patent as granted was novel over document (1) since the opponents had not demonstrated that the aqueous formaldehyde used in the process disclosed in document (1) contained less than 0.001 wt.% metal ions which were divalent and/or more than divalent.

With respect to inventive step, document (1) was the closest prior art to the invention. The process of claim 1 of the patent as granted differed from the process of document (1) in that the formaldehyde

employed was used as an aqueous solution which contains less than 0.001 wt.% of metal ions which are divalent and/or more than divalent.

The patent proprietor did not prove that an effect had been demonstrated by this difference over the whole scope of claim 1 because the claimed process covered two alternatives i.e. with or without intermediate separation. In addition it was shown in Annex I filed with letter dated 5 October 2015 that even with a formaldehyde with up to 50 ppm of metal ion content, there was no rag layer. Consequently, the limit of 10 ppm as required by the process of claim 1 could not be associated with an effect.

The problem to be solved was considered as the provision of an alternative process for the preparation of di- and polyamines of the diphenylmethane series, since there was no evidence that the threshold of less than 10 ppm of more than divalent or divalent metal ions prevented the formation of rag layer for all the processes claimed in claim 1 of the patent as granted.

Commercial formaldehyde had usually low metal ions content. Therefore claim 1 of the patent as granted related to the same process as in document (1), starting with a formaldehyde of ordinary quality with no associated effect. The subject-matter of claim 1 of the patent as granted therefore lacked an inventive step in the light of document (1).

Claim 1 of auxiliary request 1 differed from D1 in that the formaldehyde employed was used as an aqueous solution which contained less than 0.0003 wt.% of metal ions which were divalent and/or more than divalent.

Annex I filed by the patent proprietor with letter dated 5 October 2015 showed that by increasing the amount of iron ions up to 50 ppm in a process comprising an intermediate separation, only the formation of minor amounts of solid was observed. Since such an intermediate separation was covered by claim 1, the initial amount of ions in formaldehyde was irrelevant, with the consequence that the limit of 3 ppm was arbitrary.

In the process of example 6 at page 5 of the opposed patent using formaldehyde having 3.28 ppm of metal ions, i.e. slightly above the limit of 3 ppm, the formation of some solids was observed. It was therefore not credible that if HCl having 10 ppm of ions was used, no solids would have been formed. Therefore, the limit of 3 ppm in metal ion content in formaldehyde was not critical, since the process of claim 1 of auxiliary request 1 allowed the presence of 10 ppm metal ions coming from the hydrochloric acid solution.

The problem to be solved was the provision of an alternative process for the preparation of di- and polyamines of the diphenylmethane series and the solution was obvious in the light of document (1). Consequently, the subject-matter of claim 1 of auxiliary request 1 also lacked an inventive step.

The opposition division did not admit auxiliary requests 2 and 3 in the proceedings, since the opponents could not have been expected to be prepared to deal with these new requests filed during the oral proceedings. Furthermore these requests were *prima facie* not suitable for overcoming the inventive step objection.

III. During the oral proceedings held on 22 April 2021 before the Board, the Appellant requested the maintenance of the patent-in-suit on the basis of a main request and two auxiliary requests, all requests having been filed with the statement of the grounds of appeal.

Claim 1 of the main request is identical to claim 1 of auxiliary request I pending before the Opposition Division, and reads as follows:

"1. A process for the preparation of di- and polyamines of the diphenylmethane series comprising reacting aniline with formaldehyde in the presence of hydrochloric acid, the hydrochloric acid containing less than 0.001 wt% of metal ions which are divalent and/or more than divalent, characterised in that the formaldehyde employed is used as an aqueous solution which contains less than 0.0003 wt% of metal ions which are divalent and/or more than divalent."

Claim 1 of auxiliary requests 1 and 2 differ from claim 1 of the patent as granted and claim 1 of the main request, respectively, in that they comprise the feature of first mixing aniline with hydrochloric acid and then adding aqueous formaldehyde solution to the mixture.

IV. According to the Appellant, contrary to the findings of the opposition division, the limit of less than 3 ppm divalent metal ions in the aqueous formaldehyde was not arbitrary, as was shown in the examples of the patent in suit. Solids were formed when more than 3 ppm was used (see experiment 6, 7 and 8) and when more than 5 ppm was used, a rag layer was formed. Thus an effect

was associated with the limit of 3 ppm. The subject-matter of claim 1 involved therefore an inventive step.

In Annex I (filed again with the statement of grounds of appeal), formaldehyde having 50 ppm divalent metal ions was used. In this experiment, there was an intermediate separation step. Most of the metal ions were removed in the aqueous phase of the intermediate separation step, however, some solids were found in the interphase. Thus when using this method for making polyamines where there was an intermediate separation step, it was also required to use formaldehyde having less than 50 ppm metal ions. The use of an aqueous formaldehyde solution containing less than 3 ppm of metal ions was working for both alternative methods of making polyamine, so that the 3 ppm limit in the aqueous formaldehyde solution was not arbitrary.

The opposition division considered that the auxiliary requests were late filed and did not admit them into the proceedings. However, these requests were filed as a reaction to inventive step arguments which came up for the first time during the oral proceeding before the opposition division. In addition, the opposition division considered that these requests did not *prima facie* overcome the inventive step objections. However, during the whole procedure, and more in particular with respect to the arguments filed in connection with Annex I, it was made clear that there were several ways to carry out the claimed process for making MDA. The opposition division considered in its decision that the limit of 10 ppm could not be associated with an effect in view of the experiment reported in Annex I. Claim 1 of auxiliary request 1 restricted the number of possibilities to carry out the claimed process excluding that of Annex 1. This limitation was a

reaction to the decision of the opposition division concerning inventive step. Hence auxiliary requests 1 and 2 should be admitted in the appeal proceedings.

- V. According to the respondents, the subject-matter of claim 1 of the main request lacked novelty over document (1), since formaldehyde solutions containing less than less than 0.0003 wt. % of metal ions which are divalent and/or more than divalent, were common and even commercially available. Furthermore it lacked also an inventive step, since no effect was shown for the alternative requiring an intermediate separation step which was covered by claim 1. The examples of document (1) disclosed also a process requiring a separation step. Annex 1 filed by the appellant showed that no rag layer formed when formaldehyde with 50 ppm metal ions was used when a separation step occurred. Some solid material was formed on neutralisation. However, as the metal ions from the formaldehyde were removed at the intermediate separation stage, the solids resulted from the hydrochloric acid. Therefore, the technical effect argued to be associated with the limit of 3 ppm divalent metal ions in the formaldehyde solution was not present across the whole scope of claim 1. The problem to be solved was the provision of an alternative method. The proposed solution was obvious, since commercially available formaldehyde with a metal ion content of less than 3 ppm was standard in the art. It would have been obvious to the skilled person to use such a formaldehyde solution. Furthermore, document (1) described a negative effect resulting from using metal ions in the reactants. Therefore, the skilled person would be minded to keep the levels of metal ions to a minimum. Therefore, claim 1 of the main request was obvious over document (1).

In the statement of the grounds of appeal, the Appellant did not explain why the first instance discretionary decision not to admit auxiliary requests 1 and 2 was wrong. Hence, there was no reason to deviate from the decision of the opposition division not to admit these requests into the proceedings.

The appellant's argument that these auxiliary requests could not have been filed earlier as they were responsive to inventive step arguments that came up for the first time during the oral proceedings was not founded. The modification in claim 1 of these auxiliary requests was intended to deal with the argument that document (1) disclosed a process comprising a step where the aqueous phase was separated from the organic phase such that the level of metal ions in the organic phase did not form a rag layer. This line of argumentation was submitted by opponent 1 in its letter dated 30 September 2015, where it pointed out that a technical effect shown for a process with no intermediate phase separation could not be relied on across the whole scope of claim 1 of the main request. Annex I was submitted by the Appellant with letter of 5 October 2015. It showed that when following the teaching of document (1), it was possible to operate a process using 50 ppm divalent metal ions in the formaldehyde while still avoiding the formation of a rag layer. The reference to this Annex was the only new point of the Opponents at the oral proceedings before the opposition division. It provided only concrete evidence of an argument that the opponents had previously made. Therefore, the appellant could and indeed should have filed requests dealing with this issue earlier in the proceedings.

For these reasons, auxiliary requests 1 and 2 should not be admitted to the proceedings.

- VI. The appellant requested that the decision under appeal be set aside and the patent be maintained on the basis of the main request, or subsidiarily, on the basis of any of the first or second auxiliary requests, which were filed with letter dated 8 April 2016.
- VII. The respondents requested that the appeal be dismissed.
- VIII. At the end of the oral proceedings held on 22 April 2021 the decision of the Board was announced.

Reasons for the Decision

1. The appeal is admissible.

Main request: inventive step

2. Document (1) represents the closest prior to the invention. This document discloses a process for the preparation of di- and polyamines of the diphenylmethane series comprising reacting aniline with formaldehyde in the presence of hydrochloric acid, wherein the hydrochloric acid employed contains less than 0.001 wt. % of metal ions which are divalent and/or more than divalent (see claim 1; examples 1 and 2)). Preferably, the hydrochloric acid employed contains less than 0.0003 wt. % of metal ions which are divalent and/or more than divalent (see claim 3).

Document (1) teaches that the content of metal ions which are divalent and/or more than divalent should be

minimized in the aqueous hydrochloric acid in order to avoid the formation of a third phase (i.e. rag or a rag layer), which makes separation of the MDA-containing organic phase difficult - see paragraph [0029].

The general instructions to carry out the process are set forth in paragraph [0033]. An aqueous formaldehyde solution is added dropwise to aniline at 80° C over 20 minutes while stirring. After the addition was completed, the mixture is stirred for a further 5 minutes and a phase separation is carried out at 70 to 80° C. Then, aqueous hydrochloric acid is added to the organic phase (the amination), at 45° C over 20 minutes, then the reaction mixture is further heated and stirred in order to bring the reaction to completion. Document (1) is silent about the content of divalent or more than divalent metal ions in the aqueous formaldehyde solution used in the examples.

3. The respondents submitted that the subject-matter of claim 1 of the main request lacked novelty over this document, as it was common for formaldehyde solutions to contain less than 0.0003% by weight of metal ions which were divalent and/or more than divalent, and there were even commercially available solutions, for example the one used in the examples of the patent in suit. However, since the subject matter of claim 1 anyway lacks an inventive step, as set out below, it is not necessary for the Board to reach a decision on this point. Thus, it is assumed, in favour of the appellant, that the subject matter of claim 1 differs from the process of document (1) by the requirement that the aqueous formaldehyde contain less than 3 ppm of divalent and/or more than divalent metal salt.

4. The appellant defined the problem to be solved as how to minimize the formation of solids during the process.
5. The comparison on which the appellant relies to show an improvement relating to the formation of the solids are made with processes where aniline is first mixed to an aqueous hydrochloric acid and then the formaldehyde solution is added to this aniline/HCl solution (see paragraphs [0041] to [0044] on page 5 of the patent in suit).

However, according to paragraph [0025] of the patent, in addition to the embodiment of the process illustrated in the examples in which, the process comprises first mixing aniline with hydrochloric acid and then adding an aqueous formaldehyde solution to the mixture, it is also possible to mix aniline, aqueous formaldehyde solution and hydrochloric acid in a different order.

Thus, paragraph [0025] of the patent in suit describes another way to perform the claimed process, which follows the general procedure disclosed in document (1). This process comprises first mixing aniline and aqueous formaldehyde solution and reacting in the absence of the acidic catalyst at temperatures of preferably 60 °C to 95 °C, where a condensation products of aniline and formaldehyde called aminal form. After the aminal formation, the water contained in the aminal is at least partly removed. Then, the aminal is mixed with hydrochloric acid, preferably at temperatures of 20 to 60°C.

For this embodiment there is no comparative examples showing an improvement. On the contrary, Annex 1 filed by the Appellant with the letter dated 5 October 2015

shows that the process described in document (1) can easily be carried out using formaldehyde having about 50 ppm divalent metal ions, without having an effect on the formation of a rag layer.

The limit of 3 ppm of metal ions in the aqueous formaldehyde solution is therefore not critical when formaldehyde and aniline are first mixed, since an intermediate phase separation occurs afterwards, thus removing any metal salts present in the aqueous formaldehyde before the hydrochloric acid is added (see annex II of the statement of grounds of appeal, point 4 of the appellant's letter dated 5 October 2015).

The Board thus observes that the comparisons on which the appellant relies to show an improvement were not carried out with respect to the process disclosed in document (1) where aniline was first mixed with the aqueous formaldehyde. For the comparison with respect to a process made according to the general procedure described in document (1) no effect of the formation of solids was shown with a formaldehyde solution having less than 3 ppm divalent or more than divalent metal salts.

6. The technical problem should therefore be reformulated into the provision of an alternative process for the preparation of di- and polyamines of the diphenylmethane series.
7. It is not contested that formaldehyde solutions having less than 3 ppm divalent or more than divalent metal salts are commercially available.

The skilled person would therefore contemplate as an obvious possibility using such a solution of

formaldehyde having very low content of divalent metal salts and would therefore arrive at the claimed subject-matter without the exercise of inventive skill.

Hence, the subject-matter of claim 1 of the main request lacks an inventive step in view of document (1) alone.

Auxiliary requests 1 and 2

8. These auxiliary requests, filed with the statement of grounds of appeal, corresponds to requests which had already been submitted during oral proceedings in the opposition proceedings and which had not been admitted by the opposition division.

The opposition division considered that claim 1 of these requests did not result from the combination of granted claims, but incorporated a feature taken from the description of the patent in suit. Thus, in view of the nature of the amendments in the then pending auxiliary requests 2 and 3, the opposition division came to the conclusion that the substance of such requests could not be dealt with by the other parties at the oral proceedings. Furthermore the opposition division considered that the prima facie allowability of these requests was questionable.

Accordingly, the board finds that the opposition division's decision to not admit the then pending auxiliary requests 2 and is not exclusively based on considerations on the unjustified lateness of the filing of such requests but is also based on considerations relating to the substance of these requests.

Hence, the board is satisfied that the opposition division exercised its discretion to not admit auxiliary requests 2 and 3 in a reasonable manner and in accordance with the right principles.

According to the appellant, these auxiliary requests were filed in reaction to arguments put forward for the first time at the oral proceedings before the opposition division.

However, the appellant did not identify which new argument had been put forward during the oral proceedings before the opposition division. Hence, the Board cannot take this unsubstantiated argument into consideration.

Consequently, the Board does not consider it justified to overrule the finding of the opposition division and exercises its discretion under Article 12(4) RPBA 2007, Article 25(2) RPBA not to admit auxiliary requests 1 and 2 into the appeal proceedings.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



C. Rodríguez Rodríguez

P. Gryczka

Decision electronically authenticated