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**Datasheet for the decision
of 22 April 2015**

Case Number: T 0997/11 - 3.3.05

Application Number: 05781446.9

Publication Number: 1807163

IPC: B01D9/00

Language of the proceedings: EN

Title of invention:

PROCESS AND APPARATUS FOR CARRYING OUT CRYSTALLIZATION

Applicant:

Nederlandse Organisatie voor toegepast-
natuurwetenschappelijk onderzoek TNO

Headword:

Crystallisation/TNO

Relevant legal provisions:

EPC Art. 56

Keyword:

Inventive step - (no) - no improvement over prior art -
obvious alternative

Decisions cited:

Catchword:



**Beschwerdekammern
Boards of Appeal
Chambres de recours**

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Case Number: T 0997/11 - 3.3.05

**D E C I S I O N
of Technical Board of Appeal 3.3.05
of 22 April 2015**

Appellant:
(Applicant)

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Decision under appeal:

**Decision of the Examining Division of the
European Patent Office posted on 22 November
2010 refusing European patent application No.
05781446.9 pursuant to Article 97(2) EPC.**

Composition of the Board:

Chairman G. Rath
Members: G. Glod
C. Vallet

Summary of Facts and Submissions

- I. The appeal lies from the decision of the examining division of 6 October 2010 to refuse European patent application No. 05781446 for lack of inventive step.
- II. The documents cited during the examination proceedings included the following:
- D1: WO-A-03 084631
D2: DE-C-39 20 999
- III. With the statement of grounds of appeal, the applicant (appellant) requested that a patent be granted on the basis of a request filed with the examining division on 28 January 2008.
- IV. In its communication under Article 15(1) of the Rules of Procedure of the Boards of Appeal (RPBA), the Board expressed its preliminary non-binding opinion that this request was not admissible under Rule 12(4) RPBA, since it again contained the apparatus claims that had been deleted during examination proceedings. In addition, it was found that the reasoning of the examining division concerning the process claims was still applicable.
- V. By letter dated 23 March 2015, the appellant submitted a new main request as sole request.

Claim 1 of the request is as follows:

"1. Reactive or anti-solvent crystallization process, which comprises a step wherein a liquid comprising a solvent and two or more compounds dissolved therein, is subjected to crystallization conditions in the presence of Dean vortices, which Dean vortices result from said

liquid flowing through a channel having at least one curvature, wherein said crystallization conditions comprises [sic] forming supersaturation conditions for said liquid by initiating a chemical reaction and/or addition of an anti-solvent."

VI. Oral proceedings took place on 22 April 2015.

VII. The arguments of the appellant can be summarised as follows:

The present invention encompassed steps of both nucleation and subsequent crystallisation as explained on page 3, line 30 to page 4 line 1, while D2 only disclosed nucleation in the presence of Dean vortices. It was implicit that the precipitation step was part of the process according to claim 1.

Cooling crystallisation was a much slower process than the types of crystallisation of the present invention.

Crystallisation was influenced by many factors and predicting crystallisation processes was extremely difficult. Many crystallisation processes were known. The skilled person was not expected to consider that a process beneficial for one type of crystallisation was also beneficial for other types. Therefore, the skilled person would not consider D2 to be suitable for crystallisation processes other than cooling. During cooling crystallisation, nucleation occurred at the wall of the crystalliser and it was unlikely that the presence of Dean vortices was advantageous in such a process.

In the present invention, both the formation of nuclei and crystals occurred in the bulk of the fluid.

D1 was directed to a batch crystallisation process on a surface. The growth zone 32 shown in figure 1A of D1 should not be regarded as a spiral but only as a long tubing. There was no disclosure of Dean vortices in D1. D1 related to turbulent flow, which was not the case in the present invention. D1 could not be combined with D2.

VIII. Requests

The appellant requests that the decision of the examining division be set aside and that a patent be granted on the basis of the sole request on file, submitted with the letter of 23 March 2015.

Reasons for the Decision

1. Article 56 EPC (inventive step)

1.1 Invention

The invention concerns a process for continuous crystallisation (see page 1, lines 1 and 2).

1.2 Closest prior art

D2 (DE-C-3920999) [the application in suit refers to DE-A-39 20 999; application in suit, page 1 line 26] can be considered to be the closest prior-art document, since it relates to a process for the continuous production of crystalline nitroguanidine. Crystals of nitroguanidine are continuously obtained by forming nitroguanidine seeds in an externally cooled spiral condenser. The uniform turbulence of the spiral flow leads to a narrow size distribution (D2: page 2, lines

58 to 61). Further crystallisation is effected by an agitated cascade process (D2: page 2, lines 62 and 63). The crystallisation of nitroguanidine from a mixture of solvent (DMF), nitroguanidine and water is described in example 1.

The crystallisation process of claim 1 of the sole request is only characterised by the formation of supersaturation conditions and does not include any step of precipitation and does not give any details on the properties of the crystals to be formed. It is certain that seed formation is part of the crystallisation process, as is also evident from the description (application: p.6, lines 12 to 14).

1.3 Problem

According to the application (page 1, lines 7 to 9), the problem underlying the application is to obtain particles with a narrow size distribution and controlled morphology.

1.4 Solution

As a solution to this problem the patent in suit proposes a process according to claim 1 characterised in that the process is a reactive or anti-solvent process, wherein the formation of supersaturation conditions includes the initiation of a chemical reaction and/or the addition of an anti-solvent.

1.5 Success of the solution

As to the success of the solution, figure 4 shows that crystals obtained with a a helix crystalliser (called "spiral" on the graph) are smaller in size

and have a narrower distribution than crystals obtained with the straight tube.

However, this comparison is in no relationship to the proposed solution (chemical reaction and/or the addition of an anti-solvent vs. cooling). The graph, however, shows that a narrow size distribution is obtained.

So, it is accepted that the problem formulated in the application, i.e. obtaining a narrow size distribution and controlled morphology, is indeed solved, but no improvement with respect to D2 can be accepted, since there is no example showing the advantage of initialising a chemical reaction and/or addition of an anti-solvent compared to cooling.

In fact, the problem is identical to the provision of an alternative process to the one of D2.

1.6 Obviousness

The question that remains to be decided is whether the proposed solution is obvious or not.

According to Rule 43(1) EPC, the claims shall define the matter for which protection is sought in terms of the technical features of the invention. This means that technical features not present in the claim cannot be considered as part of the invention and are not to be taken into account when assessing inventive step. In particular, this means that the expression "crystallisation" is not understood as covering both the nucleation step and growth step, but includes any individual step of a crystallisation process in line with the definition given on page 6, lines 12 to 17 of

the application. This is also in line with the fact that claim 1 does not include any limitation with respect to the size of the crystals that should be formed.

The formation of supersaturation conditions by different crystallisation methods including reactive or anti-solvent crystallisation processes is well-known in the art. D1 clearly teaches that antisolvent addition and chemical reaction are alternatives to cooling for generating supersaturation for the generation of nuclei as well as for growing the crystals (D1: page 5, lines 13 to 18; page 6, lines 10 to 14; page 8, lines 34 to 37; page 13, lines 32 to 35).

In addition, D1 teaches that the process is beneficial to both batch and continuous crystallisations (page 9, lines 21 to 23). It is also indicated that the process of D1 is beneficial to any process in which narrow particle size distribution and/or control of other crystal characteristics is desired (page 9, lines 34 and 35).

D1 even teaches that good radial mixing is advantageous for obtaining uniform growth (page 14, lines 1 to 4), which is completely in line with the teaching of D2 that discloses that the fluid conditions present in the spiral tube lead to a narrow size distribution of the nuclei and a uniform growth ("gleichmäßig wachsen") (D2: page 2, lines 58 to 60). Good radial mixing occurs when Dean vortices are present, which are formed in a spiral tube.

The key question is whether the skilled person would consider that the process of D2 that makes it possible to obtain a narrow size distribution of the crystals

via supersaturation due to cooling was also suitable for obtaining crystals via supersaturation due to a method different from cooling. As explained above, D1 clearly indicates that such is the case, so that the skilled person has a clear incentive to use the different methods disclosed in D1 as possible alternatives to cooling. He would at least have tried the methods for causing supersaturation disclosed in D1 as possible alternatives to cooling with a reasonable expectation of success.

In applying the teaching of D1 to D2, the skilled person would arrive at the subject-matter of claim 1. In that respect, it should be noted that claim 1 does not contain any limitations with respect to flow conditions (laminar vs. turbulent), does not exclude the presence of other process steps, in view of the wording "comprising", and does not restrict the meaning of the wording "crystallisation" with respect to nucleation, crystal growth and the type and size of the crystals to be obtained.

1.7 Conclusion

The subject-matter of claim 1 does not meet the requirements of Article 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



C. Vodz

G. Raths

Decision electronically authenticated