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Datasheet for the decision of 1 June 2021

Case Number: T 0089/17 - 3.3.10

Application Number: 06771056.6

Publication Number: 1890998

C07C263/10, C07C263/20, IPC:

C07C265/14, C08G18/76

Language of the proceedings: ΕN

Title of invention:

PROCESS FOR THE PREPARATION OF POLYISOCYANATES OF THE DIPHENYLMETHANE SERIES

Patent Proprietor:

HUNTSMAN INTERNATIONAL LLC

Opponent:

BASF SE

Headword:

PROCESS FOR THE PREPARATION OF POLYISOCYANATES OF THE DIPHENYLMETHANE SERIES /HUNTSMAN

Relevant legal provisions:

EPC Art. 100(b)

Keyword:

Grounds for opposition - insufficiency of disclosure (no)

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Catchword:



Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 0089/17 - 3.3.10

DECISION
of Technical Board of Appeal 3.3.10
of 1 June 2021

Appellant: HUNTSMAN INTERNATIONAL LLC

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Intellectual Property Department

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Respondent: BASF SE

(Opponent) Carl-Bosch-Strasse 38 67056 Ludwigshafen (DE)

Representative: Altmann, Andreas

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 21 November 2016 revoking European patent No. 1890998 pursuant to Articles 101(2) and 101(3)(b) EPC.

Composition of the Board:

Chair R. Pérez Carlón Members: J.-C. Schmid

F. Blumer

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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. 1 890 998, whose claim 1 reads as follows:
 - "1. A process for preparing mixtures comprising diphenylmethane diisocyanates and polyphenylpolymethylene polyisocyanates comprising the step of reacting the corresponding mixtures of diphenybnethanediamines [sic] and polyphenylpolymethylenepolyamines with phosgene in the presence of at least one solvent in stages whereby in a first stage the corresponding carbamoyl chlorides and amine hydrochlorides are formed and whereby in a subsequent stage residual carbamoyl chlorides are dissociated into the corresponding polyisocyanates and hydrogen chloride and amine hydrochlorides are phosgenated to form ultimately the corresponding polyisocyanates characterised in that a controlled amount of amine hydrochloride solids remain unreacted at the point where residual excess phosgene is removed from the reaction mixture, wherein the residual content of amine hydrochloride solids at the point where residual excess phosqene is removed from the reaction mixture is between 10 and 5000 ppm, and wherein said solid carbamoyl chlorides and/or solid ureas are not present at the point where residual excess phosgene is removed from the reaction mixture."
- II. Notice of opposition had been filed by the Respondent (Opponent) requesting revocation of the patent-in-suit in its entirety on the grounds of lack of novelty and

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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).

III. According to the Opposition Division, the grounds of opposition under Article 100(c) EPC did not prejudice to the maintenance of the patent as granted.

As regards to the ground of opposition pursuant to Article 100(b) EPC, the Opposition Division considered that claim 1 of the patent as granted covered three alternatives, two of which avoiding the formation of solid ureas completely. However, the skilled person was not able, using his common general knowledge alone, to provide a process which avoids the formation of solid ureas completely. The information in the patent that such a process required "careful design" of the mixing device, without further details, and without a single specific detailed example of a suitable mixing device, indicated that the design of such a mixing device was not a routine task which the skilled person would be able to perform without the use of inventive skill.

It was possible using analytical techniques to determine the amine hydrochloride content of a mixture of solid constituents, but not to make an instant determination of the amine hydrochloride solids, so that the skilled person did not known whether the amount of amine hydrochloride solids fell within the claimed range in order to remove the residual excess phosgene, as required by claim 1. Consequently, the skilled person could not perform the claimed process.

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The Opposition Division came therefore to the conclusion that the patent must be revoked for insufficiency of disclosure of the invention.

According to the Appellant, the invention related to a IV. process for making PMDI (polyphenylpolymethylene polyisocyanates) having improved colour. To achieve this goal, a controlled amount of solid amine hydrochlorides must be present at the point where the excess phosqene was removed from reaction mixture. Both ureas solids and carbamoyl chloride solids should not be present at that point. The process could not be operated in the presence of solid ureas and/or solid carbamoyl chlorides, as this would quickly clog the filters and/or the pipes. Solid ureas could be avoided by using appropriate mixing devices. This was known from the prior art. It was also known that carbamoyl solids could be eliminated by heating the reaction mixture. The amount of solids must be determined at the point of time at which residual excess phosgene was removed. This could be done by taking samples at that point, but samples could also be taken at later or earlier points. Since the process was continuous, the samples could be analysed within a sensible time-frame. The analysis of the solids could be done by isolating the solids from the sample. Via infra-red, it could be measured if compounds having urea groups were present. Via X-ray fluorescence (XRF), it could be measured the chlorine content to determine how much amine hydrochloride was present. In the event that amine hydrochloride solids were not in the required amount or urea and/or carbamoyl solids were present, the process conditions would be modified in a manner known to the skilled person until the correct solids content was present. The control of the amount of the solids in the reaction mixture could be done for instance as

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described in [0039] and [0050] of the patent by controlling the turbidity of the reaction mixture. By using appropriate reference solutions, the turbidity value could easily be converted to ppm values. Thus, the process of claim 1 was sufficiently disclosed to be operated by a skilled person.

V. According to the Respondent, claim 1 encompassed three embodiments depending on the composition of the solids present at the point where residual excess phosgene was removed from the reaction mixture, i.e. the solids contained (1) amine hydrochloride solids exclusively; (2) amine hydrochloride solids and carbamoyl solids, free of solid ureas; and (3) amine hydrochloride solids and solid ureas, free of carbamoyl solids. The skilled person was unable to carry out the claimed process where 0 ppm solid ureas was present (situations (1) and (2)), as well as where (huge) amount of solid ureas was present (situation (3)). This was even recognized by the Appellant. Furthermore, there was no indication in the patent in suit how to measure the residual content of the amine hydrochloride solids if mixed with other solids at the point where residual excess phosgene was removed from the reaction mixture, let alone if this measure was to be done instantly. Accordingly, the skilled person would not be able to determine the concentration of amine hydrochloride solids at that time without undue burden. Furthermore, based on Figure 1, paragraphs 37-42 and 50, of the patent in suit, the skilled person would understand that the amount or content of amine hydrochloride salt was to be determined with an in-line turbidity meter. However, carrying out a turbidity measurement to determine a relative amount in ppm would always be dependent on various parameters including the medium used for the measurements. There was no disclosure in the patent in

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suit how the relative amount in ppm of solid amine hydrochloride could be obtained from a turbidity measure.

Therefore, for these reasons, the requirements of Article 83 EPC were not met.

VI. The Appellant (Patent Proprietor) requested that the decision under appeal be set aside and that the case be remitted to the Opposition Division for further examination on the basis of the patent as granted or, alternatively, on the basis of any one of the first to eighth auxiliary requests, all auxiliary requests as filed with the statement setting out the grounds of appeal dated 21 March 2017.

The Respondent (Opponent) requested that the appeal be dismissed.

VII. At the end of the oral proceedings held on 1 June 2021, the decision of the Board was announced.

Reasons for the Decision

1. The appeal is admissible.

Main request: patent as granted

Sufficiency of disclosure of the invention Article 100(b) EPC

2. The patent relates to a method for preparing di- and polyisocyanates (PMDI). The invention aims at reducing the colour of PMDI and thus at obtaining them with a higher HunterLab colour (L). To achieve this object the method of claim 1 requires that between 10 and 5000 ppm amine hydrochloride solids remain in the reaction

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mixture at the point where residual excess phosgene is removed -see paragraphs [0021] and [0038] of the patent in suit.

Claim 1 also requires that solid carbamoyl chlorides and/or solid ureas are not present at the point where residual excess phosgene is removed from the reaction mixture.

- 3. According to the Respondent, this requirement expanses to three embodiments, i.e. the solids present at the point where residual excess phosgene is removed from the reaction mixture can be
 - 1) Amine hydrochloride solids exclusively;
 - 2) Amine hydrochloride solids and carbamoyl solids, free of solid ureas;
 - 3) Amine hydrochloride solids and solid ureas, free of carbamoyl solids.

Therefore, in order to fulfil the requirement of Article 83 EPC, the skilled person must be able to carry out the claimed process where 0 ppm solid ureas are present (situations 1 and 2), as well as where a large amount of solid ureas is present (situation 3).

According to the case law, the skilled person should try, with synthetical propensity, i.e. building up rather than tearing down, to arrive at an interpretation of the claim which is technically sensible and takes into account the background of the invention and the whole disclosure of the patent. The patent must be construed by a mind willing to understand, not a mind desirous of misunderstanding. That means that technically illogical interpretations should be excluded.

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In the present case, claim 1 requires that no solid carbamoyl chlorides and/or solid ureas should be present at said stage of the process. Having regard to the background of the invention, the formation of both of these two solids should generally be avoided in processes for preparing PMDI, although small quantities of solid ureas or carbamoyl chlorides may be tolerated. The Appellant also indicated that it was not possible to carry out the process in the presence of large quantities of solid ureas for the obvious reason that this would quickly block the pipes.

The Board first notes that the requirement of claim 1 of the patent as granted that no solid carbamoyl chlorides and/or solid ureas should be present does not characterise the invention, but places the invention in the context of prior art processes that seek to minimise or eliminate the formation of solids. Therefore, to interpret that claim 1 requires the process to be carried out with large quantities of solid ureas is contrary to a reasonable interpretation of claim 1 of the patent as granted and would violate the spirit of the invention.

Similarly, when carrying the claimed process, the person skilled in the art would not be guided by the objective of obtaining 0 ppm of solid ureas formed, as this is impossible to achieve in this process.

In the Board's understanding, the requirement that solid carbamoyl chlorides and/or solid ureas are not present at the point where residual excess phosgene is removed from the reaction mixture is satisfied by using the means known from the prior art in this respect. The presence of solid ureas can be avoided by using known mixing devices that minimise urea by-products, thereby

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avoiding the formation of insoluble polyureas. It was not disputed that carbamoyl solids are avoided by heating the reaction mixture, as indicated in the patent on page 4, lines 52-53.

Accordingly, the Respondent's arguments that the requirements of Article 83 EPC are not met because the claimed process cannot be carried out with large amounts or with 0 ppm solid ureas is rejected.

4. According to the Respondent, the skilled person was not able to carry out the claimed process without undue burden since there was nothing in the patent in suit indicating how to measure the residual content of amine hydrochloride solids mixed with other solids.

Determination of the residual content of amine hydrochloride solids needed to be done instantly, at the point where residual phosgene was removed.

However, claim 1 does not require an instant measure of the amount of amine hydrochloride. In order to carry out the process with an appropriate content of amine hydrochloride solids, the skilled person can take an aliquot of the reaction mixture at the time where the excess of phosgene is removed, filter it, weight the solids present in the mixture, and then determine the amount of the amine hydrochloride in the filtered solids. If the amount of solid amine salt present in the reaction mixture is too high or too low, the process parameters can be adjusted, for example by decreasing or increasing the residence time of the reaction mixture, until a value of the residual amine salt within the claimed range is obtained.

5. According to the Respondent, the amine hydrochloride content could not be determined by the skilled person

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without undue burden. The development of an analytical method for the determination of solid amine hydrochlorides in a complex precipitate, which contained other constituents, was extremely complex and would require several weeks of development by an expert in the field of solid analysis. This amounted to an undue burden.

First, the Board notes that it is not necessary to analyse the content of the amine hydrochloride present in the solid precipitate in the solid state. Without evidence to the contrary, the determination of the content by weight of amine hydrochloride salts in a complex mixture containing urea derivatives or carbamoyl derivatives is deemed to be achievable by numerous analytical methods, or even by separation of the amine salt fraction. The statement that the analysis of the amine hydrochloride content of the precipitate would last several weeks is unfounded. Accordingly, the Respondent's argument should be rejected, especially as the Respondent has made no (unsuccessful) attempt to analyse such precipitate.

6. According to the Respondent, the relative amount in ppm of solid amine hydrochlorides could not be deduced from a turbidity value.

The Board notes that claim 1 does not require the relative amount in ppm to be deduced from the turbidity value. The Respondent's objection is thus not convincing for this reason alone.

The objection could apply at the most to the example of the patent in suit, in which the part per million level of solid amine hydrochloride is determined by a calibrated turbidity meter. The process disclosed in - 10 - T 0089/17

the example of the patent in suit is carried out at a temperature of 80 to 95 °C, such that no carbamoyl solid is present. Furthermore, the first stage of the process is carried out in a static mixing nozzle device, which avoids the formation of solids ureas.

With the help of suitable reference compositions containing known amounts of solid amines formed by the reaction, it would have been possible to correlate the turbidity value with the amount of solid amine suspended in the solution with a given degree of accuracy. The value in ppm of solid amine can be adjusted if necessary by taking into account other solid components present in the reaction medium, such as ureas solids.

The Board therefore does not see any deficiency in the description of the invention in the fact that the example in the patent in suit indicates that the quantity of solid amine is given in ppm on the basis of a turbidity value.

This objection of the Respondent must also be rejected.

7. The Board comes therefore to the conclusion that the invention as defined in claim 1 of the patent as granted can be performed by a person skilled in the art without undue burden, using common general knowledge and having regard to further information given in the patent in suit, such that the opposition ground pursuant to Article 100(b) EPC fails.

Other issues

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8. There were requests to not admit documents to the appeal proceedings. However, as the documents are not needed by the Board to arrive at its conclusion that the invention is sufficiently disclosed, their admission into the proceedings need not be decided by the Board. If necessary, their admission may be decided by the Opposition Division following the remittal.

Remittal

9. As the opposition division has not yet ruled on the ground of opposition pursuant Article 100(a) EPC, the Board considers it appropriate to remit the case to the Opposition Division for further prosecution. This was also requested by both parties.

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the Opposition Division for further examination on the basis of the patent as granted.

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The Registrar:

The Chair:



C. Rodríguez Rodríguez

R. Pérez Carlón

Decision electronically authenticated