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# Datasheet for the decision of 29 June 2022

Case Number: T 0691/19 - 3.3.03

Application Number: 13719505.3

Publication Number: 2855559

IPC: C08G65/30

Language of the proceedings: EN

#### Title of invention:

PROCESS FOR THE PRODUCTION OF POLYETHER POLYOLS

## Patent Proprietor:

Huntsman International LLC

#### Opponent:

Covestro Deutschland AG

#### Relevant legal provisions:

EPC Art. 100(b), 54, 56

# Keyword:

Grounds for opposition - insufficiency of disclosure (no) Novelty - (yes) Inventive step - (yes)

#### Decisions cited:

G 0001/03, T 1854/14



# Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 0691/19 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 29 June 2022

Appellant: Covestro Deutschland AG (Opponent) Kaiser-Wilhelm-Allee 60 51373 Leverkussen (DE)

Representative: Levpat

c/o Covestro AG Gebäude 4825

51365 Leverkusen (DE)

Respondent: Huntsman International LLC

(Patent Proprietor) 500 Huntsman Way

Salt Lake City, UT 84108 (US)

Representative: Meissner Bolte Partnerschaft mbB

Widenmayerstrasse 47 80538 München (DE)

Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 3 January 2019 rejecting the opposition filed against European patent No. 2855559 pursuant to Article 101(2)

EPC.

# Composition of the Board:

Chairman D. Semino
Members: F. Rousseau

A. Bacchin

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# Summary of Facts and Submissions

- I. The appeal lies against the decision of the opposition division rejecting the opposition against European patent No. 2 855 559, whose claim 1 reads as follows (for ease of understanding bullet points in the text as granted have been replaced by the Board by a numbering (1) to (6) of the process steps of the claimed method):
  - "1. A method to provide polyether polyols, the method comprising the steps of
  - (1) providing a crude polyether polyol mixture comprising polyether polyol and a base catalyst;
  - (2) mixing the crude polyether polyol mixture with an acid and water, thereby neutralizing said base catalyst and providing a first neutralized polyether polyol mixture;
  - (3) removing, in a first dehydration step, at least part of the water from said first neutralized polyether polyol, thereby providing a first dehydrated neutralized polyether polyol mixture having a water content in the range 0.00 to 5.00 %w and comprising said polyether polyol and salt of said base catalyst and said acid, said salt being present as salt crystals;
  - (4) redissolving at least part of the salt by adding water to said first dehydrated neutralized polyether polyol mixture, thereby providing a second neutralized polyether polyol mixture;
  - (5) removing, in a second dehydration step, at least part of the water from said second neutralized polyether polyol mixture, thereby providing a second dehydrated neutralized polyether polyol mixture

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comprising said polyether polyol and salt of said base catalyst and said acid, said salt being present as salt crystals;

- (6) removing said salt crystals from said second dehydrated neutralized polyether polyol, thereby providing the polyether polyol mixture."
- II. The opposition proceedings were based among others on the following items of evidence:

D1: WO 2010/145899 A1,

D2: WO 99/47582 A1

D3: DE 102 27 655 A1

D4: DD 155 428

D5: M. Ionescu, Chemistry and Technology of Polyols for Polyurethanes, 2005, pages 55, 118-165, ISBN:

1-85957-491-2

D6: JP 05-111602 (A) and translation thereof in English

D7: US 2008/0300222 A1

D7a: experimental data contained in section 1.2.2 of the notice of opposition (pages 16/32 to 20/32)

D7b: experimental report contained on pages 5-7 of the patentee's letter of 29 June 2017

D8: experimental report concerning the example of D1 submitted with the opponent's letter of 24 August 2018.

- III. According to the reasons for the contested decision which are pertinent for the appeal proceedings:
  - (a) D8 was admitted into the proceedings.
  - (b) Sufficiency of disclosure was acknowledged.
  - (c) Novelty over each of D1 to D4 was given.

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- (d) An inventive step was acknowledged, the closest prior art being represented by the method exemplified in D1.
- IV. An appeal against that decision was lodged by the opponent (appellant).
- V. The patent proprietor (respondent) submitted with letter of 23 February 2022 a first and a second auxiliary request whose wording is not relevant for the present decision.
- VI. Oral proceedings before the Board were held on 29 June 2022.
- VII. The appellant requested that the decision under appeal be set aside and that the patent be revoked.
- VIII. The respondent requested that the appeal be dismissed, or alternatively that the decision be set aside and the patent be maintained on the basis of the first or second auxiliary request, both submitted with letter of 23 February 2022.
- IX. The appellant's submissions, in so far as they are pertinent, may be derived from the reasons for the decision below. They are essentially as follows:
  - (a) The method of claim 1 lacks sufficiency of disclosure, since the teaching concerning step (4) is insufficient.
  - (b) Claim 1 lacks novelty over each of the methods described with the example of D1, examples 1 to 3 of D2, example 3 of D3 and examples 1 and 2 of D4.

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- (c) Claim 1 lacks an inventive step starting as the closest prior art from any of the methods described with the example of D1, example 3 of D3 and examples 1 and 2 of D4.
- X. The submissions of the respondent, in so far as they are pertinent, may be derived from the reasons for the decision below. They are essentially as follows:
  - (a) The method of claim 1 is sufficiency disclosed.
  - (b) The method of claim 1 is novel over each of D1, D2, D3 and D4.
  - (c) The method of claim 1 involves an inventive step.

#### Reasons for the Decision

# Preliminary remark

1. The method of operative claim 1 concerns the preparation of polyether polyols, which are known to be typically prepared by reacting a starting compound having a plurality of active hydrogen atoms with one or more alkylene oxides in the presence of a base catalyst, preferably a strong base such as potassium hydroxide (patent in suit, paragraph [0002]; D5, pages 120 and 121, sections 4.1.5.1 and 4.1.5.2). According to the state of the art, such strong base must be removed from the polyether polyol, in particular when the polyether polyol is to be used with isocyanate compounds for the production of polyurethanes (patent in suit paragraph [0002]; D5, page 129, section 4.1.5.5). According to D5 (page 130, third point a)),

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neutralisation of potassium hydroxide with acids, followed by the crystallization of the resulting potassium salts and filtration is known to be applied industrially. The method of claim 1 as granted concerns the preparation of polyether polyols comprising a neutralization step of the base catalyst with an acid in order to form crystals which are removed at the end of the process. It is defined by steps (1) to (6) referred to in above section I.

## Article 100 (b) EPC

- 2. The appellant objects that operative claim 1 lacks sufficiency of disclosure, since the teaching concerning step (4) is insufficient. The appellant points out that the experiments contained in D7a show in step (4) a redissolution of less than 0,4 wt% of the salt obtained after step (3). This would demonstrate that step (4) does not result in the dissolution of salts produced during step (3) or not in a dissolution of a sufficient amount thereof so as to obtain salt crystals after performing step (6) whose size has been doubled in comparison to the salt crystals obtained at the end of step (3).
- This objection is based on an incorrect premise, namely that claim 1 requires a minimum amount of salt to be redissolved by performing step (4), let alone in order to obtain crystals whose size at the end of step (6) has been increased by a minimum level in comparison to the crystals obtained in step (3). As reminded in decision T 1845/14 of 8 November 2018 (points 9 to 9.8 of the Reasons), the achievement of a particular technical effect which is not part of the claim definition, here the dissolution of a specific quantitative amount of salt in step (4) to achieve a

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certain size of the crystals after performing step (6) in relation to the size of the crystals obtained at the end step (3), is not an issue of sufficiency of disclosure, but may be relevant for the question of inventive step (see decision of the Enlarged Board of Appeal G 1/03, OJ EPO 2004, 413, point 2.5.2, third paragraph of the reasons).

- 2.2 As pointed out by the opposition division, the experiments D7a submitted by the respondent show that at least part of the salt, even if a small part, can be redissolved when adding 5 wt% of water.
- 2.3 Moreover, having regard to common general knowledge, the formation of salt crystals from a water phase depends on its supersaturation with said salts. It is therefore credible that the addition of water to the dehydrated mixture obtained after performing step (3) results in a new equilibrium between the amount of salt dissolved in the aqueous phase and that present in the crystals, i.e. in some part of the salt in crystal formed at the end of step (3) to be redissolved in step (4). How much from the salt is redissolved is for the skilled person obviously dependent on many variables, such as (i) the amount of water present after performing step (3), as well as the amount of crystals and their size obtained at the end of said step, and (ii) the conditions applied in step (4), e.g. temperature, amount of water added and duration of that step. The skilled person is also aware based on common general knowledge that by using higher temperature, higher amount of water, as submitted by the respondent, and longer duration dissolution of some part of the salt present in crystal form at the end of step (3) will be facilitated. The general teaching in relation to step (4) concerning in particular the temperature to

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be used and the amount of water which may be added is provided in paragraphs [0026] to [0028] of the specification.

- 2.4 On that basis, it is credible that the skilled person would be in the position to perform step (4) as defined in operative claim 1, which step requires nothing more than redissolving an unspecified amount of the salt obtained at the end of step (3).
- 2.5 Accordingly, the objection that operative claim 1 lacks sufficiency of disclosure is not convincing.

Novelty over D1

- 3. The appellant objects that the subject-matter of claim 1 lacks novelty over the method described from page 8, line 23 to page 9, line 7 of D1. This method consists in (reference to steps (a) to (d) have been added by the Board for ease of understanding):
  - (a) heat treating an alkaline polyether polyol with a first charge of pure sulphuric acid and water for 30 minutes before a sample is taken to check the acid value of the polyether polyol, which is measured to be 0,01 mg KOH/g and found to be below the targeted value (between 0,05 and 0,1 mg KOH/g) (page 8, line 30 ff) and necessitates
  - (b) a second addition of pure sulphuric acid and water to reach an acid value of 0,09 mg KOH/g after additional 30 minutes of heat treatment, which is then followed by
  - (c) a dehydration step using vacuum distillation and
  - (d) a filtration step to remove the crystals formed (see also claim 1 and page 5, lines 22-26).

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Based on experimental report D8, which is alleged by the appellant to constitute a proper rework of the example of D1, the appellant submits that

- (i) a first neutralized polyether polyol within the meaning of step (2) of operative claim 1 would be obtained after 3 minutes of the above mentioned heat treatment in step (a)
- (ii) the rest of the heat treatment in step (a) would correspond to step (3) of operative claim 1, as the water content of the polyether polyol during the rest of step (a) would go from 2,82 wt% (observed after 3 minutes of step (a)) down to 2,48 wt% at the end of said step (a),
- (iii) steps (b) to (d) would correspond to steps (4) to
  (6) of operative claim 1.
- 3.1 The parties are in dispute whether a neutralized polyether polyol can be obtained after 3 minutes of the heat treatment of step (a) and whether the rest of that heat treatment of step (a) constitute a first dehydration step (3) within the meaning of operative claim 1.
- As regards the first issue, the acidity value of the polyether polyol, which is taken as an indicator whether the polyether polyol can be considered to have been neutralized, is checked in the example of D1 at the end of the 30 minutes of step (a). This, as submitted by the respondent, can only be justified by the fact that 3 minutes of treatment are contrary to the respondent's view not enough in the opinion of the skilled person to provide a neutralised polyether polyol.

As regards experimental report D8, its probative value is questionable, since the acid values of the polyether

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polyols measured during step (a) (44 and 37 ppm KOH after 3 minutes and at the end of step (a), respectively) are much higher than that of 0,01 mg KOH/g, (i.e. 10 ppm KOH) described in D1 for the end of step (a), which casts doubts on whether the method shown in D8 represents that described in D1. It can be therefore questioned whether the polyether polyol obtained in D1 after 3 minutes of step (a) is neutralized and correspond to that obtained in D8 after 3 minutes of the first acid heat treatment.

- 3.3 In any event and with particular regard to the second issue at dispute, even if D8 were taken as a proper repetition of the method disclosed in D1, D1 does not disclose a dehydration step in which at least part of the water is removed from said first neutralized polyether polyol. This, as submitted by the respondent, implies for the skilled person the use of specific active measures to evacuate the water vapour present above the liquid neutralised polyether polyol, in line with the measures preconised both in the patent in suit (paragraph [0022]) and in D1 for the above mentioned step (c) (page 5, lines 1-11). In the method exemplified in D1, specific measures which would result in an evacuation of the water vapour above the liquid neutralized polyether polyol are not employed until the beginning of step (c).
- 3.4 Considering in addition that the definition of step (2) in operative claim 1 does not exclude two successive additions of acid and water, meaning that steps (a) and (b) of the method exemplified in D1 can be seen as forming a step (2) within the meaning of present claim 1, it is concluded that the method of operative claim 1 differs from that exemplified in D1 in that it comprises in addition steps (4) and (5).

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3.5 On that basis, novelty of the subject-matter of claim 1 over D1 is acknowledged.

Novelty over D2

4. The appellant submits that examples 1 to 3 of D2 (page 8, line 10 to page 10, line 17) are novelty destroying for the method of operative claim 1.

All these examples describe in a first step a heat treatment in presence of a 30% excess of acid relative to the amount of potassium contained in the unneutralised polyether polyol, followed by a second heat treatment in the presence of added water in order to hydrolyse under such acidic milieu the propenylether end groups and cyclic ethers present in the polyether polyol as impurities (claim 1, page 3, lines 10-19 and page 4, lines 13-16). The methods described in examples 1 to 3 comprise as a third step a subsequent neutralisation step with a 50 wt% KOH solution. The neutralization step is followed by a dehydration step and a filtration step to remove the crystals.

The appellant's objection is based inter alia on the argument that the addition of acid in the first step described above corresponds to steps (2) of operative claim 1 (grounds of appeal, page 23, last paragraph). This, however, already fails to convince, since the first step of the examples 1 to 3 of D2 results in an acidic polyether polyol, a neutralized polyether polyol being only obtained after the third step of the methods of examples 1 to 3.

Moreover, the appellant's objection is based on the additional argument that water has been added through

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the addition of acid and that the final part of the first heat treatment carried out before the addition of water in examples 1 to 3 corresponds to a step (3) of operative claim 1 (grounds of appeal, page 24, first paragraph). It is argued that the water originally introduced with the acid is necessarily distributed between the liquid phase of the reaction and the vapour phase above the latter, leading to a diminution of the amount of water present in the polyether polyol. This also is not convincing for the reasons already provided in above point 3.3.

Even if to the benefit of the appellant, the three first steps of the methods described with examples 1 to 3 of D2 (first heat treatment in the presence of acid in excess, second heat treatment in the presence of added water and neutralization step) were considered to represent step (2) of operative claim 1, these examples would not describe steps (4) and (5) as defined in present claim 1. Indeed a single dehydration step followed by removal of the crystals takes place after neutralisation in D2.

4.2 Consequently, novelty of the subject-matter of granted claim 1 over D2 is also acknowledged.

# Novelty over D3

5. The appellant objects that operative claim 1 lacks novelty over example 3 of D3. It is undisputed that the first three steps used in that example correspond to steps (1) to (3) of operative claim 1, in particular that crystals are present in the polyether polyol after performing the vacuum distillation step. Example 3 of D3 describes as a subsequent step the addition of a 48% solution of KOH to the dehydrated neutralized polyether

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polyol which the appellant considers to be in accordance with step (4) of operative claim 1. The final polyether polyol product is obtained after an additional dehydration step, followed by a filtration step.

However, the addition of water as required by step (4) of granted claim 1 does not encompass for the skilled person the addition of a strong base such as a 48% solution of KOH. There is no reason to interpret the term water for the definition of step (4) as allowing for the addition of any compound comprising water. The specification does not give rise to a different understanding of the term water. According to paragraph [0026] the water added in step (4) may be e.g. distilled water (or condensate) or demineralised water. The indication in paragraph [0030] that optionally but not preferred, further components such as crystal growth promoting components (also referred to as seeds) may be added during or after the addition of the water does not justify, even if those seeds were added in admixture with water, an interpretation of the term "water" in step (4) of claim 1 to mean any compound comprising water, in particular a 48% solution of KOH.

There is also no indication in that document that the addition of said 48% solution of KOH would result in redissolution of at least part of the salt. Evidence in this respect was also not submitted. The appellant's additional argument that a sufficient disclosure of the method of operative claim 1 is to be acknowledged over the whole breadth of the claim would imply that at least part of the salt is dissolved by the addition of the 48% solution of KOH does not convince, since the the addition of such base solution is not, as shown in

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the above paragraph, in accordance with the invention defined in operative claim 1.

Under these circumstances, the subject-matter of claim 1 is novel vis-à-vis D3.

# Novelty over D4

- 6. The appellant submits that examples 1 and 2 of D4, as well as its claim 1 read in combination with the passage of the general teaching starting with the last seven lines on page 3 and continuing with the first seven lines on page 4 would anticipate the method of present claim 1.
- 6.1 It is undisputed that examples 1 and 2 of D4 describe a method using in this order steps (1) to (3) of operative claim 1, a filtration step, an additional heat treatment after water has been added and steps (5) and (6) of operative claim 1. As to the intermediate filtration step, it is explicitly described in examples 1 and 2 that the precipitated salts obtained by performing the steps corresponding to steps (1) to (3) of present claim 1 are filtered off. Operative claim 1, however, does not allow for such a step, since step (4) requires that at least part of the salt is redissolved by adding water to the first dehydrated neutralized polyether polyol mixture obtained in step (3). The description of the patent in suit does not give ground for any different reading, as paragraph [0025] only indicates that a part of the formed crystals may be removed, e.g. filtered, from the first dehydrated polyether polyol mixture. Even if theoretically for examples 1 and 2 of D4 some small crystals could have passed trough the filter and remained in the filtrated first dehydrated polyether polyol, no evidence has been

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provided that the subsequent addition of water in the step corresponding to step (5) of operative claim 1 would result in a <u>measurable amount</u> of salt to redissolve.

- The appellant's also argues that it must be assumed that the secondary phosphoric acid salts obtained in example 2 of D2, i.e. K2HPO4, must have been filtered out only at the end of the process described in example 2, since this salt is difficult to crystallize. This argument, in contradiction with the explicit disclosure in example 2 that a filtration is operated after the first dehydration step, is also not corroborated by any evidence showing that K2HPO4 crystals cannot be formed under the conditions used in example 2 of D2 for the first dehydration step. The argument that D4 teaches a single filtration operated at the end of the method is therefore not convincing.
- 6.3 The appellant's additional argument that the general teaching of D4 does not foresee a filtration step after the first dehydration step fails also to convince. The absence of an explicit teaching in this respect in the general description of the patent in suit does not constitute any teaching going against the explicit indication in claim 1 of D4 which represents the broadest definition of the invention in accordance with D4, the examples of D4 confirming also the use of a filtration step after the first dehydration step.
- 7. On that basis, the method of operative claim 1 has not been shown to be anticipated by D4.

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Inventive step starting from the method exemplified in D1

8. Both parties consider that the method exemplified in D1 constitutes a suitable starting point for assessing inventive step. The Board has no reason to have a different opinion.

Distinguishing features

9. Having regard to the analysis given in above points 3 to 3.4, the method of operative claim 1 differs from that of the closest prior art only in that it comprises additional steps (4) and (5).

Problem successfully solved

10. Having regard to the closest prior art, the appellant and the respondent take differing positions as to which problem can be considered to be successfully solved by the subject-matter of operative claim 1.

Whereas the appellant argues that the objective technical problem solved by the subject-matter of claim 1 over the closest prior merely resides in the provision of a further method for producing polyether polyols, since the comparative tests contained in the specification relied upon by the respondent would not support the technical benefit alleged by the respondent, the respondent submits that the additional use of steps (4) and (5) would result in an optimization of the particle size and particle size distribution of the crystals formed during the neutralization step.

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As shown below, even if to the benefit of the appellant, the problem successfully solved is formulated as the provision of a further method for producing polyether polyols, the claimed solution has not been shown to be obvious. Under these circumstances, the question of which of these problems can be considered to be successfully solved over the method exemplified in D1 can be left unanswered.

#### Obviousness

11. Even if the problem successfully solved is formulated as the provision of a further method for producing polyether polyols, none of the items of evidence relied upon by the appellant in combination with D1, namely D2, D3 and D4, describes or hints at a double dehydration step with an intercalated step in which at least part of the salt is redissolved by adding water to the dehydrated neutralized polyether polyol mixture obtained after the first dehydration step.

As shown in above point 4.1, D2 does not teach steps (4) and (5). Other passages of D2 have not been cited by the appellant in this respect.

Concerning D3, this document does not teach present step (4), as indicated in the second and third paragraphs of above point 5. The same holds true for D4, as shown in above points 6.1 to 6.3, which document teaches as an essential feature removal of the crystal formed after performing steps (1) to (3) and therefore does not suggest that crystal can be redissolved before performing step (5) and (6).

Accordingly, the teaching of D1 combined with that of any of D2, D3 and D4 does not result in a method

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comprising a step (4) as required by operative claim 1. Therefore, the objection that the method of claim 1 would be obvious starting from D1 as the closest prior art when seen in combination with any of D2, D3 or D4 does not convince.

Inventive step starting from the teaching of example 3 of D3 or examples 1 and 2 of D4  $\,$ 

- 12. The appellant objects that the method of operative claim 1 lacks also an inventive in view of example 3 of D3 taking into account the teaching of the example of D1, of example 1 of D2 or of example 1 or 2 of D4. The appellant's objection starting from example 1 or 2 of D4 as the closest prior art is made in the light of the overall teaching of D4 or of the above cited examples of D1, D2 or D3. However, as shown above in relation to the various objections of lack of novelty, none of these examples or even the whole document D4 describes step (4) of operative claim 1. Consequently, the additional inventive step objections raised by the appellant must also fail.
- 13. On that basis, the subject-matter of claim 1 of the granted patent involves an inventive step within the meaning of Article 56 EPC.
- 14. The appellant confirmed at the oral proceedings that only the objections of lack of inventive step dealt with above were maintained. Therefore no further objection is to be dealt with.

# Order

# For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated