Datasheet for the decision
of 9 November 2011

Case Number: T 0504/09 - 3.3.01
Application Number: 98923216.0
Publication Number: 986546
IPC: C07D 251/60
Language of the proceedings: EN
Title of invention: Crystalline Melamine
Patentee: OCI Nitrogen B.V.
Opponent: Borealis Agrolinz Melamine GmbH
Headword: Melamine/OCI
Relevant legal provisions: EPC Art. 100(b)
Relevant legal provisions (EPC 1973): -
Keyword: "Sufficiency of disclosure (no): product cannot be achieved over the whole range claimed without undue burden"
Decisions cited: -
Catchword: -
Case Number: T 0504/09 - 3.3.01

DEcision of the Technical Board of Appeal 3.3.01 of 9 November 2011

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Composition of the Board:
Chairman: C. M. Radke
Members: L. Seymour
L. Bühler
Summary of Facts and Submissions

I. European patent No. 0 986 546 was granted on the basis of nine claims, one of which was independent. Claim 1 as granted reads as follows:

"1. Multicrystalline melamine powder having the following properties:

- $d_{90}$: 50-150 $\mu$m; $d_{50} < 50$ $\mu$m
- bulk density (loose) 430-570 kg/m$^3$
- color APHA less than 17
- melamine: > 98.5 wt%
- melam: < 1 wt%"

II. The opponent sought revocation of the patent in suit pursuant to Articles 100(b) and 100(a) EPC (lack of novelty and inventive step).

The following document was cited inter alia during the opposition proceedings:

(1) WO 98/54160

III. The appeal lies from the decision of the opposition division revoking the patent. The decision was based on the claims as granted.

The sole ground for revocation was that the subject-matter claimed lacked novelty with respect to document (1), which was identified as constituting prior art under Article 54(3) EPC and Article 54(4) EPC 1973. In view of the additional experimental evidence submitted by the opponent with letter of 15 September 2008, the opposition division was satisfied that a
melamine powder having the claimed parameters relating to \( d_{90} \), \( d_{50} \) and bulk density was the inevitable result of the process according to the specific example disclosed in document (1).

IV. The patentee lodged an appeal against this decision. With its statement of grounds of appeal, the appellant filed additional experimental data in support of its contention that the claimed product properties would not be the inevitable outcome of the process according to document (1).

V. In its reply, the respondent (opponent) maintained its objection of lack of novelty with respect to document (1), and argued that the experimental data submitted by the appellant raised issues of sufficiency of disclosure.

VI. In a communication sent as annex to the summons to oral proceedings, the board stated *inter alia* that the issue of sufficiency of disclosure would be discussed at oral proceedings.

VII. With letter of 4 October 2011, the appellant filed an auxiliary request. This request differed from the main request (claims as granted) in the limitation of the range for bulk density in claim 1 to "450-550 kg/m\(^3\)". As a result, claim 7 was deleted, the subsequent claims renumbered and dependencies adjusted.

VIII. With letter of 20 October 2011, the respondent announced that it would not be attending oral proceedings.
IX. Oral proceedings were held before the board on 9 November 2011.

X. The appellant's arguments on sufficiency of disclosure, insofar as they are relevant to the present decision, may be summarised as follows:

The appellant emphasised that the claims of the patent in suit related to a melamine product having certain properties and not to a process for making melamine. It was known from the prior art that melamine of high purity could be obtained by spray drying at high pressure. However, the products had been found to have poor powder properties (cf. patent in suit, paragraphs [0004] and [0007]). By varying the process conditions, the appellant had found that, under the conditions disclosed in the example of the patent in suit, a particularly good product could be obtained. Thus, the present invention lay in the recognition that a product having a particle size distribution and density as claimed overcame the disadvantages of the prior art.

The requirements of sufficiency were fulfilled since a clear and detailed working example had been provided allowing the skilled person to obtain a product that was illustrative of the narrow ranges of claimed parameters.

Concerning the level of detail used to describe the process in the example of the patent in suit, the appellant conceded that certain parameters such as the pressure of the melamine melt, the nature of the spraying means, the outflow velocity of the melamine melt, the speed of drum rotation and rate of cooling
had not been specified. However, the appellant argued that appropriate settings for a given apparatus and scale could be determined by the skilled person without undue burden.

Similarly, the appellant acknowledged that the general disclosure of the patent specification indicated a wide variation in a number of possible process conditions. It was clear that not any combination of these would yield the desired product. It would be extremely difficult to provide more precise information, since the process parameters were all interlinked in determining the properties of the product obtained.

However, the appellant argued that, equipped with common general knowledge and the specific and general guidance of the patent in suit, the person skilled in the art would require no more than routine experimentation in order to adjust the process parameters so as to obtain a product as claimed. The effect of various process conditions on product properties, such as that of spraying velocity on particle size, were well known to the skilled person. Similarly, if the particles obtained were too large, the skilled person would consider increasing the temperature of ammonia release in order to reduce the extent of agglomeration. Further relevant information on how to modify reaction conditions was also provided in the description of the patent in suit. For example, it was disclosed in paragraphs [0021] and [0024] that residence time at high temperatures should be limited in order to avoid yellowing of the melamine. Paragraphs [0031] and [0032] taught how to prevent the formation of impurities.
The experimental data submitted with the statement of grounds of appeal had been filed in order to demonstrate that a product according to present claim 1 was not the inevitable outcome of reproducing the example according to document (1). In particular, the data demonstrated that the claimed product was not obtained at a number of different temperatures of ammonia release, namely, 25, 50, 100, 150 and 200°C. However, this did not put into question sufficiency of disclosure of the invention. It was more than likely that the claimed product would have been obtained at a temperature of ammonia release of 180°C, as used in the example of the patent in suit.

The appellant did not advance any additional arguments with respect to the auxiliary request.

XI. The respondent's arguments submitted in writing, insofar as they are relevant to the present decision, can be summarised as follows:

The process parameters disclosed in document (1) fell within those required by the patent in suit. This meant that the skilled person reworking the process according to document (1) should inevitably obtain the claimed subject-matter. However, the experimental data provided by the appellant with the statement of grounds of appeal demonstrated that, on repetition of the example according to document (1) using various temperatures of ammonia release of between 25 and 200°C, the product obtained did not in fact fall within the present claims. This threw doubt on whether the patent in suit disclosed the claimed invention in a manner
sufficiently clear and complete for it to be carried out by a person skilled in the art.

XII. The appellant (patentee) requested that the decision under appeal be set aside and that the case be remitted to the first instance for consideration of the remaining grounds of opposition, based on the claims as granted (main request) or, alternatively, on the auxiliary request filed with the letter of 4 October 2011.

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

XIII. At the end of the oral proceedings, the decision of the board was announced.

**Reasons for the Decision**

1. The appeal is admissible.

2. **Main request - Sufficiency of disclosure**  
   *(Article 100(b) EPC)*

2.1 The present invention as reflected in claim 1 relates to a multicrystalline melamine powder characterised by a number of different parameters:

   • particle size distribution: \(d_{90} \text{ 50-150 } \mu \text{m}, \ d_{50} < 50 \mu \text{m};\)
   • bulk density (loose): 430-570 kg/m\(^3\);
   • color APHA less than 17;
   • purity: melamine > 98.5 wt\%, melam < 1 wt\%.
2.2 In order to assess whether the requirement of sufficiency of disclosure is fulfilled in the present case, it must be assessed whether the patent in suit as a whole, that is, the claims and the description (including the example), makes available to the skilled person, in the light of his general common knowledge, all the information necessary for achieving the desired product in the whole range claimed without undue burden.

2.3 Concerning the general conditions to be employed in preparing the claimed multicrystalline melamine powder, the following is stated in paragraph [0020] of the patent in suit (emphasis added):

"The melamine melt, having a temperature between the melting point of melamine and 450°C, is sprayed via a spraying means into a cooling vessel. In the cooling vessel the melamine melt is cooled by means of an evaporating cooling medium in an ammonia environment at an ammonia pressure of 4.5-25 MPa, the melamine melt being converted into melamine powder having a temperature of between 200°C and the solidification point of melamine. The melamine powder is then cooled further to a temperature below 50°C, the powder mechanically agitating over at least part of the cooling range and being cooled directly or indirectly, the ammonia pressure being released at a temperature below 270°C."

Further details of this quenching process are given in paragraphs [0021] to [0024] and [0037] to [0049]. In particular, the preferred pressure in the cooling vessel is disclosed as being 6 to 11 MPa (paragraph [0038]).
2.4 This general disclosure is illustrated by means of a single specific procedure for the preparation of the claimed product, which reads as follows (see Example in patent in suit, paragraph [0051], emphasis added):

"Melamine melt having a temperature of 402°C is introduced, via a spraying device, into a high-pressure vessel and cooled with liquid ammonia which is likewise sprayed into the vessel. The temperature in the vessel is 296°C. The high-pressure vessel is designed as a rotating drum provided with a wall which can be cooled, and provided with a gas inlet. The ammonia pressure in the vessel varies between 8.6 and 12 MPa. After 1 minute the product is cooled to ambient temperature. The cooling step to 200°C took 7 minutes. When the melamine powder had a temperature of about 180°C, all the NH₃ was released and air was metered into the vessel. The end product has the following properties:

\[
\begin{align*}
    d_{90} &= 106 \, \mu m; \quad d_{50} = 38 \, \mu m \\
    \text{bulk density (loose)} &= 490 \, \text{kg/m}^3 \\
    \text{color (APHA)} &= 10 \\
    99.2 \text{ wt\% of melamine} \\
    0.4 \text{ wt\% of melam} \\
    < 0.2 \text{ wt\% of melem} \\
    \text{concentration of ammonia} &= 150 \, \text{ppm} \\
\end{align*}
\]

2.5 The appellant has argued that, starting from this example and using his common general knowledge and the guidance of the patent in suit, the skilled person would arrive at products having the properties as set out in the claims without undue burden (cf. above point X).
The board notes, however, that in the present case the skilled person is confronted with a very large number of process variables affecting the claimed parameters, such as, composition, pressure and temperature of the melamine melt, the spraying means, the outflow velocity, the temperature and pressure in the cooling vessel, residence times, the means of cooling (direct and indirect) and mechanical agitation, and the pressure of ammonia release (see patent in suit, paragraphs [0020] to [0049]).

The appellant has indicated certain general principles known to the skilled person as to how some of these process variables might affect particular product parameters. However, the appellant has also admitted that the above process parameters were all interlinked in determining the properties of the product obtained. Therefore, for the requirements of sufficiency of disclosure to be fulfilled, it is not sufficient to simply know how to vary certain of the above parameters in isolation. Rather, the patent in suit as a whole must place at the disposal of the skilled person all the information necessary to reliably achieve the desired product, that is, fulfilling all the claimed parameters in combination.

According to the established case law of the boards of appeal, even though a reasonable amount of trial and error is permissible when it comes to sufficiency of disclosure, the skilled person has to have at his disposal, either in the specification or on the basis of common general knowledge, adequate information leading necessarily and directly towards success.
through the evaluation of initial failures (see Case Law of the Boards of Appeal, 6th edition, 2010, point II.A.4.2).

2.6 In the present case, the appellant has acknowledged that the example reproduced under above point 2.4 already does not include all the process parameters required to obtain the disclosed product.

Moreover, as already mentioned (see above points 2.3 and 2.5), the description of the patent in suit only provides a very general indication of the process conditions to be applied, without specifying which of the many process conditions are critical in reliably providing all claimed parameters in combination and how these can be modified accordingly in case of failure.

2.7 This assessment is confirmed by the test report filed by the appellant with the statement of grounds of appeal, in order to demonstrate novelty with respect to document (1), which reads as follows (emphasis added):

"EXAMPLE ACCORDING TO D1 (98/54160):
Melamine melt having a temperature of 402°C is introduced, via a spraying device, into a high-pressure vessel and cooled with a liquid ammonia which is likewise sprayed into the vessel. The temperature in the vessel is 296°C. The high-pressure vessel is designed as a rotating drum provided with a wall which can be cooled, and provided with a gas inlet. The ammonia pressure in the vessel varies between 6.8 and 9.2 MPa. After 1 minute the product is cooled to ambient temperature. The cooling step to 200°C took 7 minutes."
Several experiments were performed according to D1 with different temperatures for the depressurization. The results are shown in the table below.

T(dep): temperature for the depressurization; FBD = bulk density (ASTM 1895)

<table>
<thead>
<tr>
<th>T(dep) (°C)</th>
<th>d₉₀ (micron)</th>
<th>d₅₀ (micron)</th>
<th>FBD (kg/m³)</th>
<th>color (APHA)</th>
<th>melamine (w%)</th>
<th>melam (w%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>&gt;400</td>
<td>&gt;100</td>
<td>n.a'*</td>
<td>15</td>
<td>99.3</td>
<td>0.4</td>
</tr>
<tr>
<td>50</td>
<td>&gt;400</td>
<td>&gt;100</td>
<td>n.a'*</td>
<td>15</td>
<td>99.3</td>
<td>0.4</td>
</tr>
<tr>
<td>100</td>
<td>67</td>
<td>15</td>
<td>400</td>
<td>14</td>
<td>99.5</td>
<td>0.4</td>
</tr>
<tr>
<td>150</td>
<td>63</td>
<td>15</td>
<td>400</td>
<td>15</td>
<td>99.5</td>
<td>0.4</td>
</tr>
<tr>
<td>200</td>
<td>60</td>
<td>13</td>
<td>390</td>
<td>19</td>
<td>99.4</td>
<td>0.4</td>
</tr>
</tbody>
</table>

n.a': not available due to presence of lumps in product".

2.8 Thus, these test runs were performed under conditions that fall well within the preferred conditions disclosed in the patent in suit (cf. above point 2.3). It can be seen from the above table that the disclosed reaction reliably provides a product having the required purity and particle size distribution. However, in all the entries, the bulk density does not fall within the limits of the claims. In the last entry of the table, the product also does not fulfil the requirements with respect to colour.

Moreover, it is noted that the conditions used in the last two entries of said test report are rather similar to those employed in the example according to the patent in suit (cf. above points 2.7 and 2.4, respectively, particularly the details emphasised in bold). From a comparison of the results obtained, it
must be concluded that slight changes in reaction conditions can affect the product properties significantly and in an unpredictable manner.

No information is provided in the patent in suit as to how the reaction conditions employed under point 2.7 could be modified in order to lead directly towards success through the evaluation of these failures, nor could the appellant establish that this could be achieved based on common general knowledge.

The appellant argued in this context that it was more than likely that, at a temperature of ammonia release as used in the example of the patent in suit, namely, 180°C, the claimed product would have been obtained. However, it can be seen from the table reproduced above under point 2.7 that the temperatures of depressurisation of 200 and 150°C yield very similar bulk densities. There therefore appears to be no basis for assuming that the result at 180°C would be any different. It cannot be derived from the patent in suit that the exact exemplified temperature of 180°C is in any way critical for obtaining the desired product, regardless of the remaining process conditions used. Indeed, according to the general part of the description, the temperature of depressurisation is merely required to be below 270°C (cf. above point 2.3).

2.9 In view of the above considerations, the board concludes that the patent in suit neither provides a reliable starting point in the form of an enabling specific embodiment, nor adequate instruction as to how to achieve a multicrystalline melamine powder over the whole area claimed. In this respect, the skilled person
is faced with the undue burden of employing a considerable amount of trial and error within a broad range of potential process parameters. In the absence of sufficient guidance, the patent in suit offers only an invitation to perform a research programme in order to find suitable ways of preparing the claimed product.

Consequently, the main request must be refused, because the ground for opposition according to Article 100(b) EPC prejudices the maintenance of the patent as granted.

3. **Auxiliary request - Sufficiency of disclosure**  
   (Article 100(b) EPC)

The auxiliary request mainly differs from the main request in a somewhat narrower range of values for the bulk density in claim 1 (cf. above point VII). The appellant did not advance any additional arguments in favour of the sufficiency of disclosure of this request. Indeed, the assessment presented above under point 2 applies to this request *mutatis mutandis*.

Consequently, the auxiliary request is also refused pursuant to Article 100(b) EPC.
Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:     The Chairman:

M. Schalow      C. M. Radke