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Datasheet for the decision of 13 February 2020

Case Number: T 1327/16 - 3.3.10

Application Number: 06781144.8

Publication Number: 1902016

IPC: C07C263/20, C07C265/14

Language of the proceedings: EN

Title of invention:
Method for concentrating and treating polyisocyanate residues

Patent Proprietor:
Mitsui Chemicals, Inc.

Opponent:
The Dow Chemical Company

Headword:

Relevant legal provisions:
EPC Art. 100(a), 56
RPBA Art. 12(4)

Keyword:
Inventive step - (no)

Decisions cited:

Catchword:

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Case Number: T 1327/16 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 13 February 2020

Appellant: (Patent Proprietor)	Mitsui Chemicals, Inc. 5-2, Higashi-Shimbashi 1-chome Minato-ku Tokyo 105-7117 (JP)
Representative:	Mewburn Ellis LLP City Tower 40 Basinghall Street London EC2V 5DE (GB)
Respondent: (Opponent)	The Dow Chemical Company Intellectual Property 2030 Dow Center Abbott Road Midland, MI 48640 (US)
Representative:	Beck Greener LLP Fulwood House 12 Fulwood Place London WC1V 6HR (GB)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 24 March 2016 revoking European patent No. 1902016 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	P. Gryczka
Members:	R. Pérez Carlón
	T. Bokor

Summary of Facts and Submissions

I. The appellant (patent proprietor) lodged an appeal against the decision of the opposition division revoking European patent No. 1 902 016.

II. Notice of opposition had been filed on grounds including lack of inventive step (Article 100(a) EPC).

III. Claim 1 of the patent as granted reads as follows:

"*A method for producing a polyisocyanate and concentrating polyisocyanate residues, comprising*

(i) reacting a polyamine with carbonyl chloride to produce a crude polyisocyanate containing polyisocyanate and polyisocyanate residues,
(ii) carrying out a desolvating process on the crude polyisocyanate produced in step (i);
(iii) carrying out a first concentrating process of concentrating the polyisocyanate residues from the crude polyisocyanate produced in step (ii) to a midterm concentrating rate on the way to a final concentrating rate by heating the crude polyisocyanate which is on the boil, to obtain a first concentrated component, said first process using a distillation column at a column bottom temperature within the range of 155 to 190°C and under a column inner pressure within the range of 0.05 to 30 kPa, and
(iv) carrying out a second concentrating process of concentrating the first concentrated component concentrated in the first concentrating process to the final concentrating rate by evaporation using a thin film evaporator, to obtain a second concentrated component,

a polyisocyanate of the crude polyisocyanate being selected from tolylene diisocyanate, xylylene diisocyanate, tetramethylxylylene diisocyanate, 4,4'-methylenebis (cyclohexylisocyanate), bis (isocyanatomethyl) cyclohexane, and hexamethylene diisocyanate."

IV. The documents filed during the opposition proceedings include the following:

D7 US 3,128,310
D9 US 3,140,305
D10 EP-A-0 497 538

V. The opposition division concluded that document D10 was the closest prior art and that the problem underlying the claimed invention was the provision of an alternative method for producing a polyisocyanate. The solution, which was characterised by including a desolvating step (ii) and by the pressure required in step (iii), would have been obvious for the person of the art as step (ii) was frequently used in the field of polyisocyanate synthesis by phosgenation, and adjusting the pressure and temperature of a distillation column would have fallen within the skills of the person of the art. The arguments also applied to the method of claim 1 of the sole auxiliary request then pending.

VI. With the statement of the grounds of appeal, the appellant filed seven auxiliary requests(Auxiliary Requests I to VII), of which the second and sixth were withdrawn during the oral proceedings before the board on 13 February 2020. The remaining auxiliary requests

were not renumbered.

VII. Claim 1 of the first auxiliary request requires, in addition to the features of claim 1 of the main request, that step (iii) of claim 1 is carried out

"to distill polyisocyanate together with a Cl-containing gas"

and further requires that

"*the content of the polyisocyanate is 95-60 weight% and the content of the polyisocyanate residues is 5-40 weight per 100 weight% of the first concentrated component".*

Claim 1 of the third auxiliary request contains all the features of claim 1 of the first auxiliary request, adding a further step (v) as follows:

"*(v) carrying out a process of treating the polyisocyanate residues wherein the second concentrated component produced in step (iv) is put in contact with high temperature and high pressure water and is decomposed to polyamine.*"

Claim 1 of the fourth, fifth and seventh auxiliary requests contains all the features of claim 1 of the main request and the first and third auxiliary requests, respectively, and further requires the polyisocyanate to be tolylene diisocyanate (TDI).

VIII. The arguments of the appellant relevant to the present decision were as follows:

The first, second, fifth and seventh auxiliary requests

had been filed with the statement of grounds of appeal and represented a reaction to the decision under appeal. Thus, they should be admitted into the proceedings.

Document D10 was the closest prior art. Step (iii) of claim 1 of the patent as granted required distilling polyisocyanate together with chlorine-containing volatile components, prior to the thin film distillation step. This feature was responsible for reducing residue viscosity and the time required. If, however, the problem were considered to be merely the provision of an alternative method to that of D10, the solution, which was characterised by distilling polyisocyanate together with chlorine-containing volatile components in step (iii) according to claim 1, would not have been obvious for the person of the art and therefore inventive.

Claim 1 of the first auxiliary request explicitly required polyisocyanate to be distilled together with chlorine-containing volatile components in step (iii) and thus solved any issue which could derive from a wrong interpretation of claim 1 of the patent as granted. The arguments with respect to inventive step were the same as above.

Claim 1 of the third auxiliary request required a further step not disclosed either in D10 or D9. For this reason alone, the method of claim 1 of this request was inventive.

The appellant agreed that the examination of inventive step of claim 1 of the remaining auxiliary requests would not differ from that of those mentioned above.

IX. The arguments of the respondent (opponent) relevant to the present decision were as follows:

The first, second, fifth and seventh auxiliary requests could have been filed before the opposition division and should therefore not be admitted into the proceedings. If any of them were admitted, the case should be remitted to the opposition division to not deprive the opponent from two instances.

Document D10 was the closest prior art. It disclosed all the features of claim 1 with the exception of step (ii) and the pressure required by step (iii) of claim 1 of the main request, given that step (iii) of claim 1 did not require the separation of polyisocyanate from the starting mixture. The problem of providing an improved method was not credibly solved by the features of claim 1 given the lack of fair comparative data with respect to D10. Thus, the technical problem had to be reformulated as the provision of an alternative method for producing a polyisocyanate and concentrating polyisocyanate residues. The solution, which was characterised by the features mentioned above, would have been a straightforward choice for the person skilled in the art. The claimed process was thus not inventive.

Even if step (iii) of the main request required separating polyisocyanate from the mixture, as argued by the appellant, such an option would have been obvious for the skilled person seeking an alternative. Document D9 disclosed a two-step distillation using conditions very similar to those required by claim 1. For this reason, the method of claim 1 of the main request and the first auxiliary request were not inventive.

Lastly, document D10 hinted at the obtaining of diamine from diisocyanate distillation bottoms. Document D7 disclosed the experimental details of such a step. For this reason also the method of claim 1 of the third auxiliary request was not inventive.

Claim 1 of the fourth, fifth and seventh auxiliary requests was also not inventive for the same reasons provided for the preceding requests.

X. The final requests of the parties were as follows:

- The appellant requested that the decision under appeal be set aside and that the patent be maintained as granted (main request) or in an amended form on the basis of one of the auxiliary requests I, III to V or VII filed with the grounds of appeal dated 3 August 2016.

- The respondent requested that the appeal be dismissed. Additionally, it requested non-admission of auxiliary requests I, III, V and VII and, if any of these were admitted, a remittal to the opposition division for further prosecution.

XI. At the end of the oral proceedings, the decision was announced.

Reasons for the Decision

1. The appeal is admissible

Inventive step, main request

2. Claim 1 relates to a method for producing a polyisocyanate and concentrating polyisocyanate residues comprising four steps. Step (i) requires phosgenation of a polyamide, which is desolvated in a subsequent step (ii). These steps are followed by two concentration steps, (iii) and (iv).

The first of the concentration steps (iii) requires concentrating polyisocyanate residue using a distillation column at a defined bottoms temperature and inner pressure.

In a last step (iv), the product of step (iii) is concentrated using a thin film evaporator.

3. Interpretation of step (iii) of claim 1

It was a point of dispute between the parties whether step (iii) of claim 1 required polyisocyanate to be distilled off during the process. The opposition division considered, in agreement with the respondent, that this was not the case. The board informed the parties during the oral proceedings that it was also of this view.

Nevertheless, in this decision it will be considered that step (iii) of claim 1 requires distilling off polyisocyanate together with chlorine-containing gas, as argued by the appellant.

The conclusion on the issue of inventive step, even taking the most favourable interpretation for the appellant, is negative for the reasons below.

4. Closest prior art

The parties and the opposition division considered document D10 to be the closest prior art, and the board sees no reason to differ.

Like the patent in suit, document D10 relates to the issue of viscosity of the residues obtained in the synthesis of polyisocyanates (page 3, lines 6-11).

The parties agreed that document D10 did not disclose the desolvating step (ii) and the pressure required by step (iii).

The appellant argued that document D10 did not disclose the step of distilling polyisocyanate together with a chlorine-containing gas. In the appellant's favour, this is considered to be the case in the following.

5. Problem underlying the claimed invention

The appellant defined the problem underlying the claimed invention as the provision of a method for producing a polyisocyanate and concentrating polyisocyanate residue which allowed reduced viscosity of the residue and could be carried out in less time.

6. Solution

The claimed solution is the method of claim 1, characterised by distilling polyisocyanate together with a chlorine-containing gas at reduced pressure. The appellant conceded that the desolvating step (ii) of claim 1 did not contribute to the solution of the problem as defined above.

7. Success of the claimed solution

There is no direct comparison between the claimed process and the process disclosed in the closest prior art D10 which could show the effect of the distinguishing features of the claimed invention, either in terms of reduced viscosity or total duration of the purification procedure.

The appellant argued that the patent in suit [0038] disclosed that polyisocyanate entrained chlorine-containing gases during distillation. Due to this entrainment, the separation of chlorine-containing gases was more efficient. The patent in suit and D10 disclosed the influence of the amount of chlorine-containing gases on the viscosity of the distillation residue. For this reason, it was credible that the claimed process solved the problem of decreasing viscosity of polyisocyanate residue.

However, there is no clear technical reason why the entrainment relied on by the appellant should make the separation of chlorine-containing gases more efficient. In fact, entrainment is often a hindrance in a separation rather than a help. In addition, no evidence has been provided suggesting that entrainment is an advantage in the situation of the claimed process. This argument of the appellant is thus unconvincing.

It is thus not credible that the problem as defined by the appellant is solved by the method of claim 1.

8. Reformulation of the technical problem

As the problem put forward by the appellant cannot be considered as solved, it should be reformulated as the provision of an alternative method for producing

polyisocyanate and concentrating polyisocyanate residues.

The parties agreed that the features of claim 1 credibly solved this problem, and the board sees no reason to differ.

9. It remains to be decided whether the proposed solution would have been obvious for the skilled person in view of the prior art.

The skilled person, seeking an alternative method, would have taken into account the teaching of a document such as D9, which discloses, like D10, a process for obtaining diisocyanates such as TDI by phosgenation of the corresponding diamine.

Document D9 discloses a method of preparing TDI by (i) reacting toluene diamine with phosgene (column 4, line 37), followed by (ii) stripping off the solvent (column 4, line 39), (iii) distilling a part of the TDI in a distillation column at 10 mmHg (1.3 kPa) and at a temperature of 120°C to 137°C (column 4, lines 40-43) and transferring the bottoms to a wiped-film evaporator where additional TDI is separated (iv).

The skilled person would have combined the teaching of D9 and D10 and thus would have arrived at the claimed invention without using inventive skills.

The sole feature of claim 1 not explicitly disclosed in D9 is the distillation temperature of the bottoms (155-190°C) required by step (iii). Instead, D9 discloses a TDI distillation temperature (120-137°C), which inevitably implies a higher bottom temperature.

Document D10 discloses heating as crucial for decomposing hydrolysable chlorine-containing products into HCl and isocyanates and reducing the solubility of HCl in refluxing vapour (page 3, lines 50-53). Example 1 discloses heating crude TDI at 186°C. The skilled person would thus have been taught that this temperature is essential for maintaining the effect sought by D10. This temperature is further compatible with the distillation step in document D9. Seeking an alternative, the skilled person would have used these teachings and arrived at the claimed invention without using inventive skills.

For this reasons, the method of claim 1 of the patent as granted is not inventive (Article 56 EPC) even when taking the position most favourable to the appellant, namely that claim 1 requires distillation of polyisocyanate in step (iii) and that document D10 does not disclose this feature.

The ground for opposition under Article 100(a) EPC thus precludes the maintenance of the patent as granted.

10. Admission of Auxiliary Requests I, II, V and VII

The respondent has requested the non-admission of these auxiliary requests.

The board considers that the requests were filed with the statement of grounds of appeal and represent a reasonable response to the decision of the opposition division, in particular with respect to the interpretation of step (iii) of claim 1. The board sees no reason not to admit these requests into the proceedings (Article 12(4) RPBA 2007 in conjunction with Article 25(2) RPBA 2020).

The respondent requested that the case be remitted to the opposition division if any of these requests were admitted.

The board considers the issues under examination to remain within the framework of the discussion conducted in respect of those requests which were clearly admissible, and thus sees no reason for remittal. As the appeal is not allowable for other reasons, there is no need to further elaborate on this point.

Inventive step, Auxiliary Requests I, IV and V

11. As the board has examined the ground of lack of inventive step of claim 1 of the patent as granted considering that it requires the distillation of polyisocyanate in step (iii), the arguments put forward above apply analogously to claim 1 of the first auxiliary request which explicitly requires such a distillation. The appellant did not rely on the feature defining the content of polyisocyanate and residue in the first concentrated component for the issue of inventive step. This feature is disclosed in document D9, according to which a first concentrated component having 84.6% TDI is formed (column 4, line 45).

Auxiliary request I is thus not inventive and therefore not allowable.

12. Claim 1 of Auxiliary Requests IV and V contains all the features of claim 1 of the main request and Auxiliary Request I, respectively, and further requires the polyisocyanate to be TDI. As both D9 and D10 relate to the preparation of this compound, the arguments given above for lack of inventive step of the main request

and Auxiliary Request I also apply to claim 1 of Auxiliary Requests IV and V.

Inventive step, Auxiliary Requests III and VII

13. Claim 1 of these requests have all the features of claim 1 of the main request and Auxiliary Request I, respectively, and require a further step (v) of treating the residue of step (iv) with water to decompose it into polyamine.

Claim 1 of Auxiliary Request VII differs from that of Auxiliary Request III by requiring the polyisocyanate to be TDI.

14. Closest prior art

The parties agreed that document D10 is also the closest prior art for the method of claim 1 of these requests.

15. Problem underlying the claimed invention

Without a comparison reflecting the distinguishing features of the invention, the problem underlying the claimed invention is the provision of an alternative method for producing a polyisocyanate and concentrated isocyanate residues which allows using these residues.

16. Solution

The claimed solution to this problem is the method of claim 1, characterised by a desolvation step (ii), distillation at reduced pressure so that polyisocyanate is separated together with chlorine-containing product, and a step of recovering polyamine from the residue (v)

by putting the residue obtained in step (iv) in contact with high temperature and high pressure water.

It was not in dispute that this problem has been credibly solved.

17. Document D10 hints at hydrolysis of TDI residue into toluene diamines on page 2, lines 46 and 47.

The skilled person seeking a method that would allow using the distillation residues would have found the experimental conditions for hydrolysing TDI into toluene diamines in a document such as D7. D7 discloses (column 2, lines 6 to 21) that aromatic amines can be obtained from the distillation residue from the preparation of aromatic isocyanates such as TDI by hydrolysis at 160°C to 250°C with super-heated steam in a pressure vessel.

For the reasons given above in the context of the main request (see point 9.), the separation of TDI by distillation in step (iii) and the desolvating step (ii) would have been obvious for the skilled person having regard to D10 and D9.

For these reasons, the method of claim 1 of the third and seventh auxiliary requests are not inventive, and these requests are thus not allowable.

18. As the appeal cannot be allowed for the reasons above, the board does not need to decide on any other point.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

C. Rodríguez Rodríguez P. Gryczka

Decision electronically authenticated