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Datasheet for the decision of 26 July 2010

Case Number: T 1237/08 - 3.3.01

Application Number: 00964775.1

Publication Number: 1224176

IPC: C07D 251/60

Language of the proceedings: EN

Title of invention:
Process for the preparation of melamine

Patentee:
DSM IP Assets B.V.

Opponent:
AMI Agrolinz Melamine International GmbH

Headword:
Preparation of melamine/DSM

Relevant legal provisions:
EPC Art. 56

Relevant legal provisions (EPC 1973):
-

Keyword:
"Main request: inventive step (no), obvious combination of prior art teachings"

Decisions cited:
-

Catchword:
-

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Case Number: T 1237/08 - 3.3.01

D E C I S I O N
of the Technical Board of Appeal 3.3.01
of 26 July 2010

Appellant: (Patent Proprietor)
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Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 22 April 2008 revoking European patent No. 1224176 pursuant to Article 101(2) EPC.

Composition of the Board:

Chairman: P. Ranguis
Members: L. Seymour
C.-P. Brandt

Summary of Facts and Submissions

I. European patent No. 1 224 176, which was filed as application number 00 964 775.1, based on international application WO 01/25221, was granted on the basis of seven claims, one of which was independent.

Independent claim 1 as granted reads as follows:

"1. Process for the preparation of melamine comprising a reaction step, a gas/liquid separation step in which a melamine melt is separated from off-gases, a stripping step and a cooling step, **characterised in that** the stripping step is operated at a pressure of between 5 MPa and 17 MPa and a temperature of between 330°C and 450°C and **in that** the melamine melt obtained in the preceding steps is pressurised in the cooling step to a pressure of between 15 MPa and 35 MPa, with the pressure in the cooling step being higher than the pressure in the stripping step and with the temperature in the cooling step being adjusted between the melting point of the melamine melt at the prevailing pressure and 365°C."

II. An opposition was filed and revocation of the patent in its entirety requested pursuant to Article 100(a) EPC, for lack of novelty and inventive step.

III. The following documents were cited *inter alia* during the opposition proceedings:
(1) WO 96/20182
(9) EP-A-0 808 836

IV. The appeal lies from the decision of the opposition division revoking the patent under Article 101(2) EPC.

The opposition division considered the subject-matter of claim 1 of the patent in suit to be a novel selection from the generic teaching of document (1). No specific disclosure could be found in document (1) of a single embodiment having all the features of claim 1 of the patent in suit.

With respect to the issue of inventive step, the opposition division identified document (1) as representing the closest prior art and defined the problem to be solved as lying in the provision of an alternative process resulting in melamine with a high purity. The opposition division considered that the proposed solution to said problem lacked an inventive step in view of document (1). In particular, examples 8 to 13 suggested carrying out the cooling step at a higher pressure and at a temperature above the melting point of melamine as a solution to the problem posed.

V. The appellant (patentee) lodged an appeal against this decision.

With its letter of response, the respondent (opponent) filed counterarguments.

Both appellant and respondent made conditional requests for oral proceedings.

VI. In a letter dated 21 May 2010, the appellant stated that it would not attend the oral proceedings scheduled for 14 September 2010.

The board informed the parties by letter of 15 July 2010 that the oral proceedings were cancelled.

VII. The appellant's arguments submitted in writing can be summarised as follows:

The appellant contested the opposition division's analysis with respect to inventive step.

In a first line of argument, the appellant defined the problem to be solved, starting from document (1) as closest prior art, as lying in the provision of an alternative process resulting in melamine with a high purity. In this context, the appellant identified several features that distinguished the processes disclosed in examples 1 to 5 according to document (1) from the subject-matter claimed in the patent in suit.

Firstly, the appellant emphasised that that document (1) could not be read as disclosing a stripping step. Thus, in step b of examples 1 to 5, the pressure was increased by at least 10 bar with respect to the pressure in the reactor, whereas the temperature was reduced by 5°C compared to the reaction temperature. This was a clear indication that the pressure had been increased by adding ammonia without removing any gas phase.

Secondly, in document (1) the cooling step was conducted at the same pressure as the treating step b, whereas according to claim 1 of the patent in suit the pressure in the cooling step was higher than the pressure in the stripping step.

Finally, in the cooling step of examples 1 to 5 according to document (1), the end temperature of 280°C was below the melting point of melamine at the prevailing pressure.

In the appellant's opinion there was no pointer in the prior art to modify the process of document (1) accordingly, in order to arrive at the process claimed in the patent in suit.

In a second line of argument, the appellant submitted that the problem to be solved could be defined in more ambitious terms as lying in the provision of a process for the preparation of melamine having a reduced content of oxygen-containing impurities. This problem had been solved by employing a higher pressure in the cooling step than in the stripping step. To support this submission, the appellant referred to the comparative data provided in paragraph [0048] of the patent in suit. Lower levels of oxygen-containing compounds had been obtained in examples 1 and 3, in accordance with the patent in suit, compared to those observed in examples A and B, respectively, which reflected the teaching of the examples of document (1). The appellant maintained that this effect was unexpected in view of the prior art.

VIII. The respondent's arguments submitted in writing can be summarised as follows:

The respondent contested the appellant's analysis that document (1) did not disclose a stripping step. In

particular, the respondent argued that in step b according to examples 1 to 5, melamine was treated with NH_3 at a pressure of 85 bar (8.5 MPa) for 15 minutes. In order to maintain a constant pressure over this period of time, it was evident that the ammonia gas introduced must simultaneously be removed from the system.

Furthermore, respondent disputed that the comparative data set out in the patent in suit demonstrated that the claimed process provided melamine having a reduced content of oxygen-containing impurities relative to that obtained according to the process of document (1). Thus, from a comparison of examples 1 and A, it could only be deduced that a lower content of oxygen-containing impurities could be obtained by lowering the pressure in the stripping step. This data did not, however, demonstrate that increasing of the pressure in the cooling step relative to that in the stripping step led to a positive effect.

The problem to be solved was therefore to be defined as lying in the provision of an alternative process for the preparation of highly pure melamine.

The respondent argued that the proposed solution to the above problem would have been obvious to the skilled person in view of the teaching of document (1) alone, or *inter alia* that of document (9), which disclosed the possibility of executing a cooling step at an increased pressure, and at a temperature of between 1 to 50°C above the solidification point of the mixture (see page 4, column 5, lines 1 to 13).

IX. The following requests were made in writing during the appeal proceedings:

The appellant (patentee) requested that the decision under appeal be set aside and patent be maintained as granted.

The respondent (opponent) requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. In view of the fact that the appellant waived the right to be heard in oral proceedings and the respondent requested oral proceedings only on condition that the board did not intend to allow its request (see points V, VI and IX above), the present decision could be taken without holding oral proceedings, based on the written submissions of the parties (Article 113(1) EPC).

3. *Novelty (Articles 52(1) and 54 EPC)*

The board is satisfied that the claimed subject-matter is novel over the cited prior art.

In the contested decision, the opposition division acknowledged the novelty of the subject-matter claimed. Moreover, in its written submissions during appeal proceedings, the respondent did not challenge the reasoning of the opposition division with respect to

novelty. Hence, no detailed reasoning in this respect is required.

4. *Inventive step (Articles 52(1) and 56 EPC)*

4.1 The board considers, in agreement with the opposition division, that document (1) represents the closest prior art. This document was also used as a starting point for assessing inventive step by both parties in the present appeal proceedings.

Document (1) relates to a process for the production of highly pure melamine (page 2, second paragraph; claim 1).

In a first step urea is converted in a reactor to melamine, NH_3 and CO_2. This reaction is generally conducted at a temperature of between 370 to 430°C and at a pressure of about 70 to 250 bar (7.0 to 25.0 MPa) (see page 1, first paragraph; page 3, first complete paragraph).

The CO_2/NH_3 gas phase is then separated off from the melamine liquid phase. Preferably the temperature and pressure are about the same as those in the reactor (see page 3, second complete paragraph; step a).

Following the gas separation, or at the same time, gaseous NH_3 can be introduced which reduces the amount of CO_2 dissolved in the melamine. Again, the temperature and pressure are preferably at about the same values as those in the reactor (see page 3, third complete paragraph; step b).

The liquid melamine is then optionally allowed to stand for some time in the presence of ammonia at a partial pressure of 50 to 400 bar (5.0 to 40.0 MPa). The pressure in this step may be set to a higher value than in the reactor. The temperature is at a value between the melting point of melamine and 430°C (see paragraph starting at the bottom of page 3; step c).

The liquid melamine is then cooled to a temperature of between 270 and 330°C, at a defined cooling rate. Like the preceding steps, this step is carried out in the presence of ammonia. The ammonia partial pressure is 50 to 400 bar (5.0 to 40.0 MPa), preferably about 70 to 200 bar (7.0 to 20.0 MPa). Again, a higher pressure than in the reactor may be employed (see page 4, first complete paragraph; step d).

In examples 1 to 5 according to document (1), the conversion of urea to melamine is performed at 375°C and at 70 to 75 bar (7.0 to 7.5 MPa). After separating off the off-gases (step a), the liquid melamine is treated with NH_3 for about 15 minutes at 370°C and 85 bar (8.5 MPa) (step b). The liquid melamine is then allowed to remain at this temperature and pressure for about 60 to 90 minutes (step c), and the liquid melamine is allowed to cool exponentially to 280°C by switching off the heating (step d). The reaction vessel is then depressurized and slowly cooled to room temperature. A similar reaction sequence is disclosed in example 7. In example 8, step c is omitted.

4.2 Contrary to the appellant's opinion, the board considers that step b according to document (1) is a stripping step. Thus, according to the patent in suit,

in order to carry out the stripping step, a stripping gas, normally NH_3, is passed through the melamine melt and strips it of dissolved CO_2 (see paragraph [0026]). Similarly, in step b according to document (1), the amount of dissolved CO_2 is reduced by introduction of gaseous NH_3 (see page 3, third complete paragraph). This passage therefore clearly discloses a stripping step, since no such reduction of dissolved CO_2 would be achieved without removal of NH_3 from the system. In addition, as correctly pointed out by the respondent, the constant pressure maintained in step b according to examples 1 to 5 of document (1) would only be observed if the ammonia gas introduced was also removed from the system.

Moreover, as outlined above under point 4.1, the stripping step b is specifically disclosed in examples 1 to 5 of document (1) as being performed at 370°C and 8.5 MPa. Therefore, although placed in the characterising portion of claim 1 of the patent in suit, it cannot be accepted that the feature "the stripping step is operated at a pressure of between 5 MPa and 17 MPa and a temperature of between 330°C and 450°C" constitutes a distinguishing feature with respect to the process of document (1).

The distinguishing feature of the present process can therefore only be seen in the second feature listed in the characterising portion of claim, namely, "the melamine melt ... is pressurised in the cooling step to a pressure of between 15 MPa and 35 MPa, with the pressure in the cooling step being higher than the pressure in the stripping step and with the temperature in the cooling step being adjusted between the melting

point of the melamine melt at the prevailing pressure and 365°C".

4.3 In view of the closest state of the art, it must now be determined which problem the claimed invention addresses and successfully solves.

The appellant has submitted a definition of the problem to be solved as lying in the provision of a process for the preparation of melamine having a reduced content of oxygen-containing impurities, and referred to comparative data in the patent in suit in support thereof (see point VII above).

However, according to the consistent case law of the boards of appeal, if comparative tests are chosen to demonstrate an inventive step with an improved effect, the comparison with the closest state of the art must be such that the effect is convincingly shown to have its origin in the distinguishing feature of the invention.

As explained above under point 4.2, the distinguishing feature of the present process is to be seen in the conditions employed in the cooling step.

In the comparative data referred to by the appellant, the conditions used in the comparative examples differ from those used in the examples according to the patent in suit with respect to the pressure employed in the stripping step, rather than in the cooling step (see patent in suit, paragraph [0048], Table 1, comparative examples A and B vs. examples 1 and 3, respectively). Therefore, based on the comparative data provided in

the patent in suit, it cannot be concluded that the conditions of the cooling step are at the origin of any reduction in content of oxygen-containing impurities.

Moreover, the pressure in the reaction, stripping and cooling steps of comparative experiments A and B are maintained at much higher levels than those employed in examples 1 to 5 according to document (1). It cannot therefore be accepted that these examples accurately reflect the teaching of the examples of document (1).

As is well established in the case law of the boards of appeal, alleged but unsupported advantages cannot be taken into consideration in respect of the determination of the problem to be solved.

Consequently, the board cannot accept that the claimed subject-matter plausibly solves the purported problem of providing a process for the preparation of melamine having a reduced content of oxygen-containing impurities.

4.4 The problem to be solved in the light of the closest state of the art, must therefore be reformulated in a less ambitious manner as lying in the provision of an alternative process for the preparation of highly pure melamine.

The solution as defined in claim 1 relates to a process characterised in that "the melamine melt ... is pressurised in the cooling step to a pressure of between 15 MPa and 35 MPa, with the pressure in the cooling step being higher than the pressure in the stripping step and with the temperature in the cooling

step being adjusted between the melting point of the melamine melt at the prevailing pressure and 365°C".

Having regard to the embodiments described in paragraph [0048] of the description of the contested patent, the board is satisfied that the problem has been plausibly solved.

4.5 It remains to be investigated whether the proposed solution is obvious to the skilled person in the light of the prior art.

As becomes evident from the analysis under point 4.1 above, document (1) already envisages that a higher pressure may be employed in the cooling step (step d) than in the stripping step (step b). However, the state of the melamine at the end of the cooling step is not specified in document (1). As pointed out by the appellant, at the final temperature of 280°C disclosed for the cooling step d according to examples 1 to 5 of document (1), melamine would be solid at the prevailing pressure.

The skilled person faced with the above-mentioned problem would have been aware of other documents relating to processes for the preparation of high-purity melamine, such as document (9) (see e.g. column 2, lines 10 to 14). In this document a process is disclosed comprising a reaction step (column 4, lines 32 to 37), a gas/liquid separation step in which a melamine melt is separated from off-gases (column 4, lines 47 to 54), and a stripping step (column 4, lines 37 to 44, 47 to 54; see also column 6, lines 23 to 29).

Document (9) further discloses that, after the melamine melt has been transferred to one or more mixing vessels, ammonia is added, and an ammonia pump is utilized to increase the pressure to about 10 to 100 MPa, preferably to about 20 to 90 MPa and more preferably above 30 MPa. The temperature in the mixing vessels is preferably reduced from the reactor temperature to about 1 to 50°C above the solidification point of the mixture. This cooling has the effect of converting the condensation products of melamine back into melamine (column 4, line 57 to column 5, line 17). It is noted that the same effect is disclosed for the cooling step in the patent in suit (see page 4, lines 28, 29).

Accordingly, document (9) discloses a cooling step performed under conditions that substantially overlap with those defined in claim 1 of the patent in suit. In addition, document (9) teaches that this step reduces the level of certain impurities. Therefore, it would be an obvious measure for the skilled person, seeking an alternative process for the preparation of highly pure melamine, to modify the process within the general teaching of document (1) according to the specific teaching of document (9).

4.6 Consequently, the appellant's main and sole request is rejected for lack of inventive step of claim 1 (Articles 52(1) and 56 EPC).

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

B. Atienza Vivancos P. Ranguis