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Case Number: T 2095/16 - 3.3.10

Application Number: 06732932.6

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IPC: C07C273/16, B01D53/94

Language of the proceedings: EN

Title of invention:
PROCESS FOR THE PREPARATION OF A UREA-COMPRISING AQUEOUS STREAM

Patent Proprietor:
Stamicarbon B.V.

Opponents:
YARA INTERNATIONAL ASA
CASALE SA

Headword:

Relevant legal provisions:
EPC Art. 56

Keyword:
Inventive step - (no)

Decisions cited:

Catchword:

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Case Number: T 2095/16 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 15 October 2019

Appellant: (Patent Proprietor)	Stamicarbon B.V. Mercator 3 6135 KW Sittard (NL)
Representative:	V.O. P.O. Box 87930 2508 DH Den Haag (NL)
Respondent: (Opponent 1)	YARA INTERNATIONAL ASA P.O. Box 2464 SOLLI 0202 Oslo (NO)
Representative:	Pappaert, Kris De Clercq & Partners Edgard Gevaertdreef 10 a 9830 Sint-Martens-Latem (BE)
Respondent: (Opponent 2)	CASALE SA Via Giulio Pocobelli 6 6900 Lugano (CH)
Representative:	Zardi, Marco M. Zardi & Co. SA Via Pioda 6 6900 Lugano (CH)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 14 July 2016 revoking European patent No. 1856038 pursuant to Articles 101(2) and 101(3)(b) EPC.**

Composition of the Board:

Chairman	P. Gryczka
Members:	R. Pérez Carlón
	F. Blumer

Summary of Facts and Submissions

I. The appeal lies from the decision of the opposition division to revoke European patent No. 1 856 038.

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

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

D7 News from Hydro, dated 2 December 2003: Hydro Agri and Brenntag conclude European agreement for the distribution of "AdBlue"

D7a From Internet Archive WaybackMachine https://web.archive.org/web/2003010073256/ http://brenntag.de/german/news/index.html and https://web.archive.org/web/20031219015546/ http://www.brenntag.de/german/news/artikel/2003/Bi_Hydro_Agri.htlm (German version of D7 with evidence for publication date)

D17 Kirk-Othmer Encyclopedia of Chemical Technology, Third Edition, Volume 23, Thyroid and Antithyroid Preparations to Vinyl Polymers, John Wiley & Sons Inc. 1983, pages 548-562

D18 Ullmann's Encyclopedia of Industrial Chemistry, Volume A 27, Fifth Edition 1996, VCH, pages 343-353

D19 Urea synthesis: a status report - I *Nitrogen* No. 185, May-June 1990, pages 22-29

IV. Claim 1 of the patent as granted, which is the main request of the appellant (patent proprietor), reads as follows:

"Process for the preparation of a urea-comprising aqueous stream, that is suitable for use in a unit for the reduction of NOx in combustion engine exhaust gases, ***characterized in that*** *the urea-comprising aqueous stream is separated directly from or after a recovery section in a urea production process and is thereafter diluted with water until the urea-comprising stream comprises 30-35 wt% urea."*

V. The opposition division concluded that the right to priority had not been validly claimed and that, consequently, document D8 could be considered for inventive step and in fact was the closest prior art. It disclosed a process for preparing AdBlue from urea melt. The problem underlying the claimed invention was to provide a method for producing a 30-35 wt% urea aqueous stream free of formaldehyde or other additives. The solution, characterised by diluting an aqueous stream separated directly from or after the recovery section, would have been obvious for the person skilled in the art, with the consequence that the claimed process was not inventive.

VI. With the grounds of appeal, the appellant filed auxiliary requests 1 to 3. Auxiliary request 4 was filed with a letter dated 19 February 2019.

Claim 1 of auxiliary requests AR1 and AR2 require the aqueous stream to be separated "*directly from*" a recovery section.

Claim 1 of auxiliary request AR3 requires the aqueous stream to be separated *"directly from or after a recovery section in a CO_2 stripping urea production process".*

Lastly, claim 1 of auxiliary request AR4 contains all the features of claim 1 of the main request and adds the following:

"*wherein the urea-comprising aqueous stream comprises 60-90 wt% urea*".

VII. The arguments of the appellant were as follows:

Claim 1 related to the preparation of urea solutions suitable for NO_x reduction in combustion engine exhaust gases fulfilling the requirements set by Vornorm V70070.

Document D7a was the closest prior art. It disclosed a process for preparing the urea solution "AdBlue" from "very pure urea", which was solid urea. The problem underlying the claimed invention was to provide an improved process for the preparation of a 30-35 wt% urea aqueous solution, both in terms of economical balance and the biuret content. The problem was solved by diluting an aqueous urea-comprising stream separated directly from or after a recovery section of a urea production plant. The skilled person would only have arrived at the claimed solution with the benefit of hindsight. Only solid urea was considered suitable for obtaining very pure products, biuret content was known to increase in solution, and urea solutions contained further impurities and were thus not suitable for the purpose of obtaining "AdBlue".

For these reasons, the process of claim 1 of the main request and auxiliary requests AR1, AR2 and AR4 was inventive.

At the oral proceedings before the board, the appellant

argued that the process of claim 1 of auxiliary request AR3 allowed obtaining a feed containing less ammonia, which was a problem not addressed by the authors of D7a. The claimed solution, characterised by diluting an aqueous stream separated from or after a recovery section in a CO_2 stripping urea process, was for this reason inventive.

VIII. The arguments of the respondents (opponents) were as follows:

If D7 was considered the closest prior art, the problem formulated by the appellant had not been credibly solved over the whole scope of the claimed subject-matter.

If, nevertheless, the problem had been solved, the claimed solution would have been obvious for the person skilled in the art having regard to the common technical knowledge, as reflected in documents D17, D18 and D19. The process of claim 1 of the main request was thus not inventive. The situation was the same with respect to the process of claim 1 of auxiliary requests AR1, AR2 and AR4.

Respondent 1 requested that the line of argument of the appellant with respect to the process of claim 1 of auxiliary request AR3 not be admitted into the proceedings as it was raised late, and the respondent was not prepared to react to it. Nevertheless, the respondents argued that it was generally known that carbon dioxide stripping reduced ammonia content in the prepared urea.

IX. Oral proceedings before the board of appeal took place on 15 October 2019.

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 or, alternatively, on the basis of any of auxiliary requests 1 to 4, auxiliary requests 1 to 3 as filed with the grounds of appeal dated 23 November 2016, auxiliary request 4 as filed with a letter dated 19 February 2019.

- Respondents 1 and 2 requested that the appeal be dismissed.

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

Reasons for the Decision

1. The appeal is admissible.

Inventive step

2. Claim 1 relates to a process for preparing a 30-35 wt% aqueous urea solution suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases.

The appellant argued that any urea solution would not be suitable for that use unless it were free from catalyst poison and an eutectic solution according to Vornorm V70070. One example of such a solution was generally known as "AdBlue". For this reason, the

limitations set by the Vornorm V70070 standard had to be fulfilled and were thus also features of claim 1, although not explicitly mentioned in the claim.

The board does not share this interpretation. The feature "suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases" does not require any specific level of efficiency or exclude catalyst poisons, let alone limit the aqueous urea solutions prepared by the claimed process only to those conforming to Vornorm V70070.

It was, however, not disputed that AdBlue is a urea solution obtainable by the process of claim 1. Since the board's conclusion on inventive step is negative even with respect to a process according to claim 1 for preparing AdBlue, there is no reason to further elaborate on other embodiments of this claim.

Claim 1 requires a 30-35 wt% urea solution to be prepared from an aqueous stream separated directly from or after a recovery section in a urea production process.

3. Closest prior art

The parties were divided as to which document was the closest prior art. In the following, it will be examined whether the claimed subject-matter is inventive considering D7a to be the closest prior art, in support of the appellant's view in this respect.

The parties were divided on what D7a disclosed.

According to D7a, AdBlue is prepared "direkt aus hochreinem Harnstoff aus dem Produktions-

prozess" (directly from very pure urea from the synthesis process). D7a does not further disclose what type of urea is used as the starting material.

The appellant argued that "very pure urea" could only mean solid urea. In favour of the appellant, inventive step will be examined under this assumption.

4. Technical problem underlying the invention

The appellant defined the technical problem underlying the claimed invention as providing an improved process for preparing AdBlue from an economical point of view, while keeping a low biuret content in the prepared urea solution.

5. Solution

The claimed solution to this technical problem is the process of claim 1, characterised by diluting an aqueous urea stream separated directly from or after a recovery section in an urea production process.

6. Success

The respondents considered the problem as formulated by the appellant not credibly solved by the claimed process.

However, the question of whether the problem as formulated by the appellant has been solved in all aspects can be left undecided since by even considering it to be credibly solved, the conclusion on inventive step is negative for the reasons that follow.

7. It was undisputed that industrial processes for the preparation of urea have the following sections:

- A urea synthesis section, in which ammonia and carbon dioxide react to form ammonium carbamate, which further decomposes to give urea and an equivalent of water.

- A urea recovery section, in which unreacted ammonium carbamate is decomposed into ammonia and carbon dioxide, which are recovered and recycled. The product of this section is a concentrated urea solution, containing ca. 10 to 25% water.

- An evaporation or crystallisation section, in which the aqueous solution is further concentrated to obtain a "urea melt".

- A shaping section, in which solid urea is obtained from the melt.

This knowledge is reflected for example in paragraph [0011] of the patent as granted and documents D17 and D18. This was common ground at the oral proceedings before the board.

8. In the appellant's support, the claimed solution would be considered to encompass only the dilution of a urea solution arising from the urea recovery section, which contains 60-90 wt% urea ([0020], claim 5).

9. To provide a process for the preparation of 30-35 wt% urea solutions, such as AdBlue, with better energy balance and less biuret, the skilled person would have directly diluted the urea aqueous solution arising from the recovery section for the reasons that follow.

Biuret content

Biuret is formed at the melting point of urea (D17, page 548, fourth full paragraph) with the concomitant obtention of an equivalent of ammonia. The production of biuret is hindered by the presence of ammonia in the reactor and is favoured downstream once ammonia is removed (D18, page 343, paragraph bridging both columns).

In the recovery section, unreacted ammonium carbamate is decomposed into ammonia and carbon dioxide, which are separated and recycled. At any point subsequent to this in the process, the concentration of ammonia is very low and no longer prevents biuret formation. It is common knowledge that biuret formation is minimised by minimising retention times of urea containing solutions at high temperatures (D18, above).

To reduce the handling of urea solutions at high temperatures, and thus to prevent biuret formation, the skilled person would have directly diluted the aqueous stream arising from the recovery section.

Economic balance

The claimed process seeks the production of an aqueous solution of urea of a defined concentration. The skilled person, attempting to optimise the economic balance of the process, would have realised that obtaining AdBlue from solid urea (D7a) required evaporation of water from a urea water solution, followed by water addition. Using an aqueous urea feed as the starting material thus would have been an obvious solution in order to reduce water and energy

consumption.

10. The skilled person seeking a method having improved economical balance which reduces biuret formation would thus have used, as the starting material, an aqueous feed obtained from the recovery section and would have arrived in this manner at the claimed invention.

The claimed process is therefore not inventive (Article 56 EPC).

11. The appellant argued that the claimed solution would only have become evident to the skilled person with the benefit of hindsight.

The board cannot accept this argument, as the reasons for taking the feed arising from the recovery section as the starting material are numerous and would have been within the basic knowledge of the person skilled in urea technology.

11.1 The appellant also argued that document D17 (page 562, "Finishing Processes") only disclosed "highly pure urea" in the context of solid urea. D17 only mentioned aqueous solutions in the context of agricultural applications, which did not require the stringent specifications of AdBlue. The skilled person was thus taught to use solid urea as the urea source when seeking to obtain a pure product.

However, document D17 was published in 1983, more than 20 years before the claimed invention was filed, and should be read in the context of the urea applications known at the time.

D17 applies the adjective "pure" in the context

"concentrated urea is solidified in essentially pure form as prills, granules, flakes or crystals".

The appellant acknowledged at the oral proceedings before the board that it was known before the publication of D17 that prills and granules contain additives such as formaldehyde, whereas flakes or crystals do not.

D17, however, does not make any distinction between prills, granules, flakes and crystals, despite having different degrees of additives, and thus of purity.

Contrary to the appellant's argument, D17 discloses neither that only solid urea is very pure nor that every solid urea is pure.

D17 further fails to disclose, as alleged by the appellant, urea aqueous solutions from the urea process to be unsuitable for applications in which purity could be an issue. D17 merely indicates that urea solutions, at that time, were used for agricultural purposes; nothing more, nothing less.

This argument is thus not convincing.

11.2 The appellant also argued that D17 disclosed in the passage "Finishing Processes" that "in the solid form, urea is more stable and biuret formation less likely". D18 disclosed (page 346, left column, second full paragraph) that "when urea with an extremely low biuret content is required (at a maximum of 0.3 wt%) pure urea crystals are produced in a crystallisation section". This taught the skilled person that only solid urea could have low amounts of biuret and thus led away from the claimed solution.

However, the product of the claimed process is an aqueous urea solution, which could decompose during storage and transport. If this solution is manufactured at the production site (D7a) shortly after manufacture, any effect due to enhanced storage stability of solid urea becomes irrelevant. This argument is also not convincing.

12. The appellant argued that document D17 discloses that water waste needed to be treated before discharging to eliminate ammonia (page 562, "Wastewater Treatment"). This showed that the concentration step did not merely separate water, but also other impurities. The skilled person would thus have considered the feed exiting the recovery section to be too contaminated to be used as the starting material for the process of claim 1.

However, the paragraph cited by the appellant relates to reducing ammonia content below the threshold set by the legislator for discharge (100 ppm). In contrast, Vornorm V70070 sets an upper limit of 0.2 wt% for ammonia (2000 ppm), which is 20 times higher. There is no evidence on file that the feed exiting the recovery section would have contained too much ammonia for the purposes of AdBlue production.

13. To the appellant's advantage, the board has considered claim 1 to be limited to the preparation of a urea solution according to Vornorm V70070 such as AdBlue, taken D7a as the closest prior art, read this document as disclosing the dissolving of solid urea, and assumed that the problem formulated by the appellant has been solved in all aspects and that the claimed solution did not include diluting an urea melt. Even under these assumptions, the claimed subject-matter is not

inventive (Article 56 EPC).

Inventive step, auxiliary requests

14. The appellant acknowledged that the conclusion on Article 56 EPC would be the same with respect to claim 1 of auxiliary requests AR1, AR2 and AR4.

15. The appellant argued, however, that claim 1 of auxiliary request AR3, which required diluting an aqueous stream separated directly from or after a recovery section in a CO_2 stripping process, was inventive as CO_2 stripping allowed further reducing the amount of ammonia in the stream. It relied in this respect on examples 1 of 2 of the patent as granted.

This argument was brought forward only at the oral proceedings before the board, and respondent 1 indicated that it was not prepared to address it.

Notwithstanding this, it is common general knowledge that CO_2 stripping processes provide a low ammonia and carbon dioxide stream after stripping (i.e. after the recovery section) as is confirmed by D18 (sentence bridging pages 344 and 345) and was acknowledged by the appellant at the oral proceedings before the board. The skilled person thus would have chosen a CO_2 stripping process for providing a stream containing little ammonia. Claim 1 of auxiliary requests AR3 is thus also not inventive (Article 56 EPC).

16. None of the requests on file is allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

C. Rodríguez Rodríguez P. Gryczka

Decision electronically authenticated