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**Datasheet for the decision
of 16 October 2012**

Case Number: T 0685/09 - 3.3.05

Application Number: 97921275.0

Publication Number: 902758

IPC: C01B 33/193, H01M 2/16

Language of the proceedings: EN

Title of invention:
Amorphous precipitated silica

Patentee:
PPG Industries Ohio, Inc.

Opponent:
Evonik Degussa GmbH

Headword:
Precipitated silica/PPG INDUSTRIES OHIO, INC.

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

Keyword:
"Novelty (yes) (main request)"
"Remittal (yes)"

Decisions cited:
T 0793/93, T 0464/94, T 0677/91

Catchword:
-



Case Number: T 0685/09 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 16 October 2012

Appellant: PPG Industries Ohio, Inc.
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 7 January 2009
revoking European patent No. 902758 pursuant to
Article 101(3) (b) EPC.

Composition of the Board:

Chairman: G. Raths
Members: H. Engl
S. Hoffmann

Summary of Facts and Submissions

- I. European patent EP-B-0 902 758 was granted with 5 claims.

The only independent claim reads as follows:

- "1. Amorphous precipitated silica characterized by:
- (a) a CTAB surface area in the range of from 140 to 185 m²/g;"
 - (b) a DBP oil absorption in the range of from 210 to 310 cm³/100g;
 - (c) a mean ultimate particle size in the range of from 10 to 18 nm;
 - (d) a total intruded volume in the range of from 2.6 to 4 cm³/g; and
 - (e) an intruded volume in the range of from 0.9 to 2 cm³/g for pores having diameters in the range of from 20 to 100 nm."

Dependent claims 2 to 5 define further embodiments of the precipitated silica of claim 1.

- II. The European patent was opposed under Article 100(a) EPC for lack of novelty and lack of inventive step, and also under Article 100(b) EPC (insufficiency of disclosure).
- III. The opposition division relied *inter alia* on the following documents:

D1: EP-A-0 647 591

D13: Test report on the reproduction of example 3 of D1, filed by opponent's letter dated 18 July 2008.

IV. The opposition division revoked the patent under appeal on the ground of lack of novelty having regard to D1. It was argued that - on the balance of probabilities - the silica obtainable in accordance with example 3 of D1 and as reworked by the opponent (D13) fulfilled the characteristic parameters of claim 1 of the opposed patent.

V. The patentee (henceforth: the appellant) filed a notice of appeal by letter dated 17 March 2009. The grounds of appeal, received by letter dated 18 May 2009, were accompanied by new claims constituting a first, second and third auxiliary request, as well as the new document

D19: Declaration of Mr James L. Boyner, dated 15 May 2009.

VI. The independent claims of said auxiliary requests are worded as follows:

Auxiliary request 1:

Claim 1 differs from claim 1 as granted in that subparagraph (c) reads:

"(c) a mean ultimate particle size in the range of from **13** to 18 nm;"

Auxiliary request 2:

Claim 1 differs from claim 1 as granted in that sub-paragraph (c) reads:

"(c) a mean ultimate particle size in the range of from **13** to **16** nm;"

Auxiliary request 3:

Claim 1 differs from claim 1 as granted in that it contains after sub-paragraph (e) the following additional sub-paragraph:

"(f) **an SR₁₀ of less than 0.025 Ω .m** "

VII. The respondent (henceforth: the opponent) filed its observations by letter dated 3 December 2009. A further submission dated 10 September 2012 included

D20: Test Report ("*Prüfbericht A120014305*") by Aqura GmbH, dated 22 August 2012.

VIII. A further submission of the appellant was received by letter dated 11 September 2012, containing

D21: Experimental Report (reproduction of example 3 of D1) (pages 6 and 7 of the letter of 11 September 2012)

IX. Oral proceedings before the board took place on 16 October 2012.

X. The appellant essentially argued as follows:

D1 concerned precipitated silica broadly characterised by 9 parameters according to claim 1 of D1. Two of them (CTAB surface area and DBP oil absorption) were also used to define the silica claimed in the patent in suit. Although the ranges of these two parameters overlapped, the silica described in D1 did not inherently possess the remaining properties defined in claim 1 of the patent in suit. The presently claimed silica was developed for a use entirely different from the one envisaged in D1.

According to the appellant, the contested decision was wrong in applying a principle of balance of probabilities for deciding on the novelty of the claimed subject-matter. Rather, the case law consistently called for a standard of proof "beyond all reasonable doubt".

The appellant argued that the example reproduced by the respondent in D13 did not belong to the teaching of D1 made available to the public. As pointed out in declaration D19, the silica obtained according to D13 was not a silica as described and prepared in example 3 of D1, and still less the inevitable result of repeating said example. The process description of D1 offered a high degree of freedom with respect to the selection of various essential process parameters. Therefore, different silica products would be obtained depending on the conditions chosen. The appellant argued in particular that possible variations in the precipitation, the final pH adjustment and the finishing steps were process features about which little information and guidance were given in D1 and

which would significantly affect the final properties of the product. The pore volume ratio V_2/V_1 of the respondent's sample (0.293) was more than 27% higher than the range of values reported in D1 (0.2080 to 0.2299). Contrary to the respondent's assertion, the pore volume ratio V_2/V_1 disclosed in D1 was not an obvious error but an indication of a pore size distribution which was considerably different from the one of the product obtained by the respondent when reworking example 3 of D1. It was evident that the respondent had failed to select all process parameters adequately. It had in fact obtained a silica product outside the teaching of D1.

The appellant submitted a test report (D21) containing four different attempts at reworking example 3 of D1. After identical precipitation procedures, products were obtained which exhibited different values of CTAB, DBP absorption and total intruded volume, depending on the drying conditions (spray or rotary drying) and optional milling (jet milled / no milling) (see D21, Table 2). In summary, the appellant's attempts to reproduce example 3 failed, as did the respondent's. The appellant concluded that example 3 was not an enabling disclosure, as it was not reproducible without undue burden.

Therefore, novelty over D1 should be accepted.

XI. The respondent essentially argued as follows:

The opposition division had stated correctly that the claimed invention was a selection among the silica products disclosed in D1. However, the claimed silica

was characterised by arbitrarily selected parameters, the significance of which remained unclear. The skilled person would have worked in the range of overlap and would have obtained the claimed silica by, for instance, reworking example 3 of D1.

The reworking of example 3 of D1 performed by the respondent (D13) demonstrated that 8 of 9 product parameters could be reproduced with a precision of $\pm 6.5\%$. In particular, pore volume, BET surface, CTAB and DBP oil absorption differed by only 6% from the values reported in D1. As the V_2/V_1 pore volume ratio was influenced by the other product parameters, the reproduced silica sample had to exhibit the pore structure and the same V_2/V_1 value as the one reported in D1. The appellant had failed to demonstrate how it was possible to obtain a product which agreed in 8 parameters with the one disclosed in D1, yet differed in V_2/V_1 .

Therefore, D13 was a true and proper reproduction of the silica according to example 3 of D1. Consequently, the subject-matter of the opposed patent was not novel over D1.

As regards the mean ultimate particle size parameter, the opposition division had acknowledged the fact that the claimed range was not novel with respect to D1. In test report D20 it was established that the measurement method for this parameter as disclosed in the patent in suit was not sufficiently clear and complete for a skilled person to determine it with the precision required to decide whether or not a given embodiment fell under the claims. It was shown that, by

differently selecting the 256 particles forming the basis of the test, mean ultimate particle sizes between 10.3 and 12.7 nm could be measured. Consequently, this parameter was not suitable to establish the novelty over D1 of what was claimed in auxiliary requests 1 and 2, which defined narrower ranges of 13 to 18 and 13 to 16 nm, respectively, for the mean ultimate particle size.

XII. Requests

The appellant requested that the contested decision be set aside and the European patent maintained as granted or, in the alternative, that the patent be maintained in amended form on the basis of the claims in accordance with auxiliary requests 1, 2 or 3, filed with the statement of grounds of appeal.

The respondent requested that the appeal be dismissed.

Should the board decide to set the contested decision aside, both parties requested that the case be remitted to the department of first instance for discussion of novelty having regard to documents not considered so far, and for discussion of inventive step and sufficiency of disclosure.

Reasons for the Decision

1. Novelty

- 1.1 According to the respondent, D1 was novelty-destroying for the subject-matter of granted claim 1.

1.2 D1 discloses a precipitated silica having the following physical and chemical characteristics (see claim 1):

BET surface area: 35 to 350 m²/g;
CTAB surface area: 30 to 350 m²/g;
DBP oil absorption of 150 to 350 ml/100g;
DBP/CTAB: 1.2 to 2.4
BET/CTAB: 0.8 to 1.1
Silanol group density (V₂): 6 to 20 ml
V₂/V₁ ratio: 0.19 to 0.46;
Pore volume PV: 1.6 to 3.4 cm³/g; and
Mean aggregate particle size: 250 to 1500 nm.

D1 also discloses a process for preparing a precipitated silica having the above-mentioned properties, characterised in that alkaline silicate is reacted with mineral acid at a pH of 7.5 to 10.5 under continuous stirring until a solid suspension of 90 to 120 g/l is obtained, the pH value is adjusted to a value of 5 or lower, and the precipitated silica is removed from the suspension by filtration, washed, dried and optionally milled or granulated (see claim 2).

1.3 More specifically, example 3 of D1 concerns the preparation of an amorphous precipitated silica having the following properties (see page 8, lines 15 to 29):

BET surface area: 184 m²/g;
CTAB surface area: 165 m²/g;
DBP oil absorption: 255 cm³/100 g;
Sears number (V₂): 15.7 ml;

V_2/V_1 ratio as determined by mercury porosimetry:
 0.2080 to 0.2299;
 Total intruded volume: 2.26 cm³/g; and
 Aggregate particle size: 381 nm.

The CTAB and DBP values fall under the respective ranges given for the silica claimed in claim 1 of the opposed patent.

However, no explicit data are available from D1 regarding the intruded volume for pores having diameters in the range of from 20 to 100 nm and for the mean ultimate particle size (10 to 18 nm), said parameters further characterising the precipitated silica claimed in granted claim 1 of the opposed patent.

The question therefore arises whether or not the silicas disclosed in D1, and in particular the silica of example 3, implicitly exhibit these additional characteristics as well.

1.4 To determine these additional parameters and to substantiate its assertion of lack of novelty, the respondent filed a test report (D13) containing a reworked example 3 of D1.

The precipitated silica produced according to D13 exhibited the following properties, in comparison with the respective values reported in D1:

	D1, Example 3	D1 reproduced by respondent (D13)	EP-B1-902 758, Claim 1
BET (m ² /g)	184	177	-

CTAB (m ² /g)	165	157	140 - 185
DBP (ml/100 mg)	255	269	210 - 310
DBP/CTAB	1.545	1.615	-
Sears V ₂ (ml/5g)	15.7	16.7	-
Grindability (µm)	8.7	9.2	-
Aggregate size (nm)	381	358	-
Hg - pore volume of 7 to 500 bar (ml/g)	2.26	2.41	-
Pore volume ratio V ₂ /V ₁	0.2080 to 0.2299	0.293	-
Primary particle size (nm)	-	11.679	10 - 18
Hg - total pore volume from 0.1 to 227 MPa [cm ³ /g]	-	3.59	2.6 - 4

1.5 The respondent concluded that all measured parameters fell, with reasonable margins of experimental error, within the respective ranges defined in claim 1 of the opposed patent.

1.6 With respect to the pore volume ratio parameter V₂/V₁ (0.293), which deviates significantly (by approximately 27%) from the values reported in example 3 of D1 (0.2080 to 0.2299), the respondent argued that V₂/V₁ ratios as determined by mercury porosimetry were always quoted to three decimals only. Thus the values given in D1 were obviously erroneous and should correctly read 0.280 to 0.290 (see D13, page 4, footnote 1).

The board does not find this explanation plausible, for the following reasons. The V₂/V₁ parameter is not measured directly, but calculated from measured values. As such, it could well be quoted to four decimals, the

degree of significance of the last digits of course being doubtful. The range of from 0.2080 to 0.2299 is not in contradiction with other parts of the disclosure of D1 concerning the V_2/V_1 ratio, in particular Figure 5 and claim 1 (0.19 to 0.45). It is therefore not obvious that an error exists. Even if one assumed - hypothetically - that an error existed, the board sees no cogent reason as to why the correct values should be those proposed by the respondent.

So, there is nothing to suggest to the skilled person that the V_2/V_1 ratio of from 0.2080 to 0.2299 was incorrect.

- 1.7 Therefore, the fact remains that the respondent apparently found a significantly different V_2/V_1 ratio from that in D1. According to the appellant this was a clear indication that a product different from the one described in example 3 of D1 had been obtained.

The board finds this argument plausible. In fact, it was common ground that certain gaps in information in D1 concerning the preparation method, and which needed to be filled by the skilled person's general knowledge, for instance the exact stirring times, the stirring speed, the exact pH value up to which sulphuric acid is added, and the aging time (if any), could give rise to such deviations in the final product. It was also accepted by both parties that the properties of the precipitated silica were sensitive to slight variations in the silica precipitation process. In conclusion, one cannot accept D13 as a true reworking of the prior art, as in fact a different product from the one described in D1 was obtained.

In the board's view, the case is similar to the situation in decision T 793/93 of 27 September 1995 (see Reasons, point 2.3.3). In this decision, the board ruled that an experimental report did not represent a true reworking of the relevant disclosure of a document. It followed from a certain product property (the tensile strength of 1.53 N/tex indicated in this report for the fibre) that the process conditions had to have been different from those applied according to the said document which disclosed a different tensile strength of only about 0.9 N/tex. Therefore, according to the board in T 793/93, it could not be concluded that by adhering to the prior art's disclosure one would inevitably arrive at a product (a fibre) having the characteristics claimed.

- 1.8 Furthermore, in the board's view the skilled person, confronted with the situation that this first attempt of reworking example 3 of D1 yielded a silica product having different characteristics, would try to modify the preparation process so as to arrive precisely at the product sought after, namely the silica having the characteristics disclosed in example 3 of D1.

By suitable adjustments to the preparation process of example 3, within the limits of the disclosure of D1, it may then be possible to arrive at a sample of precipitated silica having the desired V_2/V_1 ratio in the range of from 0.2080 to 0.2299. However, in all likelihood the remaining characteristics, such as CTAB, DPB and pore volume, would also be affected by the changes in the preparation process, so that the silica thus obtained would differ from the product obtained in

accordance with D13 also in these other characteristics. The respondent conceded that the V_2/V_1 pore volume ratio was influenced by the other product parameters, such as CTAB and BET area and pore structure (see letter of 3 December 2009, page 5, third paragraph). In the board's opinion, a sample of reproduced silica having a V_2/V_1 value different from the one obtained in D13 would also exhibit a different pore structure. Therefore, it cannot be concluded from D13 that a silica sample, prepared strictly according to example 3 of D1 and having a V_2/V_1 ratio in the range of from 0.2080 to 0.2299, would necessarily exhibit all the characteristics falling under the definition of claim 1 of the patent in dispute.

- 1.9 It is settled EPO case law that, for a prior document to be novelty-destroying, it must directly and unambiguously disclose subject-matter falling under the claim. See for instance T 464/94 (of 21 May 1997, Reasons, point 16); and T 677/91 (of 3 November 1992, Reasons, point 1.2).

As shown above, an unambiguous disclosure was not proven for D1, either on its own or implicitly, taking into account the additional experimental evidence filed as D13.

In this context, the board is not convinced by the argument of the opposition division in point 2.6 of the contested decision according to which it was unlikely that the example reworked by the respondent (opponent) which exhibited the parametric conditions relative to the CTAB and DBP values would not fulfil the remaining porosity and granulometry restrictions of claim 1.

Therefore, sufficient proof for denying the novelty of the claimed subject-matter was accepted on a balance of probabilities. The board considers, in conformity with the case law, that in assessing novelty the deciding body should apply the criterion of conviction rather than balance of probability (see T 464/94, *l.c.*).

- 1.10 The respondent argued that the V_2/V_1 pore volume ratio did not belong to the product parameters characterising the silica defined in claim 1 of the patent in suit. The reworking (D13) of example 3 of D1 would in any case take away the novelty of the claimed subject-matter, because the product obtained showed all the product characteristics required by claim 1, even if it differed in one particular characteristic (i.e. the pore volume ratio not forming part of the claim definition) from the silica disclosed in example 3.

In the board's view this argument must fail, because it presupposes that the content of D13 formed part of the prior art. However, D13 belonged to the prior art only if it were a true reproduction of the disclosure of D1, which is not the case.

- 1.11 As D1 does not directly and unambiguously disclose the claimed product, novelty over D1 must be conceded. This finding applies to the subject-matter of claim 1 in accordance with the main request, to the subject-matter of the claims dependent thereon and, due to the additional restrictions in the respective claims, also to the auxiliary requests on file.

As far as D1 is concerned, the subject-matter of claim 1 of the main request and of the subordinate

requests meets the requirements of Article 54(1) and (2) EPC.

Consequently, the contested decision cannot stand.

2. Remittal

The board notes that the impugned decision is only based on the finding of lack of novelty having regard to document D1. The board also observes that in appeal proceedings the parties have argued exclusively on this particular novelty issue.

There are, however, a number of other important issues pending, *inter alia* the alleged lack of novelty having regard to document D2 (EP-A-407 262) and an alleged anticipation by prior use (based on documents D3, D4, D9, D10 and D11), none of which have so far been considered in the first-instance proceedings. Furthermore, the objections of insufficiency of disclosure and of lack of inventive step also need to be examined.

Under these circumstances and in view of the express requests to this effect made by both parties, the board finds it appropriate to exercise its discretion under Article 111(1), second sentence, EPC, and to remit the case to the department of first instance for further prosecution.

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 for further prosecution.

The Registrar

The Chairman

C. Vodz

G. Raths