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# Datasheet for the decision of 19 March 2014

**Case Number:** T 0856/11 - 3.3.05

**Application Number:** 01970064.0

**Publication Number:** 1401563

**IPC:** B01J2/04, A61K9/16

**Language of the proceedings:** EN

**Title of invention:**
PROCESS FOR THE PRODUCTION OF MICRO AND/OR NANO PARTICLES

**Patent Proprietor:**
Micro & Nano Materials SAGL

**Opponent:**
Sociedad española de carburos metalicos, S.A.

**Headword:**
Microparticles-Nanomaterials/ SAGL

**Relevant legal provisions:**
EPC Art. 123(2), 56

**Keyword:**
Inventive step - non-obvious alternative

**Decisions cited:**

**Catchword:**

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**Beschwerdekammern**
**Boards of Appeal**
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**Case Number: T 0856/11 - 3.3.05**

# D E C I S I O N
# of Technical Board of Appeal 3.3.05
# of 19 March 2014

| | |
|---|---|
| **Appellant:** (Patent Proprietor) | Micro & Nano Materials SAGL<br>Viale S. Franscini, 1<br>6900 Lugano (CH) |
| **Representative:** | Schneider Feldmann AG<br>Patent- und Markenanwälte<br>Beethovenstrasse 49<br>Postfach 2792<br>8022 Zürich (CH) |
| **Respondent:** (Opponent) | Sociedad española de carburos metalicos, S.A.<br>C. d'Aragó 300<br>08009 Barcelona (ES) |
| **Representative:** | Oficina Ponti, SLP<br>C. Consell de Cent, 322<br>08007 Barcelona (ES) |
| **Decision under appeal:** | **Decision of the Opposition Division of the European Patent Office posted on 21 February 2011 revoking European patent No. 1401563 pursuant to Article 101(3)(b) EPC.** |

**Composition of the Board:**

**Chairman:** G. Raths
**Members:** J.-M. Schwaller
P. Guntz

## Summary of Facts and Submissions

I. The present appeal lies from the decision of the opposition division revoking European patent 1 401 563 on the grounds that claim 1 of the three requests then on file did not meet the requirements of Article 56 EPC.

II. The opposition division argued in summary as follows:

The closest state of the art was document:

D1: Nora Ventosa et al.: "*Depressurization of an Expanded Liquid Organic Solution (DELOS): A New Procedure for Obtaining Submicron- or Micron-sized Crystalline Particles*", Crystal Growth & Design, vol. 1, no. 4, pages 299-303 (2001).

This document disclosed neither the solubilisation of dense carbon dioxide in the liquid solvent in a saturation chamber loaded with high surface packing elements, nor the injection of the thus obtained solution through a thin wall injector into a precipitation vessel operated at a pressure near atmospheric pressure.

Starting from document D1, the technical problem was to be seen in the provision of an alternative process for preparing micro- or nanoparticles.

The use, on the one hand, of a high surface packing to increase adsorption and, on the other hand, of a nozzle to achieve depressurization and cooling of a solution to be granulated being known from document

D2: US 6 056 791,

the incorporation of these features into the process of document D1 was obvious for a skilled person seeking an alternative process.

The use of a nozzle to atomize a solution into small droplets required a drying operation with a warm gas to produce powders, the step of evaporating the droplets with a warm inert gas was "a necessary requirement of an obvious alternative". The opposition division concluded that the claimed subject-matter therefore did not meet the requirements of Article 56 EPC.

III. With its grounds of appeal dated 30 May 2011, the patentee (hereinafter "the appellant") contested the first instance decision and submitted two new requests in replacement of those underlying the decision. Independent claims 1, 11 and 15 of the main request read as follows:

"1. *A process for producing micro and/or nano particles of solids with a mean diameter ranging between 0,01 and 100 micrometers, the process comprising the steps of:*
*- the solubilisation of the solid in a liquid solvent or a mixture of liquid solvents, the liquid solvent or the mixture of liquid solvents having very low or zero solubility in carbon dioxide under conditions with a temperature between 30 and 100°C and a pressure between 50 and 240 bar;*
*- the solubization (sic) of dense carbon dioxide in the liquid solvent or the mixture of liquid solvents, the carbon dioxide being supercritical, wherein the solubization (sic) takes place in a saturation chamber (sat) loaded with high surface packings at process*

*conditions with a temperature value between 30 and 100°C and a pressure value between 50 and 240 bar;*
*- the injection of the thus obtained solution through a thin wall injector (lp) into a precipitation vessel (Pr) operated at a pressure value nearatmospheric (sic) pressure;*
*- delivering a flow of warm inert gas into the precipitation vessel to allow solid nano and/or micro particles to be formed by droplet evaporation; and*
*- the recovery of produced powders.*"

"*11. An apparatus for performing the process according to any of the preceding claims, comprising:*
*a saturator (Sat) charged with packing elements,*
*a precipitator (Pr),*
*a thin wall injector (lp) that connects the saturator (Sat) and the precipitator (Pr), for forming droplets,*
*a first pressure line (Lq) for delivering a liquid solution to a first heat exchanger (S3) for preheating the liquid solution to temperatures between 50 and 90°C, and subsequently to the saturator (Sat),*
*a second pressure line (Lg) for delivering dense carbon dioxide to a second heat exchanger (S2-1) for heating the dense carbon dioxide to temperatures between 40 and 90°C, and subsequently to the saturator (Sat), and*
*a third pressure line working at pressures near the atmospheric value for delivering an inert gas to a third heat exchanger (S1) for preheating the inert gas up to 100°C, and subsequently to the precipitator (Pr).*"

"*15. A powder containing micro and/or nano particles of solids produced by the process according to any of claims 1-7, the powder being amorphous or partially amorphous and partially crystalline.*"

IV. The opponent (hereinafter "the respondent") did neither respond to the grounds of appeal nor did it submit any request.

V. At the oral proceedings, which took place on 19 March 2014 in the absence of the respondent, the patentability of the claimed subject-matter was extensively discussed. After the discussion, the appellant filed an amended set of claims 1 to 11 as a main request with an amended claim 1 reading as follows (amendments in bold):

"1. *A process for producing micro and/or nano particles of solids with a mean diameter ranging between 0,01 and 100 micrometers, the process comprising the steps of:*
*- the solubilisation of the solid in a liquid solvent or a mixture of liquid solvents, the liquid solvent or the mixture of liquid solvents having very low or zero solubility in carbon dioxide under conditions with a temperature between* ***40*** *and* ***90****°C and a pressure between 50 and 240 bar;*
*- the solubi****li****zation of dense carbon dioxide in the liquid solvent or the mixture of liquid solvents, the carbon dioxide being supercritical, wherein the solubi****li****zation takes place in a saturation chamber (sat) loaded with high surface packings at process conditions with a temperature value between* ***40*** *and* ***90****°C and a pressure value between 50 and 240 bar;*
*- the injection of the thus obtained solution through a thin wall injector (lp) into a precipitation vessel (Pr) operated at a pressure value near atmospheric pressure;*
*- delivering a flow of warm inert gas of* ***up to 100°C*** *into the precipitation vessel to allow solid nano and/ or micro particles to be formed by droplet evaporation;*

*and*
*- the recovery of produced powders.*"

Dependent claims 2 to 7 represent specific embodiments of the process according to claim 1.

Apparatus claim 8 corresponds to apparatus claim 11 filed with the grounds of appeal and dependent claims 9 to 11 represent specific embodiments of the apparatus according to claim 8.

The product claim, which related to a powder, has been deleted.

VI. After closure of the debate by the chairman, the appellant requested that the decision under appeal be set aside and that the patent be maintained in an amended form on the basis of the claims according to the main request filed during the oral proceedings or, alternatively, according to the auxiliary request dated 30 May 2011.

## Reasons for the Decision

1. The appeal is admissible.

2. Relevant articles

Since the respondent was totally silent during the appeal proceedings, the only points to be discussed are

- the admissibility under Article 123(2) EPC of the amendments to claim 1,

- the ground for revocation of the patent by the first instance, i.e. inventive step (Article 56 EPC).

3. Main request - admissibility of the amendments

The amendment by which:

- the temperature ranges have been modified from "*between 30 and 100°C*" to "*between 40 and 90°C*" has a basis in claim 2 as originally filed;

- the flow of warm inert gas has been defined as being "*up to 100°C*" has a basis at page 7, lines 15 to 18 of the description as originally filed.

It follows that these amendments satisfy the requirements of Article 123(2) EPC.

4. Main request - inventive step

The board, applying the problem-solution approach, comes to the following conclusions:

4.1 The invention underlying the contested patent (see paragraph [0007]) concerns a process and an apparatus to perform atomisation assisted by carbon dioxide to produce nano- and micrometric particles.

4.2 Document D1 - that the appellant and the opposition division acknowledged as representing the closest state of the art - discloses a process for the production of micron- and submicron-sized crystalline particles comprising the following three steps (D1: page 299, right column, last paragraph to page 300, middle of the left column):

(i) Dissolution of the solute to be crystallised in a conventional solvent, e.g. an organic solvent, at atmospheric pressure and at a working temperature to form a solution with a solute concentration which is below the saturation limit.

(ii) Addition of a compressed fluid CF (e.g. $CO_2$) over the organic solution to obtain a volumetric expanded liquid solution, at the working temperature and at a high working pressure containing a given molar fraction of the CF. The solute concentration in this step must remain below the saturation limit in the expanded mixture of the conventional solvent and the CF.

(iii) Rapid reduction of the pressure of the expanded solution, from the working to the atmospheric pressure, through a non-return valve. During this depressurisation process, the evaporation of the CF from the volumetric expanded solution takes place producing a large, fast, and extremely homogeneous decrease of the solution temperature down to the final temperature. As a consequence, a pronounced and homogeneous increase of the supersaturation ratio over all the solution takes place and the phenomenon of catastrophic nucleation occurs causing the precipitation of submicron- or micron-sized crystalline particles with a narrow particle size distribution.

In the experimental section, the parameters influencing the yield and the characteristics of solid particles obtained through the above process were studied in detail for the crystallisation of the colorant solvent blue 35, using acetone as the solvent and $CO_2$ as the CF. The operational procedure and experimental conditions used in this study were as follows: A known volume of a

solution of the colorant in acetone, with an initial supersaturation ratio of 0.8, was loaded in the high-pressure vessel R1. A circulating water jacket was used to maintain the working temperature at 293 K (19.85°C) inside this vessel. The initial solution was then pressurized up to a working pressure of 5, 10 or 15 MPa, i.e. 50, 100 or 150 bar, by addition of a given amount of $CO_2$ through the top of the vessel R1. After leaving the system at the same conditions for 30 to 60 minutes to achieve a complete homogenisation and thermal equilibration, the solution was depressurised over the non-return valve to the atmospheric pressure. This abrupt pressure reduction produced a large solution temperature decrease. During the depressurisation, solid particles precipitated and were collected on a filter located inside the precipitation vessel R2. During this step, the pressure of the solution inside R1 was maintained constant by a continuous addition of pressurised nitrogen gas from the top of the high-pressure vessel. After filtration, the cleaning of the precipitate was carried out with pure liquid $CO_2$ at 6 MPa and 293 K. The median diameter of the particles thus produced was found to be between 0.5 and 3.9 microns (Table 1).

D1 does not disclose the use of supercritical carbon dioxide nor the delivery of a flow of warm inert gas to the precipitation vessel. The use of surface packings inside the saturation vessel is also not disclosed.

4.3 The problem underlying the contested patent is defined as residing in the provision of a process for producing smaller particles while using practically all liquid solvents and while controlling the distribution and the mean size of the particles.

4.4 As a solution to this problem, the contested patent proposes the process according to claim 1 at issue, which is in particular characterised in that:

- the process conditions (temperature between 40 and 90°C; pressure between 50 and 240 bar) are such that the carbon dioxide is supercritical;

- the saturation chamber is loaded with high surface packings;

- a flow of warm inert gas is delivered at up to 100°C into the precipitation vessel to allow droplet evaporation.

4.5 As to the success of the solution, the examples in the patent show that with the claimed process it is possible to produce particles with mean diameters between 0.2 microns (example 1) and 2.5 microns (example 2). This range of diameter is similar to the one disclosed in document D1. Furthermore, there is no evidence in the patent that all liquid solvents might be used with the claimed process or that the distribution and the mean particle size of the particles can be controlled in a better manner than or in a way different from the one used in D1. It follows that the problem is to be reformulated in less ambitious terms, namely the provision of an alternative process.

4.6 As to the question whether the solution proposed by the contested patent is obvious or not from the state of the art, in particular document D2, the board notes that D2 concerns a different technique, which involves a solventless precipitation method and which requires the rapid cooling of the sprayed melted solid, and so

it is questionable whether the process steps in D2 are combinable with those in D1.

Even if, for the sake of argumentation, the content of these documents could be combined, the board is of the opinion that the skilled person looking for a process alternative to the one known in document D1 would not arrive in an obvious manner at the subject-matter of present claim 1, because in contrast to documents D1 and D2, the claimed subject-matter does not require a cooling step during atomisation, but a step in which warm inert gas is delivered into the precipitation vessel to induce production of the solid material during its solidification by drying. This step is, in the board's view, not obvious since this would be against the sense of a cooling step, as in documents D1 and D2.

For the board, the remaining documents also do not contain any information which would point towards the claimed solution of the problem stated above.

For the same reasons as those indicated above, the subject-matter of independent apparatus claim 8, which includes all the essential features of independent claim 1, is also not derivable in an obvious manner from the state of the art, and so it involves an inventive step, too.

Dependent claims 2 to 7 and 9 to 11 derive their patentability from independent claims 1 and 9 on which they depend respectively.

4.7 It follows from the above considerations that claims 1 to 11 of this request meet the requirement of Article 56 EPC.

4.8 The main request is therefore allowable.

**Order**

**For these reasons it is decided that:**

1. The contested decision is set aside.

2. The case is remitted to the department of first instance with the order to maintain the patent on the basis of the claims 1 to 11 of the main request of 19 March 2014 and a description to be adapted.

The Registrar: The Chairman:

C. Vodz G. Raths

Decision electronically authenticated