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Datasheet for the decision of 12 January 2006

Case Number: T 0980/03 - 3.3.07

Application Number: 94112508.0

Publication Number: 0638362

IPC: B01J 29/04

Language of the proceedings: EN

Title of invention:
Titanosilicate catalyst particle

Patent Proprietor:
MITSUBISHI GAS CHEMICAL COMPANY, INC.

Opponent:
Degussa AG

Headword:
-

Relevant legal provisions:
EPC Art. 54, 111(1)

Keyword:
"Novelty (yes) - onus of proof to show that all features of an independent claim are met by a prior art document lies with the opponent"
"Remittal (yes)"

Decisions cited:
T 0219/83

Catchword:
-

Case Number: T 0980/03 **-** 3.3.07

DECISION
of the Technical Board of Appeal 3.3.07
of 12 January 2006

Appellant:
(Patent Proprietor)

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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 14 July 2003 revoking European patent No. 0638362 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: S. Perryman
Members: B. Struif
G. Santavicca

Summary of Facts and Submissions

I. The appeal lies against the decision of the opposition division to revoke European patent No. 0 638 362. This was granted with 18 claims. Claims 1 and 3 read as follows:

"1. A titanosilicate catalyst comprising titanosilicate primary particles which are combined together forming secondary particles, wherein the combined part of the primary particles is a crystalline substance, and said secondary particles have a median diameter of 1 µm or more and pores of diameter 5 to 30 nm (50 to 300 Å).

"3. A method for preparing a titanosilicate catalyst according to claim 1, comprising:

- reacting silicon oxide, titanium oxide and tetraalcylammonium [sic] hydroxide in the presence of water or steam to form primary titanosilicate particles;

- decreasing the pH of the reaction mixture to form a secondary particle comprising a combination of the primary particles with one another; and

- calcining the secondary particle."

II. An opposition was filed against the above patent, in which revocation of the patent in its entirety was requested on the grounds of lack of novelty and lack of an inventive step (Article 100(a) EPC). The opposition was supported *inter alia* by the following document:

D1: A.J.H.P. van der Pol and J.H.C. van Hooff, "Parameters affecting the synthesis of titanium silicalite 1", ***Applied Catalysis*** A: ***General***, 92 (1992), 93-111

III. The sole request of the proprietor before the opposition division was to maintain the patent as granted. By a decision posted on 14 July 2003, the opposition division revoked the patent for lack of novelty of the subject-matter of claim 1, holding that:

(a) D1 disclosed a titanosilicalite sample TS-35-AI which consisted of very small spherical cystallites of approximately 0.09 µm and looked like cauliflowers. The crystallites were stuck together to form large aggregates having a size of a few millimetres.

(b) The claimed feature "combined together forming secondary particles" was not restricted to a specific form of bonding so that the aggregate disclosed in D1 showed this feature. Even though the patent mentioned in the discussion of the prior art (page 2, paragraph [0006], that prior art particles suffered from a lack of being firmly connected mutually via chemical bonding, in the assessment of novelty the general meaning of a term used in the claim had to be used, and not what had perhaps been intended in the contested patent.

(c) Since in D1 all samples with crystallite sizes of 0.09 to 12 µm were stated to exhibit high crystallinity (pages 110/111 bridging paragraph), and sample TS-35-AI was the only sample of crystallite size 0.09 µm, this sample had to be treated as having high crystallinity and the claimed feature "the combined part of the primary particles is a crystalline substance" was disclosed in D1.

(d) As regards the pore diameter of 5 to 30 nm, the patent in suit mentioned nitrogen adsorption and scanning electron microscopy (SEM) as suitable methods for the determination of the pore size, the results of which showed a good correspondence. The pore feature was not restricted to an average or median pore size. Hence, if a sample exhibited at least two pores in the range of 5 to 30 nm measured by any suitable method, that feature was met. In view of the breadth of the pore size feature as claimed and of the likelihood that at least two pores in sample TS-35-AI satisfied the claimed range that feature was inherent to sample TS-35-AI of D1. As it was practically impossible to verify whether a sample met this criterion, and the patent itself gave no data on measurements it was acceptable that the opponent had not reworked TS-35AI.

(e) Therefore, the subject-matter of claim 1 was not novel.

IV. The proprietor (appellant) appealed against the above decision requesting as main request that the patent be maintained as granted, and also submitting three auxiliary requests with amended claims.

V. In reply the respondent submitted document

D7: JP-A-4-59769 (in the form of its English translation)

VI. The parties were summoned to oral proceedings. In response, by letter of 5 December 2005, the respondent submitted document

D8: German DIN Norm 66134.

VII. Oral proceedings were held on 12 January 2006 pursuant to Rule 71(2) EPC in the absence of the appellant, who had informed the Board by letter of 18 November 2005 that he would not be attending the oral proceedings.

VIII. As regards the main request to maintain the patent with the claims as granted, the appellant had argued in writing as follows:

(a) D1 concerned the production of small titanium silicalite crystallites which corresponded to the primary particles of the patent in suit. Sample TS-35-AI was an exception in the context of D1, since the crystallites stuck together to form large aggregates which however only occurred by chance.

(b) The term "stuck together" in the context of D1 had a technical meaning different from the term "combined together" used in the patent in suit, since the latter expression implied a sufficiently strong coherence to meet the required mechanical strength.

(c) Furthermore, in the patent in suit the crystallinity of the primary particles was determined by XRD and SEM techniques. However, in D1 the crystallinity of sample TS-35-AI had not been measured. Hence, it might well be that a large amount of amorphous material was formed when sticking the smaller particles together. Thus, the strong coherence feature could not be derived from D1 in a clear and unambiguous manner.

(d) No pore size was mentioned in D1, let alone an average or median pore size. The patent in suit referred to a pore size distribution measured by nitrogen adsorption showing mesopores (corresponding to the gap occurring between the primary particles) of a diameter from 5 to 30 nm. Since the nitrogen adsorption measurement was a well known method, the opponent had not discharged his onus of proof to show that sample TS-35-AI had any mesopores. Consequently, the opponent had failed to show that novelty of the claimed subject-matter as granted was destroyed.

(e) No arguments on D7 were ever submitted by the appellant nor was its introduction into the proceedings objected to.

IX. The arguments of the respondent, in respect of the main request, can be summarised as follows:

(a) For destroying novelty, it was sufficient that sample TS-35-AI of D1 fulfilled the claimed features. There was no hint in the patent in suit that the term "combined together" meant something different from the term "stuck together" used in D1.

(b) Since the Abstract of D1 and the passage bridging pages 110 and 111 referred to the synthesis of highly crystalline batches of TS-1 zeolites with a crystallite size ranging from 0.09 to 12 µm, and sample TS-35-AI was the only one indicated as having a crystallite size of 0.09 µm, these passages must be taken also as referring to sample TS-35-AI. The agglomerates of sample TS-35-AI as a whole must have shown a high crystallinity so that in that sample also the combining part between the primary particles must have been crystalline.

(c) Furthermore, there was no indication in the patent in suit, how the crystalline combined part could be determined and differentiated from other parts of the catalyst.

(d) Apart from the pore size, all of the other features of claim 1 were directly and unambiguously disclosed in D1. The decision of the opposition division, to interpret claim 1 broadly, was correct. Claim 1 could not be interpreted in a restricted sense so that the scope of protection was less than its wording (protocol on the interpretation of Article 69 EPC). Thus, it was sufficient if only two pores were within the claimed pore size range to meet the claimed requirement. Since the primary particles in D1 were spheres, the voids between them could be calculated to be 22.5 nm, which were within the claimed range.

(e) According to the description of the patent in suit, the pore size referred to the mesopores corresponding to the gap occurring between the primary particles. Since the primary particles were catalytically active, the secondary particles and its mesopores did not contribute to its catalytic activity. In examples 1, 3 and 5 as filed, the pore sizes were measured by means of SEM. There was no hint that the pore size of the exemplified samples was measured by the nitrogen adsorption method. From the pore sizes specified in the examples no median pore size could be calculated. Thus, the claimed pore size range only required that at least two pores within that range had to be present.

(f) Although in D1 the tested samples concerned products dried at 70°C, those samples were intended to be used as catalysts and thus they would have been calcined for that purpose. The physical parameters of the dried product would not be different from those of a calcined product.

(g) It was not necessary that the production method of each sample be exactly described in D1 for this to destroy novelty.

(h) Method claim 3 was anticipated by comparative example 1 of D7, in which a process for the synthesis of a titanosilicate was described as involving heating the reaction mixture having a pH of 10 to 10.5 to 150°C and afterwards to 210°C. The latter temperature resulted in a pH decrease within the range as claimed, hence in line with the patent in suit. The median particle size was 3 µm and the particles were agglomerates of primary crystallites as shown in Fig. 2.

(i) Since the specified pore diameter was not appropriate for distinguishing the claimed subject-matter from the prior art, D7 disclosed a process according to claim 3, which resulted in a product meeting the features of claim 1. Thus, the subject-matter of claim 3 lacked novelty over D7 as well.

X. The appellant (patent proprietor) requested that the decision under appeal be set aside and that the patent be maintained as granted (main request) or, alternatively, be maintained on the basis of one of the three auxiliary requests filed with the statement setting out the grounds of appeal.

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

Reasons for the Decision

1. The appeal is admissible.

Novelty (Main request)

2. The opposition division and the respondent were of the opinion that a titanium silicalite according to sample

TS-35-AI of D1 destroyed novelty of the subject-matter of the granted claims.

2.1 D1 is a scientific paper concerning the parameters affecting the synthesis of titanium silicalite-1 (abbreviated TS-1; see title). The zeolite synthesis was carried out at 175°C by using commercial silica sol and tetraethyl ortho silicate as silicon sources, and tetraethyl orthotitanate as titanium source. In order to investigate the crystallisation phenomenon, small samples were taken at different times (2, 4, 6, 8, 10 days). The solid products were isolated by means of filtration or centrifugation, washed free from alkali and dried at 70°C overnight. The degree of crystallinity of the samples was evaluated by X-ray diffraction (XRD). The morphology of the samples was investigated by using scanning electron microscopy (SEM) ("Experimental"; page 94).

2.1.1 The preparation of TS-1 is described in methods 1 and 2 of D1 (pages 95 and 96). Method 1 describes the preparation of TS-1 with Si/Ti=70 and $OH^-/Si=0.43$. Method 2 describes the preparation of TS-1 with Si/Ti=49 and $OH^-/Si=0.27$. These methods concern the specific synthesis conditions without addressing the separation, washing and drying steps of the products, which are described in the general experimental part mentioned above (point 2.1 above). The specific methods for identification of the product by X-ray powder diffraction, infrared spectra (IR), SEM and chemical analysis, mentioned on page 96 of D1 were thus made on samples which had been dried overnight at 70°C. Only before using the samples as catalysts, were they calcined in a stream of air at 550°C for 3 hours (page 96, fifth complete paragraph).

2.1.2 D1 focuses on the synthesis of crystalline titanium silicalite-1, in particular under the influence of SiO_2 source, crystallisation time, stirred and static crystallization conditions, TPAOH source, OH^-/Si ratio, Si/Ti ratio and sol concentration (see abstract). There is no information in D1 on the catalyst particles which have been calcined, and the physical data presented in D1 thus refers to samples, which have been dried at 70°C but which have not been calcined. This also applies to sample TS-35-AI.

2.2 In order to evaluate whether the titanium silicate 1 of sample TS-35-AI anticipates the claimed subject-matter, the question to be answered is whether the features of claim 1 are directly and unambiguously disclosed in D1. For that purpose it is necessary to construe claim 1.

It is a general principle of construction of documents that these are to be considered as a whole, and in this case in view of the idiosyncratic use of English terms both in the claims and in the description, it is necessary to consider both to arrive at the meaning of claim 1. This should not be taken to mean that where the claim is by itself clear, it can be notionally rewritten by reference to the description saying something different, but the meaning attributed to terms in the claims should be consistent with the meaning attributed to them in the description.

2.2.1 The requirements of Claim 1 can be split into the following separate features:

a) A titanosilicate catalyst comprising primary titanosilicate particles;
b) which (the primary titanosilicate) particles are combined together to form secondary particles;
c) wherein the combined part of the primary particles is a crystalline substance, and
d) said secondary particles have a median diameter of 1 µm or more and
e) (said secondary particles) have pores of 5 to 30 nm in diameter.

2.2.2 According to paragraph [0028] of the description, "After washing, the thus obtained titanosilicate particles are calcined from 450 to 600°C for from 1 to 100 hours, more preferably from 5 to 10 hours, to thereby prepare the inventive catalyst.". The reference in feature a) to a titanosilicalite **catalyst** (emphasis by the Board) is thus a reference to a final product, which is suitable for use as catalyst, which has been calcined, and the physical parameters indicated in the claim refer to a catalyst in the form of a calcined product (examples of the patent in suit).

2.2.3 From the above analysis of D1 (Point 2.1.2 above), the physical data presented in D1 refer to samples, which have been dried at 70°C but which have not been calcined. This also applies to sample TS-35-AI, which for use as a catalyst in accordance with D1 would first need to be calcined. A comparison of what is said in D1 about sample TS-35-AI, with the requirements of present claim 1 is not comparing like with like.

2.2.4 The argument of the respondent that there would be no differences in the product parameters which had been obtained after drying compared to after calcination is a mere conjecture which cannot be accepted in the absence of evidence. The claim requires this feature to be present in the catalyst particles, that is after calcination. The onus of proof in that respect lies with the opponent (appellant), which he has not discharged (see decision T 0219/83, published in OJ EPO 1986, 211, point 12). The opponent has thus not shown that the claimed feature a) can directly and unambiguously be derived from D1.

2.3 Feature b) of claim 1 requires that the primary titanosilicate) particles are combined together to form secondary particles. ***Prima facie*** "combined" is a broad term saying nothing about how the combination takes place, or how strongly the primary particles are held together to form the secondary particles.

2.3.1 According to the description of patent in suit "the primary particles combine or agglomerate with one another to obtain a secondary particle having an increased particle diameter" (page 3, lines 49 and 50). This again is very general. It is true that the description seeks to distinguish the particles of the invention from prior art secondary particles where the primary particles are "not firmly connected mutually via chemical bond" (paragraph [0006]). However, this is no reason to read some special restricted meaning into "combined" as appearing in feature b) of claim 1.

2.3.2 In D1 (page 105, Table 6), sample TS-35-AI is mentioned with details of the Si/Ti ratio and the OH^-/Si ratio to describe the influence of the sol concentration on the crystallization. The footnote to Table 6 referring to sample TS-35-AI reads: "Very small spherical crystallites of approximately 0,09 µm, look like cauliflowers. Crystallites are stuck together to form large aggregates". These large aggregates of fine crystallites constitute secondary particles, and this meets the requirement of feature b) of claim.

2.4 Feature c) of claim 1 requires that "the combined part of the primary particles is a crystalline substance". Firstly according to claim 1, the primary particles "are combined together forming secondary particles". Furthermore in the description, the "combined part" refers to the part "where the primary particles are combined with one another" (paragraph [0045]). "The combination part of primary particles correspond to the part where the primary particle having sphere shape contacted with one another" (paragraph [0046]). "The connected parts of the primary particles with one another were crystalline substance" (paragraph [0052]. From the above it follows that the only meaning that can be given to feature c) of claim 1 is: the "combined part" means the connecting portions between the primary particles, which cause the formation of the secondary particles.

2.4.1 The core of feature (c) is that "the combined part ... is a crystalline substance". It is noted that the claimed term "is a crystalline substance" has a limiting meaning in the sense that the connecting portions must be crystalline and not amorphous. This in line with the purpose and technical effects aimed at in the patent in suit according to which the mechanical strength of the connecting part between the primary particles, when the part is an amorphous substance, is less than that of a crystalline part (see paragraph [0008]).

2.4.2 The opposition division and the respondent however treated this feature as not being clear, since the method how the crystallinity of the connecting part was determined was not clearly described.

2.4.3 From the patent in suit the following information can be gathered: "It is apparent from the fact that in the X-ray diffraction clear diffraction pattern and no broad scattering due to the existence of amorphous part were observed and that the thus prepared catalyst is in fact crystalline substance" (paragraph [0029]). Although no detailed XRD data are given in the patent in suit, this disclosure is based on experimental results and applies to the catalyst in the form of secondary particles including the connecting portions between the primary particles. The statement makes it clear that also the connecting portions between the primary particles must be a crystalline substance and not an amorphous substance and that a clear diffraction pattern is obtained which does not show any broad scattering.

2.4.4 In D1, no data of an XRD spectrum for sample TS-35-AI are given. In that respect, a reference to "not determined" is made in connection with sample TS-35-AI (Table 8, page 106). Also for sample TS-35-AC XRD data are "not determined", although that sample is expressly stated (page 103, lines 2 and 3; footnotes in Tables 4 and 6) as including an amorphous gel on the surface and looking like cauliflower, in the same way as do the particles of sample TS-35-AI.

2.4.5 According to the patent in suit, SEM pictures have been made to illustrate feature c). In Example 3, "according to SEM observation, ... the connected parts of the primary particles with one another were crystalline substance" (paragraph [0052]). By way of contrast, in Comparative Example 5 describing a prior art catalyst sample according to US-A-4 410 501, it is noted that "according to SEM observation, the combined part where the primary particle combined with one another was an amorphous substance." Consequently, according to those SEM pictures, the connecting portion between the primary particles according to the patent in suit is crystalline.

2.4.6 The opposition division objected that SEM was not a suitable tool to determine crystallinity. Although the "degree" of crystallinity can be detected by XRD, SEM is nevertheless described in the patent in suit as a suitable additional means to determine whether there is crystalline or amorphous substance present or not. This is in line with D1, according to which a SEM picture of sample TS-35-AC was made, with the result that these particles were not neatly crystallized, since the outer surface was covered with amorphous gel (page 103, lines 2 and 3; and footnote to sample TS-35-AC in Table 4). Thus, the board sees no reasons why SEM pictures should not be taken into account to assess whether feature c) is met.

2.4.7 According to D1, very high silica concentrations give rise to the formation of aggregates of very small crystalline spheres. If the silica concentration is higher than 5 mol/l, aggregates of a few millimetres consisting of crystallites of about 0.09 µm are found (page 106, first full paragraph). Although that disclosure can be read together with the disclosure of sample TS-35-AI, there is no indication, whether the connecting portions between these crystallite particles are made of a crystalline substance or not.

2.4.8 The respondent argued that from the passage in D1 bridging pages 110 and 111 reading "It is possible to synthesize TS-1 in the range of Si/Ti = 24 to ∞ (0 to 4 mol-% Ti) with crystallite sizes of 0.09 to 12 µm, high crystallinity (XRD) and good IR spectra" and the fact that the only sample with crystallite size of 0.09 µm specifically mentioned is sample TS-35-AI, of which it is said "Very small spherical crystallites of approximately 0.09 µm, look like cauliflowers. Crystallites are stuck together to form large aggregates", it must be concluded that the connecting portions of sample TS-35-AI are crystalline. For the Board this does not follow clearly and unambiguously. Even if the reference to "high crystallinity" is to apply to sample TS-35-AI, about which there must be doubt, this does not preclude there being small amorphous connecting portions causing the primary crystallites to be stuck together, as appears to be the case for the sample TS-35-AC whose total form is also described as like cauliflowers as a result of what looks like amorphous gel precipitated on cubic crystallites. Furthermore, the word "stuck" in the footnote of Table 6 for characterizing Sample TS-35-AI ("Crystallites are stuck together to form large aggregates") would be used an unusual description of connecting portions of crystalline substance.

2.4.9 There has been no reworking by any party of the way sample TS-35-AI was obtained in D1, followed by calcination, and at least an attempt to measure the properties of connecting portions between the primary particles. In the absence of evidence such a reworking, the Board cannot accept that D1 discloses feature c) or that this feature cannot be measured. In summary, the board has not been not convinced that the disclosure of sample TS-35-AI in D1 makes directly and unambiguously available that the connecting part between the primary crystallite particles is a crystalline substance. The onus of proof lies with the opponent (appellant), who has not discharged it (T 0219/83,cited *supra*).

2.5 According to feature d) of claim 1, the secondary particles have a median diameter of 1 μm or more. The presence of this feature for many of the samples of D1, including sample TS-35-AI, is apparent from the text of D1 and this was not contested.

2.6 According to feature e) of claim 1, the secondary particles have pores of diameter of 5 to 30 nm. There is no reference to this being a "median" value. This feature is thus to be treated as met by anything having secondary particles for which measurements show that there are pores of diameter 5 to 30 nm. The meaning attributed by the Board to this feature thus differs both from the appellant's argument that the measured pore size was a median pore size, and the respondent's argument (in line with the decision under appeal) that this feature would be met by at least two pores of diameter 5 to 30 nm being present, which could be presumed to be the case even in the absence of measurements verifying this.

2.6.1 The description of the patent in suit does state in paragraph [0029] that according to SEM observation, "the titanosilicate catalyst according to the present invention is a particle that the primary particles are found that nearly sphere primary particles of 0.05 to 0.3 µm are contacted with one another at the part of its crystalline substance. The above fact meets the measuring result of pore distribution by the nitrogen absorption method, the catalyst according to the present invention has pores (meso pores) corresponding to the gap occurred between the primary particles of from 5 to 30 nm (50 to 300 Å), on the contrary, the particle prepared by the conventional method has no such pore structure, except of micro pores of 0.54x 0.56 nm (5.4x5.6 Å) which is inherent to the titanosilicate primary particle". This comment suggests that pore measurements by the nitrogen absorption method are possible for such catalysts and indeed were carried out.

2.6.2 Whilst the nitrogen absorption method is not described further in the patent in suit, the observation of the primary particles by the SEM method is carried out at 10 000 to 25 000 magnification (page 6, lines 20 and 21) and illustrated in the examples. The catalyst according to Example 1 has pores of from 10 to 30 nm (paragraph 0046). According to Example 3, the measured pores have a size of from 10 to 25 nm (page 8, lines 50 and 51). In Example 5, the pore distribution is measured to be from 10 to 26 nm (page 9, lines 19 and 20). In Comparative Examples 1 and 5 only micropores of 0.54 x 0.56 nm inherent to the titanosilicate primary particles were observed but no pore distribution of from 5 to 30 nm (paragraphs [0063] and [0071]). Thus, in the examples of the patent in suit explicitly a pore size distribution has been measured, but there is no indication that the measured pore sizes refer to a median pore size.

2.6.3 D1 does not disclose the presence of any mesopores for any samples, let alone that sample TS-35-AI may have any mesopores at all. The respondent argued that from the spherical crystallite size of sample TS-35-AI of 0.09 µm a pore size of 22.5 nm in the agglomerate could be calculated which lay within the claimed range. In the absence of evidence using SEM and nitrogen absorption to determine whether or not a reworked (and calcined) sample TS-35-AI methods has pores in the range from 5 to 30 nm the Board is not prepared to presume in the respondents favour that this is the case, nor that such measurements would be either impossible or meaningless. The burden of proof is here again on the respondent/opponent and it has not been discharged (T 0219/83, cited *supra*).

2.7 From the above it follows that the Board is not convinced that the TSW-1 samples described in D1 can directly be compared with the catalyst particles as claimed, nor that features a), c), and e) have been shown to be present in sample TS-35-AI of D1. Thus, on the evidence before the Board D1 has not been shown directly and unambiguously to disclose all of the features of claim 1, and claim 1 must be treated as novel over D1 (Article 100(a) EPC).

3. The opponent also objected that the subject-matter of claim 3 was not novel over Comparative example 1 of D7.

3.1 Document D7 was submitted by the respondent's letter of 23 April 2004 in reaction to auxiliary requests filed with the statement setting out the grounds of appeal. One of the appellant's requests was directed to a process claim 1 in which granted product claim 1 was explicitly incorporated into granted process claim 3, instead of by reference back. Since the appellant has not objected to its introduction, the Board sees no reason to disregard D7.

3.2 D7 describes a method for manufacturing an epoxide by oxidizing an olefin with hydrogen peroxide in the presence of a crystalline substance containing silicon oxides and titanium oxides.

3.2.1 According to Comparative Example 1, which does not illustrate the invention of D7, $Ti_2(SO_4)_3.8H_2O$, NaCl, tetrapropylammonium bromide, and H_2SO_4 are added to deionized water and agitated (solution A). Water glass is mixed with deionized water (solution B). NaCl, tetrapropylammonium bromide, NaOH and H_2SO_4 are added to deionized water and mixed (solution C). The above solutions A and B are simultaneously injected into the solution C by keeping solution C at a pH at 10 to 10.5. The autoclave is heated to 150°C and then 6 hours to 210°C. The samples are washed and a calcination at 550°C under air is carried out. The product is subjected to an ion-exchange treatment and then calcined for three hours at 470°C and then used as catalyst. The average size of fine crystals produced, when measured using a scanning electron microphotograph, was 30,000 Å (3µm).

3.3 The respondent argued that Comparative Example 1 disclosed the same process as claimed, since the pH of the reaction mixture was decreased by raising the temperature to 210°C for a longer period. Furthermore, in Figure 2 agglomerated particles were shown which met the features of the claimed product of claim 1. However there is no information in D7 that the method described in this example actually produces primary particles which are combined together forming secondary particles, wherein the combined part of the primary particles is a crystalline substance, and said secondary particles have pores of diameter 5 to 30 nm, as is required by the method of claim 3 by its reference back to claim 1. The burden of showing this is on the respondent/opponent, and cannot be regarded as discharged by merely a reference to Figure 2 of D7 which shows nothing clearly.

3.3.1 It is further not clear whether or not in Comparative Example 1 the pH value is indeed decreased to 5 to 10 as would be required by claim 3: this again would require experimental proof which has not been provided. Further, the process of claim 3 requires a titanium oxide compound which according to the description of the patent in suit are normally used in the form of a tetraalkyl orthosilicate and a tetra alkyl orthotitanate or a hydrolytic halogenated titanium compound such as $TiOCl_2$, respectively (page 3, lines 10 to 13). The specified titanium compounds always include a Ti-O bonding and are present in the form of a Ti(IV) compound. Contrary to this, the starting titanium compound in comparative example 1 is $Ti_2(SO_4)_3.8H_2O$, which is a Ti(III) compound and cannot be regarded as a titanium oxide compound. Thus, the first step of the claim 3 process is not fulfilled by the disclosure of Comparative Example 1.

3.3.2 Thus, D7 cannot be considered as directly and unambiguously disclosing the subject-matter of claim 3, and novelty over D7 must be acknowledged.

3.4 Since the opposition division has not yet decided on inventive step, the Board exercises its discretion to remit the case to the department of first instance (Article 111(1) EPC), so that the parties preserve their right to appeal on this issue, if they should desire to do so.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance for further prosecution.

The Registrar The Chairman

C. Eickhoff S. Perryman