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# Datasheet for the decision of 8 January 2015

Case Number: T 0834/12 - 3.3.06

04818425.3 Application Number:

Publication Number: 1687084

IPC: B01J27/122, B01J23/72,

B01J21/04, C07C17/15

Language of the proceedings: ΕN

### Title of invention:

Catalyst and gas phase method using such a catalyst

## Applicant:

SOLVAY SA

### Headword:

Oxychlorination catalyst / SOLVAY

## Relevant legal provisions:

EPC Art. 52(1), 54(1), 54(2), 56, 84, 114(2), 123(2) RPBA Art. 12(2), 12(4)

#### Keyword:

Admissibility of the main request (yes) Added matter (no) Claim amended to comprise product-byprocess features not objectionable Clarity (yes) Novelty (yes) Inventive step (yes)

### Decisions cited:

T 0150/82

# Catchword:



# Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 0834/12 - 3.3.06

DECISION
of Technical Board of Appeal 3.3.06
of 8 January 2015

Appellant: SOLVAY SA

(Applicant) Rue de Ransbeek, 310 1120 Bruxelles (BE)

Representative: Vande Gucht, Anne

Solvay S.A.

Département de la Propriété Industrielle

Rue de Ransbeek, 310 1120 Bruxelles (BE)

Decision under appeal: Decision of the Examining Division of the

European Patent Office posted on 4 October 2011

refusing European patent application No. 04818425.3 pursuant to Article 97(2) EPC.

#### Composition of the Board:

Chairman B. Czech
Members: L. Li Voti

S. Fernández de Córdoba

- 1 - T 0834/12

# Summary of Facts and Submissions

- I. This appeal lies from the decision of the Examining Division to refuse European patent application no. 04 818 425.3.
- II. In its decision, the Examining Division considered inter alia the following documents:
  - D1: FR 1 360 473 A;
  - D2: DE 14 43 703 A;
  - D4: US 2002/0007097 A1;
  - D5: Brochure "PURALOX®/CATALOX® High purity activated aluminas" by SASOL, bearing the indications "PURALOX CATALOX 01/03 GB" and "Status 01/2003";
  - D6: PRODUCT INFORMATION "PURALOX SCCa-5/150 Product Nr. 562 121" by Sasol Germany GmbH (07/03);
  - D7: PRODUCT INFORMATION "PURALOX SCCa-5/200 Product No. 562 129" by SASOL Germany GmbH;
  - D13: Declaration by Sasol Germany GmbH dated 11 October 2009.

Moreover, it considered also the alleged prior use, supposed to be evidenced by document bundle D15 regarding a commercialised catalyst for oxychlorination, lot MEDC000036B2 (delivery to DOW Deutschland Inc.), invoked in third party observations.

- III. The Examining Division came *inter alia* to the following conclusions (points 14 to 21 of the decision under appeal):
  - Claims 1 and 14 according to then pending main request contravened the requirements of Article 123(2) EPC.

- 2 - T 0834/12

- The claims at issue relating to a catalyst characterized by features relating to the process used for its preparation did not meet the requirements of Article 84 EPC.
- The subject-matter of independent claims 1, 14 and 16 according to then pending main request lacked novelty over example 9 of document D1 and examples 9 and 11 of document D2.
- The commercially available alumina used for the preparation of the catalysts exemplified in D4 was "obtained from SASOL, which produces alumina with a process including a Ziegler catalyst containing titanium, that inevitably leads to the presence of titania in the obtained alumina. In other words, the titanium has been introduced into an alumina precursor at a stage prior to the formation thereof, i.e. in one of the steps of the aluminium hydrate production."

Therefore, the subject-matter of independent claims 1, 14 and 16 according to then pending main request lacked novelty also over the examples of document D4.

- Out of the four auxiliary requests submitted during oral proceedings, only auxiliary request 4 was admitted into the proceedings.

However, claims 1 and 2 according to the auxiliary request 4 contravened the requirements of Article 123(2) EPC.

As regards the alleged prior use allegedly proved by D15, the Examining Division observed (part IV of the decision under appeal) that doubts remained as to whether or not the sold oxychlorination catalyst of lot

T 0834/12

MEDC000036B2 mentioned in the packaging list, allegedly being a catalyst as claimed in the present application, was actually identical to that of lot MEDC(std)lot20020036/B15324, the analysis of which was given in a test report. Therefore, the alleged prior use appeared not to have been proven up to the required standard ("up to the hilt").

IV. With its statement of the grounds of appeal the Appellant filed eight sets of claims as main request and 1<sup>st</sup> to 7<sup>th</sup> auxiliary requests, respectively. It contested the reasoning of the Examining Division and submitted that the claimed subject-matter was novel and involved an inventive step.

More particularly, the Appellant submitted *inter alia* (points 6.2, 6.3 and 4 of the statement) the following:

- Referring to decision T 150/82 (OJ 1984, 309), it held that since the process for the preparation of the claimed catalyst resulted in a close mixture of alumina and titanium, the exact structure of which was not defined in the application and was, in fact, unknown, the characterization of the claimed catalyst in terms of features of its method of preparation was admissible in the present case.
- Moreover, by specifying in claim 1 that titanium is to be added in one of the steps of the aluminium hydrate production before its calcination, it was clear that titanium was distributed throughout the body of the catalyst particles.
- None of the cited prior art provided any teaching or hint that the oxygen content in the tail gases of a process comprising the gas phase oxidation reaction of

- 4 - T 0834/12

a hydrocarbon could be maintained constant by using a catalyst as claimed containing an amount of titanium as specified, distributed throughout the body of the catalyst particles, as shown in the examples and figures of the present application.

- V. A third party filed observations under Article 115 EPC by letter of 5 June 2012, reiterating (only) the allegation of prior use of an oxychlorination catalyst allegedly proven by the document bundle D15. An additional declaration by Mr. Dürr and Mr. Casale (undated), was also submitted in this connection.
- VI. In a communication pursuant to Article 15(1) RPBA, the Board expressed its preliminary opinion, in particular, with respect to the admissibility of the Appellant's requests as well as with respect to novelty and inventive step of the claimed subject-matter. In this respect, the Board referred additionally to document
  - D8: A table labelled "Product information High purity activated aluminas PURALOX®, CATALOX®" by CONDEA (10/99), filed by a third party during the examination proceedings.

Moreover, the Board commented on the potential relevance of the prior use invoked with reference to D15 and indicated why the other prior uses invoked by a third party in the first instance proceedings appeared to be less relevant.

VII. The Appellant submitted by letter of 4 December 2014 four additional sets of amended claims as 8<sup>th</sup> to 11<sup>th</sup> auxiliary requests, together with arguments concerning some of the points raised in the Board's communication.

- 5 - T 0834/12

VIII. During the oral proceedings held on 8 January 2015, following a discussion on some outstanding deficiencies of the pending claims, the Appellant withdrew the then pending main request and the first and second auxiliary requests.

The Appellant submitted amended description pages adapted to the wording of the claims according to the new main request, i.e. the former third auxiliary request.

- IX. The independent claims according to said new main request of the Appellant read as follows:
  - "1. Catalyst containing active elements including copper deposited on an alumina, said alumina containing at least 0.03 g of titanium, expressed in metal form, per kg of alumina, and said alumina having a mean particle diameter between 5 and 200 µm, characterized in that the alumina results from the calcination of an aluminum hydrate and in that the titanium has been introduced in one of the steps of the aluminum hydrate production."
  - "8. Use of an alumina containing at least 0.03 g of titanium, expressed in metal form, per kg of alumina, and having a mean particle diameter between 5 and 200 µm as support for catalyst containing active elements including copper, characterized in that the alumina results from the calcination of an aluminum hydrate and in that the titanium has been introduced in one of the steps of the aluminum hydrate production."
  - "9. Method involving a gas phase reaction, characterized in that the gas phase reaction is

- 6 - T 0834/12

catalysed by a catalyst according to any one of Claims 1 to 7."

The dependent claims 2 to 7 relate to particular embodiments of the catalyst according to claim 1, whilst dependent claims 10 to 12 relate to particular embodiments of the method according to claim 9.

- X. The Appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the set of claims labelled third auxiliary request filed with letter of 31 January 2011 (main request) or, in the alternative, on the basis of the claims according to one of the auxiliary requests 4 to 7, also filed with letter of 31 January 2011, or auxiliary requests 8 to 11, filed with letter of 4 December 2014.
- XI. The arguments of the Appellant of relevance regarding the new main request are essentially the ones submitted in its statement of grounds of appeal (point IV *supra*).

### Reasons for the Decision

Admissibility of the main request at issue

- The claims according to the Appellant's main request (labelled "Third auxiliary request") were submitted for the first time with the statement setting out the grounds of appeal.
- 1.1 For the Board, the filing of these amended claims represents a legitimate reaction to the detailed reasoning given in the appealed decision regarding the objections having led to the refusal of the application

- 7 - T 0834/12

and a bona fide attempt to overcome the objections.

1.2 Therefore, the Board decided to admit this request into the proceedings despite its late filing (Articles 114(2) EPC and 12(2),(4) RPBA).

Main request - Compliance with Article 123(2) EPC

- 2. The Board is satisfied that the amended application documents meet the requirements of Article 123(2) EPC for the following reasons.
- 2.1 Amended claim 1 at issue finds support in claim 1 of the application as filed (reference being made hereinafter to the published version WO 2005/046866 A2 of the original PCT application) in combination with the essential and preferred features of the invention disclosed on page 2, lines 6 to 7, 23 to 25 and 27 to 28 of the description.
- 2.2 The wordings of claims 2 to 7 at issue are identical to those of claims 2 to 7 as of the application as filed.
- 2.3 Claim 8 at issue finds support in claim 8 of the application as filed in combination with the essential and preferred features of the invention disclosed on page 2, lines 1 to 7, 23 to 25 and 27 to 28 of the description.
- 2.4 The wordings of claims 9 to 12 at issue are identical to those of claims 10 to 13 of the application as filed, except for the adapted back-references in claims 10 to 12.
- 2.5 The Board is also satisfied that the adapted description pages are not objectionable under Article

- 8 - T 0834/12

123(2) EPC.

Main request - Compliance of the claims with the requirements of Article 84 EPC

- 3. The Board is also satisfied that all the claims at issue comply with the requirements of 84 EPC.
- 3.1 The catalyst of claim 1 is characterized inter alia by means of the product-by-process features "... that the alumina results from the calcination of an aluminum hydrate and in that the titanium has been introduced in one of the steps of the aluminum hydrate production."
- 3.2 For the Board, the wording of claim 1 at issue, by requiring that the titanium has to be introduced in one of the steps of the aluminum hydrate production before its calcination, will be understood by the skilled person as resulting in a catalyst wherein the titanium is implicitly distributed throughout the volume of each of the alumina particles rather than being contained prevalently on their surface.
- 3.3 This definition of the claimed catalysts is clear and supported by the description (Article 84 EPC).

  The Board is also satisfied that the remaining claims at issue, which were modified, as necessary, to conform with the more limited ambit of claim 1 at issue, comply with the requirements of Article 84 EPC.
- 3.4 Product-by-process features in claim 1 at issue
- 3.4.1 As submitted by the Appellant, the application as filed does not expressly describe any compositional or structural feature or any parameter which could define the intended structure of the catalysts according to

- 9 - T 0834/12

the invention (see point 3.2 *supra*) in a clear and supported manner.

- 3.4.2 The Board thus accepts that on the basis of the textual disclosure of the application as filed such a structure of the catalyst could only be expressed in claim 1 by incorporating some features of the process used for its preparation.
- 3.4.3 Hence, in the Board's judgement, the characterization of the claimed catalyst by means of features pertaining to its preparation and conferring patentability (see infra regarding novelty and inventive step) is admissible and can be allowed in the present case in accordance with established case law (see e.g. decision T 0150/82, point 10, last sentence, of the reasons).

Main request - Novelty

- 4. The catalyst of claim 1
- 4.1 The catalyst of claim 1 at issue contains active elements including copper, deposited on alumina resulting from the calcination of an aluminum hydrate, as well as titanium introduced in one of the steps of the aluminum hydrate production before calcination.

As already explained above (point 3.2), the wording of claim 1 implies that the titanium present is distributed throughout the alumina particles rather than being contained prevalently on their surface.

- 4.2 Novelty over D1 and D2
- 4.2.1 As correctly found in the decision under appeal (point 16, first full paragraph) both documents D1 (example 9) and D2 (examples 9 and 11) disclose the preparation of

- 10 - T 0834/12

an oxychlorination catalyst containing copper, titanium and other active elements, wherein the alumina carrier is calcined at 1060°C before deposition of titanium, copper and other active elements thereon.

- 4.2.2 Therefore, there can be no doubt that the structure of the catalysts prepared according to these examples, differs from that of the catalyst according to claim 1 at issue. In the latter, titanium is necessarily distributed throughout the alumina particles, and is not contained prevalently on their surface as in the former.
- 4.2.3 Documents D1 and D2 do not thus take away the novelty of the subject-matter of claim 1 at issue.
- 4.3 Novelty over D4
- 4.3.1 As correctly found in the decision under appeal (point 17, first full paragraph), document D4, filed on 3 May 2001 and published on 17 January 2002, discloses (example 1 and comparative example 1, respectively) the preparation of copper, magnesium and potassium containing oxychlorination catalysts by depositing said active elements onto an alumina of the type PURALOX® SCCa 5/150 or PURALOX® SCCa 5/200, respectively.

The description of D4 does not disclose whether the prepared catalysts contain any titanium and if yes, in which concentration, but it appears to indicate that the used aluminas had been commercially available from Condea (D4, paragraph [0023]).

4.3.2 The other items of evidence on file relating to PURALOX<sup>®</sup> aluminas do not convincingly establish that one or both of the specific aluminas used according to D4

- 11 - T 0834/12

implicitly had to contain titania in a concentration as required by claim 1 at issue.

i) Document D5, apparently relating to commercial products available on January 2003 (see last two pages, right corner at the bottom), i.e. before the priority date of the present application but after the filing (and publication) date(s) of D4, concerns inter alia aluminas of the PURALOX® type marketed by Sasol. For the Board, it is not derivable from D5 whether the PURALOX® aluminas commercialized by Sasol were necessarily identical to those previously commercialized under said trade name by Condea.

The Board remarks also that page 1 of this document (right column) reads: "Unlike other alumina manufacturing processes which use less pure bauxite derivatives as a starting material, Sasol has pioneered a process based on aluminum alkoxide which produces synthetic boehmite aluminas of high purity. Examples of some trace impurities are shown in table 1." (emphasis added by the Board). Table 1 on page 2 reports as impurities iron, sodium and silicium oxides but not titania.

In the Board's view, there are thus serious doubts that Sasol's PURALOX<sup>®</sup> aluminas were prepared in the same way as the PURALOX<sup>®</sup> products previously commercialized by Condea. Therefore, it is more than questionable that these products were identical, in particular in terms of their impurities contents, let alone in terms of an impurity not even mentioned in D4 and D5. Moreover, document D5 does not contain any support for the statement in the decision under appeal (point 17) that "SASOL produces alumina with a process including a Ziegler catalyst containing titanium, that inevitably

- 12 - T 0834/12

leads to the presence of titania in the obtained alumina". But even if this were true, there is still no evidence on file showing that the same process used by Sasol was previously applied for preparing the aluminas marketed by Condea and used according to document D4.

Therefore, document D5 cannot be used as evidence that the aluminas used in D4 necessarily contained titanium, let alone in a concentration as required by claim 1 at issue.

- ii) The Board remarks also, for the sake of completeness, that the cited documents D6 and D7, referring to  $PURALOX^{®}$  SCCa 5/150 or  $PURALOX^{®}$  SCCa 5/200 aluminas marketed by Sasol, do not list titania as a component or impurity of these products.
- iii) Document D8 concerns a table reporting chemical and physical features of various grades of PURALOX $^{\circledR}$  and CATALOX $^{\circledR}$  aluminas marketed by Condea, apparently commercially available in October 1999 (sheet 1/2, left bottom corner).

Therefore, this document could be representative also for the alumina products used in document D4.

This table reports inter alia the generic composition of the PURALOX<sup>®</sup> SCCa (90-210) class of aluminas, to which the aluminas used in D4 belong. According to this table such aluminas consist of 98%  $Al_2O_3$ , 0.002%  $Na_2O$  and 2% "L.O.I.", i.e. loss on ignition materials.

A line below this table reads: "Chemical purity: C: 0.05%,  $SiO_2$ : 0.01-015%,  $Fe_2O_3$ : 0.005-0.015%,  $TiO_2$ : 0.01-0.20%.

- 13 - T 0834/12

However, the components listed in the table already add up to 100%.

Hence, for the Board, the line below the table merely is a list of impurities which **may** be contained in some, but which must not necessarily be present in each of the products described, depending, for instance, on the method of preparation used.

Therefore, in the Board's judgement, document D8 does not permit to safely conclude that the PURALOX® SCCa 5/150 or PURALOX® SCCa 5/200 alumina marketed by CONDEA and used in D4 contained titanium, let alone in a concentrations as required by claim 1 at issue.

- iv) Finally, document D13 is a declaration concerning aluminas of the series PURALOX $^{(8)}$  SCCa 30/180, 30/200, 20/220 and 30/150, which are different from those used in document D4.
- 4.3.3 Since there is no evidence on file which convincingly establishes that the aluminas used in the examples of document D4 contained titanium, document D4 is not, in the Board's judgment, novelty-destroying either for the subject-matter of claim 1 at issue.
- As regards the alleged prior use supposedly evidenced by document bundle D15, the Board remarks that the third party maintained in its letter of 5 June 2012 its earlier observations under Article 115 EPC, already submitted during examination by letter of 30 December 2010, and submitted the additional declaration by Mr. Dürr and Mr. Casale which reads:

  "...the lot included in the Packing List of Sept. 27, 2002 having number MEDC000036B2 sent to Dow Deutschland Inc. client is the same as the lot having number MEDC(std)lot20020036/B15324. The difference between the two lots is due to the practice established since long

T 0834/12

time according to which the lot numbers of aluminas used as raw material to prepare our catalyst are not included in the Packing Lists provided to the clients.".

- 4.4.1 From this declaration it can be understood that "B2", the last part of the identification number of the allegedly sold and delivered lot of catalyst "number MEDC000036B2", does not necessarily imply that the alumina carrier of said catalyst was identical to that of lot "MEDC(std)lot20020036/B15324" since it had apparently been used to represent also aluminas having a lot number starting with B but being different from B15324. Considering also the fact that no complete and fully comparable analysis of the two lots of products MEDC000036B2 and MEDC(std)lot20020036/B15324 was filed, it cannot be derived with certainty from the available items of evidence that the sold and delivered lot MEDC000036B2 was identical to the lot MEDC(std)lot20020036/B15324.
- 4.4.2 Therefore, if only for this reason, the Board concludes that said alleged prior use is not proved to the required standard. Hence, it was disregarded by the Board in its assessment of the patentability of the claimed subject-matter.
- 4.5 The Board is also satisfied that the other prior art documents and allegations of prior use referred to during examination are not more relevant than the ones addressed before with regard to novelty (see point VI above).
- 4.6 In summary, the subject-matter of claim 1 at issue thus is novel (Articles 52(1) and 54(1)(2) EPC).

- 15 - T 0834/12

Consequently, the use according to claim 8 at issue of an alumina as defined in claim 1 as support in a catalyst as defined in claim 1, and the methods according to claim 9 to 12 at issue, involving a gas phase reaction catalysed by a catalyst according to claim 1, are also novel (Articles 52(1) and 54(1)(2) EPC).

Main request - Inventive step

- 5. The invention
- 5.1 The present invention concerns catalysts containing active elements including copper deposited on alumina, as well as the use of such an alumina as support for catalysts containing active elements including copper, and methods involving a gas phase reaction catalysed by such catalysts
- As explained in the description of the present application (page 1, lines 5 to 25), it was known to use catalysts containing active elements including copper deposited on alumina in gas phase reactions like the oxychlorination of hydrocarbons. Moreover, it was customary to recycle the tail gases produced in such reactions. In particular, "Insofar as a combustible gas is recycled via a compressor, the oxygen content of this gas plays a key role in maintaining the safety of the system. Depending on the pressures and temperatures encountered, various oxygen limitations are imposed."
- 5.3 Therefore, the present application addresses specifically the issue of providing a catalyst which is suitable for maintaining a constant oxygen content in the tail gases of reactions like the oxychlorination of hydrocarbons (see page 1, lines 25 to 31).

- 16 - T 0834/12

# 6. Closest prior art

The cited prior art does not deal explicitly with the specific issue mentioned under point 5.3 supra. Under these circumstances, the Board considers that the oxychlorination catalysts containing copper and titanium deposited on alumina described in D1/example 9 or D2/examples 9 and 11 to represent the closest prior art (see point 4.2.1 above).

## 7. Technical problem underlying the invention

In the light of the closest prior art identified above the technical problem underlying the present invention can be seen in the provision of a catalyst suitable for maintaining a constant oxygen content in the tail gas of an oxychlorination reaction.

## 8. The solution

As the solution to the technical problem indicated above, the application proposes a catalyst according to claim 1 at issue, which is characterised in particular in that said alumina

- contains "at least 0.03 g of titanium, expressed in metal form, per kg of alumina",
- has a "mean particle diameter between 5 and 200 μm",
- and "results from the calcination of an aluminum hydrate",

and in that

"the titanium has been introduced in one of the steps of the aluminum hydrate production".

#### 9. Success of the solution

- 17 - T 0834/12

- 9.1 Example 5 of the present application shows that, by using a catalyst as claimed (described in Example 1), the oxygen content in the tail gases produced in the oxychlorination of ethylene remains fairly constant over time during a 24 hours test period (see plot A in figure 3). This is not the case when using, under comparable reaction conditions, the comparative catalyst of example 3 (see plot A in figure 2), which differs only in terms of the titanium content of the alumina obtained by calcination of an aluminum hydrate, which is inferior to the lower limit of 0.03 g, expressed in metal form, per kg of alumina required according to claim 1 at issue.
- 9.2 Therefore, the Board accepts that the technical problem identified above has been convincingly solved by means of a catalyst having all the features of claim 1 at issue.
- 10. Non-obviousness of the solution
- 10.1 There is no suggestion in any of documents D1 or D2 to provide a catalyst wherein titanium is present throughout the alumina particles rather then being deposited onto the calcined alumina surface, let alone in amounts of at least 0.03 g, expressed in metal form, per kg of alumina, in order to maintain a constant oxygen content in the tail gas of the oxychlorination reaction.
- 10.2 Neither does document D4 disclose any element of information that could suggest that the use of an alumina containing such a concentration of titanium, introduced in one of the steps of the aluminum hydrate production before calcination, as support in the catalysts disclosed therein, could bring about the

- 18 - T 0834/12

technical advantage of a relatively stable tail gas oxygen concentration when used in reactions of the type mentioned in the application.

- 10.3 The Board thus concludes that, starting from a catalyst as disclosed in the examples of D1 or D2, the skilled person seeking to prepare a catalyst containing copper deposited on alumina providing such a relatively stable tail gas oxygen concentration upon use, was not induced by any of D1, D2 or D4 to modify the catalyst preparation method described in D1/D2 such as to obtain a catalyst with all the compositional and structural features required by claim 1 at issue.
- 10.4 The Board is also satisfied that none of the other items of evidence on file call into question the non-obviousness of the claimed catalyst.
- 10.5 The Board thus concludes that the subject-matter of claim 1 at issue involves an inventive step (Articles 52(1) and 56 EPC).

Consequently, the use according to claim 8 at issue of an alumina as defined in claim 1 as support in a catalyst as defined in claim 1, and the methods according to claim 9 to 12 at issue, involving a gas phase reaction catalysed by a catalyst according to claim 1, likewise involve an inventive step (Articles 52(1) and 56 EPC).

- 19 - T 0834/12

## Order

# For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the department of first instance with the order to grant a patent in the following version:

## Claims:

No. 1 to 12 (labelled third auxiliary request) filed with letter of 31 January 2011.

## Description:

Pages 1 to 9 filed during oral proceedings of 8 January 2015.

# Figures:

Sheets 1/3 to 3/3 as published under the PCT (WO 2005/046866 A2).

- 20 - T 0834/12

The Registrar:

The Chairman:



D. Magliano B. Czech

Decision electronically authenticated