

**Internal distribution code:**

- (A)  Publication in OJ  
(B)  To Chairmen and Members  
(C)  To Chairmen  
(D)  No distribution

**Datasheet for the decision  
of 28 November 2006**

**Case Number:** T 0806/02 - 3.3.05

**Application Number:** 95901459.8

**Publication Number:** 0681557

**IPC:** C01B 15/10

**Language of the proceedings:** EN

**Title of invention:**

A method for preparing sodium percarbonate particles with improved stability

**Patentee:**

KEMIRA OY

**Opponent:**

Degussa AG

**Headword:**

-

**Relevant legal provisions:**

EPC Art. 123(2), 100(b), 83, 54, 56

**Keyword:**

"Sufficiency of disclosure (yes)"

"Novelty, inventive step (yes)"

**Decisions cited:**

G 0010/91, T 0012/81, T 0401/94, T 0211/93, T 0175/86,  
T 0557/04

**Catchword:**

-



Case Number: T 0806/02 - 3.3.05

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.05  
of 28 November 2006

**Appellant:** Degussa AG  
(Opponent) Benningssenplatz 1  
D-40474 Düsseldorf (DE)

**Representative:** Hartz, Nikolai  
Wächtershäuser & Hartz  
Patentanwälte  
Weinstrasse 8  
D-80333 München (DE)

**Respondent:** KEMIRA OY  
(Patent Proprietor) PL 44  
SF-02271 Espoo (FI)

**Representative:** Setna, Rohan P.  
Boult Wade Tennant  
Verulam Gardens  
70 Gray's Inn Road  
London WC1X 8BT (GB)

**Decision under appeal:** Interlocutory decision of the Opposition  
Division of the European Patent Office posted  
29 May 2002 concerning maintenance of European  
patent No. 0681557 in amended form.

**Composition of the Board:**

**Chairman:** M. Eberhard  
**Members:** H. Engl  
H. Preglau

## Summary of Facts and Submissions

I. European patent EP 0 681 557 B1 was maintained in amended form by a decision of the opposition division posted on 29 May 2002, on the basis of the third auxiliary request filed during oral proceedings.

The only independent claim of said third auxiliary request was worded as follows:

"1. A method for preparing sodium percarbonate particles coated with a first layer of sodium bicarbonate and a second coating layer on top of said first layer, characterized in that the method comprises:

a) the step of spraying an aqueous coating agent solution in a fluidized bed on the surface of the sodium percarbonate particles by using carbon dioxide, or a gas rich in carbon dioxide, as a fluidizing gas, producing a reaction on the surface of the sodium percarbonate particles to form said first layer on the surface of the sodium percarbonate particles, and said second coating layer on top of the first layer, wherein at least a part of said first layer is generated from the sodium carbonate of the sodium percarbonate particles and from the carbon dioxide in the gaseous phase, or

b) a first step of spraying water or an aqueous coating agent solution in a fluidized bed on the surface of the sodium percarbonate particles by using carbon dioxide, or a gas rich in carbon

dioxide, as a fluidizing gas, producing a reaction on the surface of the sodium percarbonate particles to form said first layer on the surface of the sodium percarbonate particles, and optionally a second coating layer on top of the first layer, and a second step of spraying an aqueous coating agent solution in a fluidized bed on the surface of the product from the first step, by using carbon dioxide, or a gas rich in carbon dioxide, or any other gas, as a fluidizing gas, to form an additional coating layer on the surface of the product from the first step, wherein at least a part of said first layer is generated from the sodium carbonate of the sodium percarbonate particles and from the carbon dioxide in the gaseous phase."

II. The following documents were *inter alia* cited during the opposition procedure:

- D3: DE C 28 10 379
- D5: "Eidesstattliche Versicherung" by Mr Heßberger, dated 17 December 1999
- D6: DE A 24 17 572
- D8: US A 3 864 454
- D9: Statutory Declaration by Mr T Södervall, dated 7 July 2000
- D10a: "Eidesstattliche Versicherung" by Mr Heßberger, dated 18 February 2002
- D10b: Statutory Declaration of Mr P Pekonen, dated 16 January 2002

- III. The opposition division held that the method of claim 1 of the main request and of the first and second auxiliary requests lacked novelty with respect to document D3. The claims of the third auxiliary request were considered patentable. The opposition division granted the patentee the benefit of doubt in view of contradictory experimental evidence and accepted that the first coating comprising sodium bicarbonate led to an additional stabilisation of the sodium percarbonate particles.
- IV. An appeal was filed against this decision by the opponent (henceforth: the appellant) on the grounds that claim 1 of the opposed patent (in the version as maintained) violated Article 123(2) EPC; that the patent did not satisfy the requirements of Article 83 EPC; and that the claimed subject matter lacked novelty and inventive step. A new test report, namely D12: "Eidesstattliche Versicherung" by Mr K Zimmermann, dated 17 Oct 2002, was filed with the statement of the grounds of appeal. In a further letter, the appellant relied on "Ullmann's Encyclopedia of Industrial Chemistry", VCH Verlagsgesellschaft, Weinheim, Germany, 1990, Vol. B1, pages 1-133 and 1-134.
- V. In a communication annexed to the summons to oral proceedings the board informed the parties of its provisional opinion on the issues under dispute.
- VI. VIn reply thereto the appellant submitted new arguments and a test report
- D15: 10 pages of Raman spectra annexed to the letter dated 23 October 2006.

Two additional documents were cited by the appellant in further letters, in particular

D16: "Ullmann's Encyclopedia of Industrial Chemistry", VCH Verlagsgesellschaft, Weinheim, Germany, 1988, Vol. B3, page 18-1.

VII. The respondent filed an auxiliary request on 27 October 2006.

VIII. Oral proceedings took place on 28 November 2006. The respondent filed two new documents, in particular a sheet (D12a) containing graphs based on data contained in D12.

IX. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

The respondent requested that the appeal be dismissed (main request) or in the alternative that the decision under appeal be set aside and the patent be maintained on the basis of claims 1 and 2 as filed with letter of 27 October 2006 (auxiliary request).

X. The appellant's arguments may be summarized as follows:

Objections under Article 123(2) EPC:

The wording "a first layer of sodium bicarbonate" was objected to by the appellant as going beyond the original disclosure of claims 1, 2 and 5 which contained the expression "a layer containing sodium bicarbonate".

Furthermore, it was argued that the description as published in WO A 95/15291 would disclose a causal connection between the reaction on the surface of the sodium percarbonate particle and the production of a layer of sodium bicarbonate which was missing in amended claim 1 of the main request.

Another objection concerned allegedly added subject matter in subpart a) of claim 1 of the main request. The claimed combination of features of step a) relating to a process for producing a first layer of sodium bicarbonate and a second layer was nowhere disclosed in the application documents.

Sufficiency of disclosure:

According to the appellant, the scope of the claims was limited to processes which actually produce sodium percarbonate particles coated with a first layer of sodium bicarbonate. However, it was impossible to ascertain whether a said first layer was formed or not by the claimed process. The appellant argued that the presence of said first layer could not be detected directly, for instance not by the Raman spectroscopy carried out on sample nr. 2.2 of D5. It could also not be deduced indirectly from comparative stability tests, contrary to what was suggested in the opposed patent itself. In any event, the stability tests on file (D5, D9, D10a, D12 and the examples in the patent in suit) would not conclusively demonstrate an improvement in stability against decomposition.

The appellant relied in particular on experimental reports D5, D10a and D12 to show that a coating process carried out in accordance with the claimed process, using CO<sub>2</sub> or a gas rich in CO<sub>2</sub> as a fluidizing gas, produced sodium percarbonate particles whose stability did not differ significantly from a comparison product produced in air. Data based on D12 were analysed statistically in order to show that the error in determining the oxygen loss upon storage for 2 and 4 weeks was in the same order as the alleged effect of the sodium bicarbonate layer. The claimed benefit of the invention could thus not be achieved. Evidently, the stabilizing sodium bicarbonate layer was ineffective or missing. Indeed, the data of Raman spectroscopy filed in report D15 showed that the claimed formation of a bicarbonate layer had in effect not occurred.

The appellant concluded that the skilled person was unable to decide whether a particular embodiment fell within the definition of the claims; furthermore, that the description did not enable the skilled person to achieve the claimed effects in terms of an increase in the product stability.

Since the presence of the first layer of sodium bicarbonate could not be detected analytically or in any other way, it was impossible to ascertain whether in a product obtained in accordance with the claimed process this essential feature of the claims was present or not. The claimed process was partly defined by the product to be produced, so that this deficiency would also affect the claimed process. The appellant



further made reference to decision T 557/04 (not published in OJ EPO).

#### Concerning novelty

Novelty was disputed having regard to document D3. Coating of sodium percarbonate particles in a fluidised bed was disclosed in D3, together with a limited list of gases to choose from. The appellant argued that the selection of the fluidizing gas would not be independent from the selection of the coating apparatus because the use of a fluidized gas was necessary. Therefore, he could not recognise a situation of choosing from several lists.

#### Concerning inventive step

The appellant argued during oral proceedings on lack of inventive step having regard to D3 alone. The only potentially novel element consisted of a novel combination of the features of D3. Inventive step would in such a case depend on the achievement of an effect. Since neither in the patent itself nor in the other experimental evidence on file an effect had been demonstrated in a statistically sound manner, any combination of the features disclosed in D3 would be equally obvious and an inventive step could not be acknowledged.

Under a second approach, the claimed process was obvious having regard to a combination of document D6 as the closest prior art and document D3. Said document D6 disclosed a process for producing stabilised sodium percarbonate, the process parameters disclosed in

Table 1 thereof would resemble those of the claimed process except for the use of CO<sub>2</sub> or a gas rich in CO<sub>2</sub> as a fluidizing gas. The apparatus and the technique used for the coating were not critical, therefore advantageous variations would be considered by the skilled person. In accordance with D6, the fluidized bed was operated at an elevated temperature of between 50 and 60°C (see Table 1) so that the fluidizing gas had to be heated. It was part of the skilled person's general knowledge that in direct heating the simplest method was the use of hot combustion gases which always contained carbon dioxide, as shown by D16. The skilled person would furthermore be aware from D3 that carbon dioxide could be used as a fluidizing gas.

As a still further alternative, the claimed process was obvious having regard to a combination of documents D6 and D8.

XI. The respondent essentially argued as follows:

As regards the alleged insufficiency of disclosure, the respondent stressed that the appellant had been able to repeat the method according to the invention. The objection was thus unfounded.

The respondent disagreed with the conclusions drawn by the appellant from D12. He noted that the decrease in O<sub>2</sub> content after four weeks was 4.5% for the product prepared in air, but only 3.6% for the product prepared under CO<sub>2</sub>. Therefore, D12 did in fact support the claimed invention.

As regards test reports D5 and D10a, these tests had not been made in comparison with the prior art, but using information contained in documents

D13: WO 95/06615 A (first sheet thereof only) and  
D14: WO 97/19890 A (first sheet thereof only). As D13 and D14 were both published after the priority date, these tests were not relevant.

The respondent relied on D9 (in particular on the comparison of samples 400 and 407) to demonstrate the effect of the invention in terms of improved stability against oxygen loss.

On novelty, the respondent argued that document D3 clearly indicated the use of carbon dioxide only under inert conditions, that is, conditions where no reaction with the sodium percarbonate would occur. Accordingly, the method disclosed in D3 would not result in formation of a sodium bicarbonate coating, but of a glassy silicate film. Moreover, the skilled person had to choose from the variables disclosed in D3 combining several separate items to arrive at the claimed invention which would not be permissible to argue lack of novelty. The respondent also argued that a sodium silicate solution sprayed on the percarbonate particles in a first process step would form a dense coating of silica and thus prevent any reaction between carbon dioxide and the percarbonate particles.

The claimed subject matter also involved an inventive step. The product resulting from the claimed process did not rely on the additives used in D3 which are either harmful to the environment, such as sodium perborate, or disadvantageous for rapid dissolution,

such as silicates. The respondent defined the technical problem as providing an alternative method for producing stabilized and moisture - protected sodium percarbonate particles. The process steps recited in claim 1 were not derivable from D3 or any other cited prior art in an obvious manner.

Document D6 disclosed stabilizing coatings different from those of D3 or the opposed patent. It did not suggest using carbon dioxide as a fluidizing gas and there was no incentive to combine it with D3.

Document D8 concerned a different technical field which the skilled person would not have considered in view of the technical problem of the invention.

### **Reasons for the Decision**

1. The appeal is admissible.
2. *Amendments (main request)*
  - 2.1 Claim 1 of the main request is based on the PCT application documents published as WO A 95/15291, in the following manner:

The pre-characterising part of the claim is directly and unambiguously derivable from claim 1 of the PCT application in combination with the description, page 2, lines 28 - 31 and page 3, lines 22 - 28, the latter passage disclosing the presence of a second, additional coating on top of the first coating.

The features "producing a reaction on the surface of the sodium percarbonate particles" and "wherein at least a part of said first layer is generated from the sodium carbonate of the sodium percarbonate particles and from the carbon dioxide in the gaseous phase" in both variants a) and b) find a basis in the description, page 2, lines 28 - 30, and page 3, lines 17 - 20. The board considers that it is clear in the context of the description that the said passage on page 3 applies likewise to variants a) and b) described in the preceding paragraphs, the description containing no indication of the contrary.

The fluidized bed spray process is disclosed in original claims 4 and 5 and in the description. In particular, variant a) of amended claim 1 is based on the description, page 3, lines 9 - 15, and on examples 10 and 15. Variant b) is based on the description, page 2, line 34 - page 3, line 7, and on example 12.

- 2.2 The feature "*a first layer **of** sodium bicarbonate*" has been objected to by the appellant as going beyond the original disclosure of claims 1, 2 and 5 which contained the expression "*a layer **containing** sodium bicarbonate*" (emphasis added by the board). This amendment was already present in claim 1 as granted. The appellant has not raised an objection under Article 100(c) EPC during opposition procedure; nor was the matter taken up by the opposition division on its own motion. The objection is therefore considered to be a fresh ground of opposition. According to G 10/91 (OJ EPO 1993, page 420, point 3 of the opinion), fresh grounds of opposition may be considered in appeal proceedings only with the approval of the patent

proprietor. Said approval was not given, so that the board has no power to examine this objection further. The appellant has referred to paragraph 19 of the reasons of G 10/91 stating that *"in case of amendments of the claims or other parts of a patent in the course of opposition or appeal proceedings, such amendments are to be fully examined as to their compatibility with the requirements of the EPC (e.g. with regard to the provisions of Article 123(2) and (3) EPC)"*. He argued that the objection here in question would arise out of the two other amendments concerning the features mentioned before, namely *"producing a reaction on the surface of the sodium percarbonate particles"* and *"wherein at least a part of said first layer is generated from the sodium carbonate of the sodium percarbonate particles and from the carbon dioxide in the gaseous phase"*, so that the patentee's approval would not be required. However, the board does not share the appellant's opinion, because the question already arose in the claims as granted and is thus independent of any subsequent amendments of the claims. An allegedly missing causal relationship between said feature *"a first layer **of** sodium bicarbonate"* and the feature *"producing a reaction on the surface of the sodium percarbonate particles"* discussed in the following subsection, has no bearing on the fact that the objection against the feature could have been raised in the opposition procedure.

- 2.3 The appellant furthermore argued that the description on page 2, penultimate paragraph, disclosed a causal connection between the reaction on the surface of the sodium percarbonate particles and the production of a

layer of sodium bicarbonate. He argued that this connection was missing in claim 1.

The board notes that during the opposition procedure claim 1 had been amended to include the feature "*producing a reaction on the surface of the sodium percarbonate particles*", which is followed by the phrase "*to form said first layer on the surface of the sodium percarbonate particles*" and the feature "*wherein at least a part of said first layer is generated from the sodium carbonate of the sodium percarbonate particles and from the carbon dioxide in the gaseous phase*", which explains what kind of reaction occurs at the surface of the particles to generate at least partly the first layer of sodium bicarbonate.

The added features read in combination with the remaining features of the claim, in particular with the preamble, which refers to a first layer of sodium bicarbonate, thus clearly and unambiguously establish a causal connection between the reaction on the surface of the sodium percarbonate particles and the production of the layer of sodium bicarbonate. Therefore, in the board's view, the claim does not encompass embodiments where sodium bicarbonate is being formed solely by other processes, for instance by a conventional coating process.

- 2.4 Another objection raised by the appellant in his letter dated 2 July 2004 (point 15) concerns allegedly added subject matter in subpart a) of claim 1 of the main request. More specifically, it was said that the claimed combination of features of step a) relating to a process for producing a first layer of sodium

bicarbonate and a second layer was nowhere disclosed in the PCT application WO A 95/15291.

However, as already stated above, said features of the claim are to be found in WO 95/15291 in claims 1 and 5 and on page 3, lines 17 - 20 and page 2, lines 28 - 32 of the description. The feature relating to the second coating disclosed in the description, page 3, lines 22 - 28, clearly applies to the embodiment of variant a) for producing a first sodium bicarbonate coating as described on the same page 3, lines 9 - 15, the description containing no suggestion of the contrary. The additional coating is applied as "*an optional method of the invention*" (see page 3, line 22), wherein the expression "*invention*" clearly refers to variants a) and b).

2.5 Dependent claims 2 and 3 are based on claims 2 and 3 and the description, page 3, lines 22 - 28, of the PCT application. Claim 4 is based on claim 7 of the PCT application.

2.6 The board therefore considers that the claims of the main request meet the requirement of Article 123(2) EPC.

2.7 Since claim 1 contains further limiting features compared with the claims as granted, the requirement of Article 123(3) EPC is also met.

### 3. *Insufficient disclosure (Articles 100(b) and 83 EPC)*

3.1 The appellant's objection is mainly based on the argument that the presence of the layer of sodium bicarbonate, which is produced by spraying water or an



aqueous coating agent solution in a fluidized bed using carbon dioxide, or a gas rich in carbon dioxide, as a fluidizing gas, onto the sodium percarbonate particles, cannot be detected analytically or by any other, indirect means. This layer of sodium bicarbonate is a key feature of the invention, as is set forth in the patent in suit, paragraph [0012]. According to the appellant, it is thus impossible to ascertain whether in a product obtained in accordance with the claimed process an essential feature of the claims is present or not. Since the claimed process is partly defined by the product to be produced, this deficiency would also affect the claimed process.

3.2 There exist several proposals as to how the presence of the first sodium bicarbonate layer on the sodium percarbonate particles could be ascertained. The method proposed in the patent in suit, paragraph [0021], in particular lines 25 to 30 thereof, involves analytic determination of the active oxygen content of the sodium percarbonate before and immediately after coating (without storage), the observed reduction being attributed to the formation of the sodium bicarbonate layer. Since, however, as the appellant has rightfully argued, it cannot be excluded that loss of active oxygen occurs during the coating process for other reasons, for instance through partial decomposition of the sodium percarbonate particles, it does not appear to the board that this method can be relied on for assessing whether a coating has formed.

3.3 A direct method was employed in the experimental report D15 filed by the appellant. This report, which was filed within the time period set out in the board's

communication, can be considered as a response to the questions raised by the board in the said communication. Its filing thus does not constitute an abuse of the procedure, contrary to the respondent's allegation.

Said report D15 consists of Raman spectra taken on a surface of a cross-sectional fracture of sodium percarbonate particles prepared in accordance with sample 2.2 of document D5. Said sample 2.2 was produced by coating sodium percarbonate particles by spraying a 20 wt.-% aqueous solution of  $\text{Na}_2\text{SO}_4$  in a fluidised bed using air and 4%  $\text{CO}_2$  as the fluidizing gas. The Raman spectra, recorded at the particle's surface and at distances of 2, 4, 6, 8, 10, 12 and 36  $\mu\text{m}$  from the surface, exhibit typical peaks which have been identified by the appellant as follows: 869  $\text{cm}^{-1}$  and 1080  $\text{cm}^{-1}$  (sodium percarbonate,  $2\text{Na}_2\text{CO}_3 \cdot 3\text{H}_2\text{O}_2$ ); 1081  $\text{cm}^{-1}$  (sodium carbonate,  $\text{Na}_2\text{CO}_3$ ); 1057  $\text{cm}^{-1}$  (Wegscheider's salt,  $3\text{NaHCO}_3 \cdot \text{Na}_2\text{CO}_3$ ); and 1064  $\text{cm}^{-1}$  (sodium sesquicarbonate,  $\text{NaHCO}_3 \cdot \text{Na}_2\text{CO}_3 \cdot 2\text{H}_2\text{O}$ ). However, a Raman peak characteristic for sodium bicarbonate ( $\text{NaHCO}_3$ ) at 1047  $\text{cm}^{-1}$  was not detected. According to the appellant, the spectra were recorded at a resolution of 2  $\text{cm}^{-1}$ , enough to distinguish between peaks separated by as little as approximately 10  $\text{cm}^{-1}$ , in particular between sodium bicarbonate at 1047  $\text{cm}^{-1}$ , Wegscheider's salt at 1057  $\text{cm}^{-1}$  and sodium sesquicarbonate at 1064  $\text{cm}^{-1}$ . Since, in spite of this, peaks attributable to sodium bicarbonate were not detected in any of the spectra, the appellant concluded that no such layer had actually formed in the process.

However, the board finds this spectroscopic evidence not conclusive to demonstrate that the claimed process could not be worked, for the following reasons:

Firstly, the board considers that sample 2.2 of D5 is not a product typical for the claimed process. Although its preparation conditions formally fall within claim 1, the carbon dioxide content in the fluidized bed was selected to be only 4%, which is far below the lowest values employed in the examples of the opposed patent (pure CO<sub>2</sub> in examples 10 and 12; an unspecified mixture of air and carbon dioxide in example 15; and 12% in examples 40 and 43 which were not carried out in a fluidized bed). In the respondent's experimental report D9, pure CO<sub>2</sub> was used (test 407). Samples produced under more favourable conditions would have been available to the appellant (for instance Hes 407 and Hes 409 of D10a, coated under pure CO<sub>2</sub> or under air containing 15% CO<sub>2</sub>, respectively). The board thus considers that the concentration of carbon dioxide of as low as 4% chosen by the appellant is not a fair interpretation of a "*gas rich in carbon dioxide*" as stipulated in the claim. Moreover, it is apparent from D5 (Table) that the stability data of samples 2.2 and 2.1 (produced under identical conditions, but in an air atmosphere) after 4 and 8 weeks are practically identical. This already suggests that only a very thin, inefficient coating, or no sodium bicarbonate coating at all had formed in sample 2.2, a fact which could be attributed to the low carbon dioxide concentration. The appellant argued that a gas containing 4% of carbon dioxide should be considered to be a "*gas rich in carbon dioxide*" within the meaning of claim 1 of the opposed patent in comparison with ambient atmosphere

which contains a mere 0.04% of carbon dioxide. This argument is not convincing because the term "*a gas rich in carbon dioxide*" should be interpreted in the context of the claim and the description, having regard to the purpose of the feature, *i.e.*, the generation of at least part of the first layer of sodium bicarbonate from the sodium carbonate of the sodium percarbonate and from the CO<sub>2</sub> in the gaseous phase, as stated in claim 1. As pointed out above, a concentration of 4% CO<sub>2</sub> is well below the concentration used in the examples.

Secondly, the board is not convinced that the resolution of the Raman spectra was such as to allow a detection of peaks of sodium bicarbonate at 1047 cm<sup>-1</sup> in the presence of strong and broad peaks of Wegscheider's salt at 1057 cm<sup>-1</sup> and sodium sesquicarbonate at 1064 cm<sup>-1</sup>. It should be born in mind that the sodium bicarbonate layer produced in accordance with claim 1 of the opposed patent can be very thin (see patent in suit, page 2, line 44 and page 3, lines 28, 29). Such a thin layer could give rise to comparatively weak Raman peaks that would be hidden by closely adjacent, much stronger peaks at 1057 cm<sup>-1</sup> and 1064 cm<sup>-1</sup>. The board notes that the enlarged graphs of the spectra recorded at the particle surface (page 10 of D15) do not show a baseline signal at a wavenumber of 1047 cm<sup>-1</sup>, but a rising portion of the strong peak at 1064 cm<sup>-1</sup>. Whether some weak signal is hidden below that rising portion remains obscure. The respondent has also rightfully pointed out that the Raman spectroscopic data submitted by the appellant do not report important parameters, such as number of scans, number of samples analysed, as well as information how the sample(s) was (were) collected,

handled and activated. The respondent further observed that from the several known basic vibrational bands of several species, in particular  $\text{HCO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{CO}_3^{2-}$  and  $\text{H}_2\text{O}_2$ , only one is shown in the spectra. This also casts doubt on the significance of the data.

The board concludes that the failure of the appellant to spectroscopically detect a layer of sodium bicarbonate in a particle taken from sample 2.2 of D5 is not a sufficient proof that the claimed process would generally not lead to its formation.

- 3.4 Indirect detection of the formation of the sodium bicarbonate layer can in principle be based on the observation - after storage of the sodium percarbonate particles - of a stabilisation or additional stabilisation induced by it. In Table 1 of the opposed patent, such stabilisation is observed in example 10 (decomposition 9.5% after 190 hours in open vessels in a climate chamber at 30°C and 70% humidity), as compared with comparative sample 13, having a coating layer of sodium bicarbonate deposited under air (decomposition 22% under identical conditions). However, product samples mixed with a zeolite (50/50) behaved differently. Sample 10+Z prepared in accordance with the claimed process showed a higher decomposition than sample 13, whereas sample 12+Z, prepared in accordance with variant b) of claim 1 exhibited a considerably lower decomposition than sample 13 and thus performed much better.

Test report D9 submitted by the respondent reveals a markedly reduced decomposition of sample 407 (in accordance with the invention), admixed with 50 wt.-%

zeolite 4A, after 2 and 4 weeks in open cups in a climate chamber (at 30°C and 70% humidity), compared with uncoated sodium percarbonate, and sodium percarbonate sprayed with water in air (sample 408) or sodium sulphate solution in air (sample 400).

On the balance of the results achieved, this evidence appears to speak in favour of the formation of a stabilizing first layer of sodium bicarbonate.

However, the test reports D5, D10a and D12 submitted by the appellant do not appear to exhibit the said stabilizing effect.

D5 reports on the oxygen stability of samples prepared on a laboratory scale by spray coating sodium percarbonate particles in a fluidised bed using air or air admixed with either 4 or 15 vol.-% of CO<sub>2</sub>. Oxygen stability was determined in a packaged washing powder composition consisting of a mixture of the coated sodium percarbonate particles, 36% zeolite and 5.6% TAED, after 4 and 8 weeks at 30°C and 80% relative humidity. These conditions differ from the ones used in accordance with the patent in suit (see paragraph [0029]) so that a direct comparison is not possible. Initial oxygen content is 14.4%. The particle size distribution of the starting product, which also might affect the stability, is not reported. The test data appear to show that there is no increase in oxygen stability in the samples 1.2, 1.3 and 2.2, produced under either 4 or 15 vol.-% of CO<sub>2</sub>, in comparison with the reference samples 1.1 and 2.1 produced under air. Minor differences (e.g., 77% vs. 75% or 86% vs. 87%; see Table on page 2) are in the board's view within the

expected experimental error. However, as pointed out by the respondent, the stability test carried out in D5 differs significantly from the one employed in the patent in suit by storage of the product in detergent packages (*i.e.*, in closed packages), instead of open cups. It cannot be excluded that under the more severe test condition of storage in open cups an improvement in stability might have been detected. Under these circumstances one cannot conclude from the absence of an improvement in the samples produced under 15 vol.-% of CO<sub>2</sub> that no layer of sodium bicarbonate has formed.

Document D12 was submitted by the appellant during the appeal procedure. A sodium percarbonate starting product was prepared in a spray granulation apparatus of medium size in a "*known manner analogous to WO 95/06615*" [D13]). This starting product after sieving had a narrow particle size distribution which, according to the appellant, largely corresponded to the respondent's commercial sodium percarbonate ECOX-C (internal code 01/02). The coating was carried out in a fluidized bed drier (Aeromatic Strea-1) in accordance with the opposed patent using air or 100% pure CO<sub>2</sub>. The second coating consisted of sodium sulphate. Every sample was repeated once to check reproducibility. Stability tests were carried out on the coated samples admixed with zeolite 4A and stored for 2 and 4 weeks in open cups in a climate chamber at 30°C and 70% relative humidity. The results are compiled in Table 2 and summarized in Table 3 of D12. Initial oxygen content (O<sub>a</sub>) varied between 12.4% and 12.91% (Table 2). Oxygen content after storage was as follows:

Sample Nr.	Gas in first step	Residual Oa content (%), relative to initial Oa content			
		after 2 weeks		after 4 weeks	
Ov 143.02	air	97.7	95.4	94.6	93.9
Ov 145.02	CO <sub>2</sub>	95.5	95.5	96.0	94.3
Ov 144.02	air	98.8	98.9	98.0	95.8
Ov 146.02	CO <sub>2</sub>	97.6	97.3	97.1	98.3

According to the appellant, the variation of the results between identically produced samples (Ov143.02 and Ov144.02 on the one hand; Ov145.02 and Ov146.02 on the other hand) is such that the claimed improvement in stability could not be observed. More specifically, the appellant argued in the letter of 23 October 2006 that the data were not statistically significant to demonstrate any difference between the samples produced in air and carbon dioxide.

Based on the same data, the respondent observed in its letter of 15 April 2003 that the **average** decrease in Oa values after 4 weeks was 4.5% in air, but only 3.6% for the samples coated under CO<sub>2</sub>, which amounted to a relative improvement of 20%. He also filed during oral proceedings a graph (D12a) representing the said average Oa values taken from D12 after 2 and 4 weeks. In the respondent's view, the graph suggests lower stability of the inventive samples after 2 weeks, but higher stability (lower oxygen loss) after 4 weeks.

Having regard to D12, the board considers that the statistical significance of the stability data is poor, taking into account that the differences between



repeated samples namely 0% to 2.3% (absolute) are in the same order as the differences between inventive and comparison samples (from 0.1% to 3.4%). The board is also not convinced that the graphs of D12a (which contain only 4 data points, namely two for the air samples and two for the CO<sub>2</sub> samples) are significant without an indication of the error margins associated with these data points. But even if D12a were accepted as it is, then an improvement in stability after 4 weeks of storage is still counterbalanced by the negative results after 2 weeks.

In the board's view, the data submitted in D12 are also flawed by the circumstance that the starting product has been prepared in a manner analogous to that of D13 (see page 2 of D12), *i.e.*, a document published after the priority date of the opposed patent and thus not comprised in the state of the art. It cannot thus be excluded that the samples have been prepared with the use of information not available at the priority date of the opposed patent which is not permissible. For all these reasons, the board is unable to draw from D12 a safe conclusion as to whether or not an improvement in oxygen stability exists.

In test report D10a, examples Hes 407 and Hes 410 (produced under 100% CO<sub>2</sub>) and Hes 409 (15 vol.-% CO<sub>2</sub>) exhibited essentially the same (2 examples) or higher (4 examples) oxygen loss after 2 and 4 weeks compared with reference sample Hes 408 (coated with sodium sulphate in air as a fluidizing gas) (see D10a, page 6, table). However, the question also arises whether the starting material of D10a has been prepared using post-

published information like the starting sodium percarbonate of D12.

In summary, the evidence on file submitted by both parties is partly insufficient, partly contradictory, to an extent which does not allow one to prove or disprove the formation of the first layer of sodium bicarbonate and the beneficial effect on oxygen stability associated with it.

The board had already observed in the communication annexed to the summons for oral proceedings that the effect of improving the stability of the sodium percarbonate particles is not a requirement of the claims. Therefore, failure of achieving said improvement could affect inventive step, but does not automatically signify that the skilled person could not carry out the claimed process steps.

The formation of the first layer of sodium bicarbonate was already present in the description and the claims of the granted patent. It would have been up to the opponent (now appellant) to demonstrate that this layer does not form. Under these circumstances, and in view of the further evidence submitted by the respondent (*i.e.* D9), the board considers that the evidence submitted by the appellant does not justify reversing his burden of proof for demonstrating that the claimed process was not disclosed in a manner sufficiently clear and complete for it to be carried out by the skilled person. The board will thus consider that a first layer of sodium bicarbonate is actually formed in the claimed process, as stated in claim 1 and in the

patent in suit, since the contrary was not convincingly proven.

- 3.5 The remaining arguments put forward by the appellant regarding the alleged insufficiency of the description are not convincing either, for the following reasons:

One argument concerned the fact that the stability values reported in the patent in suit for samples containing zeolite are apparently better than those obtained with samples without zeolites. In the appellant's submission, one should have expected the reverse. The board considers that even if this result may be surprising, this does not mean that the data are incorrect.

The appellant also referred to decision T 557/04 (of 19 July 2006, not published in OJ EPO). He argued that in the present case the burden of proof for showing the formation of the first layer of sodium bicarbonate should also be shifted to the respondent, in analogy to T 557/04.

The board is of the opinion that case T 557/04 is distinct from the present one in several respects. Firstly, in T 557/04 the crucial presence of a double layer on the surface of particles of calcium carbonate could not be inferred from the experimental evidence submitted by **both** parties, whereas in the present appeal case there is partly contradictory evidence. Secondly, in T 557/04 the model used for evaluating the ESCA data was admittedly critical, but not taught in the patent. Furthermore, using different known models, none of the experts came to the conclusion that the

coating was in form of a double layer. Because of these significant differences, the appellant's line of argument is not convincing.

3.6 In view of the above, the patent in suit meets the requirements of sufficiency of disclosure.

4. *Novelty (main request)*

4.1 Novelty was disputed having regard to document D3. Said document discloses spraying a supersaturated solution of sodium perborate separately or in admixture with sodium silicate on sodium percarbonate particles, in order to coat said particles with a dense coating of said reagents (see column 1, lines 49 - 53; 59 - 65; and column 2, lines 19 - 24). The coating process can be carried out in a rotating drum, in screw extruders, granulating plates, or in a fluidized bed. The latter may contain air, nitrogen or carbon dioxide as an inert fluidizing gas (see column 1, lines 54 - 62). D3 does not disclose any example involving spraying of the solution in a fluidized bed while using CO<sub>2</sub> as a fluidizing gas, nor does it teach the formation of a first layer of sodium bicarbonate.

In the board's opinion, in order to arrive at the claimed process, it would be necessary to choose independently from several lists of options disclosed in D3, that is, firstly choosing a fluidized bed as a reactor, secondly selecting CO<sub>2</sub> as the fluidizing gas, and, thirdly, separate spraying of the reagents in order to produce two layers. The claimed process may thus be seen as a selection from several lists disclosed in D3.

In accordance with decision T 12/81 (OJ EPO 1982, 296; see Reasons, points 13 and 14.1), an end product resulting from the reaction of a specific pair of starting substances may be seen as a novel selection for patent purposes if its preparation requires using entities from two classes of starting substances given in two lists of some length. This criterion has been applied to mixtures of two substances, selected from two lists (T 401/94 dated 18 August 1994, not published in OJ EPO: see Reasons, point 4.4) and has been confirmed in subsequent decisions (T 211/93 dated 11 July 1995, not published on OJ EPO: see Reasons, point 3, third paragraph; and T 175/86 dated 6 November 1990, not published in OJ EPO: see Reasons, point 5).

The board considers that the above cited case law is applicable to the present case which also concerns the selection of individual features from two lists of some length, the first list being represented by the list of coating apparatuses (rotating drum, screw extruders, granulating plates, fluidized bed), the second list by the list of fluidizing gases (air, nitrogen, carbon dioxide), and wherein only the specific selection of both a fluidized bed and of carbon dioxide as the fluidizing gas leads to the reaction of the sodium percarbonate particles and to the formation of a first layer of sodium bicarbonate.

Contrary to the appellant's argument, the board sees no implicit or inherent connection between the elements of the two lists. It is true that once a fluidized bed is chosen, a fluidizing gas must also be chosen. Still, both steps constitute distinct choices which have to be

made independently in order to arrive at the claimed process.

Therefore, in accordance with the above cited case law, the board accepts novelty of the subject matter of claim 1 of the main request having regard to document D3.

4.2 Other documents have not been cited for novelty. The board is also satisfied that no other document on file discloses the features of claim 1 in combination.

5. *Inventive step (main request)*

5.1 Document D3 has been considered by the parties to represent the closest prior art.

The test data in the patent itself as well as the additional experimental evidence filed by the parties do not unambiguously show that the claimed spray coating in an atmosphere containing CO<sub>2</sub> as a fluidizing gas produces coated sodium percarbonate particles having an improved oxygen stability, compared with particles conventionally coated in air as a fluidizing gas (see point 3.4. above). There is also no evidence on file to show an improvement having regard to the sodium percarbonate prepared in accordance with the closest prior art D3.

Starting from D3, the technical problem underlying the claimed process is therefore to provide a further process for producing stabilized particles of sodium percarbonate.

The proposed solution consists in the process as defined in claim 1 which leads to the formation of two layers of coatings, the first layer being of sodium bicarbonate. The board is satisfied that the above defined technical problem is solved by the claimed process in view of the examples of the patent in suit and the test report D9.

In the following discussion, the board will also considers, for the sake of the argument, document D6 as an alternative to D3 as the closest prior art. Since this argument was presented for the first time during oral proceedings, there is no evidence on file regarding which improvement the claimed invention would bring about having regard to D6. As pointed out by the respondent at the oral proceedings, starting from D6 the same technical problem as the one stated above could be formulated.

- 5.2 The appellant's arguments concerning the obviousness of the claimed process in view of D3 alone, or D6 in combination with D3, or D6 in combination with the common general knowledge or with D8 (see point X above) are not convincing, for the following reasons:

D3 indeed discloses a limited number of gases, including CO<sub>2</sub>, for the fluidizing bed. However, in the same sentence, these gases are called "*inert*", in the board's view signifying that they should neither react with the walls of the apparatus nor with the substances contained in the fluidized bed. The board therefore doubts that D3 would suggest to the skilled person that a coating of sodium bicarbonate could be obtained by reacting the sodium percarbonate particles with the

fluidizing gas CO<sub>2</sub>, a gas which is stated in D3 to be inert under the conditions of the fluidized bed spray coating process. In the board's view, the essential step of the claimed process is therefore not obvious having regard to document D3.

The claimed process is also not obvious starting from D6. Said document describes a process for stabilizing peroxy compounds, in particular sodium percarbonate, by applying a coating consisting of a mixed compound which is formed by crystallizing sodium carbonate with other mineral salts such as sodium bicarbonate and/or sodium sulphate. The coating process is carried out by spraying the coating solutions in a fluidized bed at 50 to 60°C using air as a fluidizing gas (see claims 1 and 5; page 5, fifth paragraph, to page 6, second paragraph; Table I). Air temperature is 80°C to 120°C (Table I). In examples 8 and 7R, on which the appellant relied during oral proceedings, the coating is formed either from an aqueous solution containing sodium carbonate and sodium bicarbonate or from a sodium sulphate solution. According to page 4, second paragraph, the apparatus and technique used for the coating is not critical. However, there is no apparent motivation to combine said teaching with D3. In any case, the skilled person would not change the gas of the fluidising bed from air to CO<sub>2</sub> in the expectation that a reaction would occur leading to the formation of a sodium bicarbonate layer on the sodium percarbonate particles because - as pointed out above - D3 contains no information suggesting that the "inert" fluidizing gas might react with the sodium carbonate of the sodium percarbonate particles.



The skilled person's general knowledge about direct heating with circulating heat carriers (the Ullmann article D16) would not have suggested the claimed process, either. D16 concerns an unrelated technical field which is removed from the technical field of fluidised bed apparatus and associated processes. There is no suggestion in D16 that hot combustion gases could be used directly as a fluidizing gas in a process for preparing coated sodium percarbonate particles.

Document D8 concerns a process for producing alkali-metal percarbonates comprising the steps of holding the percarbonate for a specific period of time at a specific temperature in a thin layer and then drying it at 20°C to 90°C using air containing carbon dioxide or pure carbon dioxide. The drying step can be performed in a fluidized bed. See column 1, lines 33 - 60; column 2, lines 15 - 31, 57 - 61; claims 1 and 2. This document does not relate to a process for stabilizing sodium percarbonate particles. It is therefore doubtful that the skilled person would have consulted it for the solution of the technical problem stated above. But even if he had done so, he would not have learned from D8 that CO<sub>2</sub> as a fluidizing gas would react in the presence of water with the sodium carbonate of the sodium percarbonate particles to form a first layer of sodium bicarbonate. Therefore, this document does not lead in an obvious manner to the claimed invention, no matter whether one combines it with D6 or any other cited prior art. The appellant's argument based on the combination of the teachings of D6 and D8 is therefore in the board's view based on an analysis of the case with hindsight.

5.3 No other documents have been cited for inventive step. The board is also not aware of any other document which, taken in combination with one of the above discussed documents, would render the claimed subject matter obvious.

The board concludes that claim 1 of the main request meets the requirements of Article 56 EPC.

5.4 Claims 2 - 4 of the main request are dependent on claim 1 and likewise allowable.

5.5 Since the main request can be allowed, there is no need to consider the auxiliary request.

## **Order**

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

The appeal is dismissed.

The Registrar

The Chairman

C. Vodz

M. Eberhard