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Datasheet for the decision of 21 March 2017

Case Number: T 0472/14 - 3.3.03

Application Number: 07728114.5

Publication Number: 2004704

IPC: C08F4/22, C08F4/16, C08F10/02

Language of the proceedings: EN

Title of invention:

CHROMIUM-BASED CATALYSTS

Patent Proprietor:

TOTAL RESEARCH & TECHNOLOGY FELUY

Opponent:

Ineos Europe AG

Headword:

Relevant legal provisions:

EPC Art. 100(b), 111(1)

Keyword:

Grounds for opposition - insufficiency of disclosure (no) Appeal decision - remittal to the department of first instance (yes)

Decisions cited:

G 0002/03, G 0003/14, T 2403/11, T 0608/07, T 0466/05

Catchword:



Beschwerdekammern Boards of Appeal Chambres de recours

European Patent Office D-80298 MUNICH GERMANY Tel. +49 (0) 89 2399-0 Fax +49 (0) 89 2399-4465

Case Number: T 0472/14 - 3.3.03

DECISION
of Technical Board of Appeal 3.3.03
of 21 March 2017

Appellant: TOTAL RESEARCH & TECHNOLOGY FELUY

(Patent Proprietor) Zone Industrielle C 7181 Seneffe (BE)

Representative: Dennemeyer & Associates S.A.

Postfach 70 04 25 81304 München (DE)

Respondent: Ineos Europe AG
(Opponent) Avenue des Uttins 3
1180 Rolle, Vaud (CH)

Representative: King, Alex

Mathisen & Macara LLP Communications House

South Street

Staines-upon-Thames, Middx TW18 4PR (GB)

Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 3 January 2014 revoking European patent No. 2004704 pursuant to

Article 101(3)(b) EPC.

Composition of the Board:

Chairman D. Semino
Members: O. Dury
R. Cramer

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Summary of Facts and Submissions

- I. The appeal by the patent proprietor lies against the decision of the opposition division revoking European patent No. EP 2 004 704.
- II. Claims 1 and 2 of the granted patent read as follows:
 - "1. A process for preparing a supported chromium-based catalyst for the production of polyethylene comprising the steps of
 - a) providing a silica-based support having a specific surface area of at least 250 m 2 /g and of less than 400 m 2 /g and comprising a chromium compound deposited thereon, the ratio of the specific surface area of the support to chromium content being at least 50000 m 2 /g Cr;
 - b) dehydrating the product of step a);
 - c) titanating the product of step b) in an atmosphere of dry and inert gas containing at least one vaporised titanium compound of the general formula selected from $R_n Ti\left(OR'\right)_m$ and $\left(RO\right)_n Ti\left(OR'\right)_m$, wherein R and R' are the same or different hydrocarbyl groups containing from 1 to 12 carbon atoms, and wherein n is 0 to 3, m is 1 to 4 and m+n equals 4, to form a titanated chromium-based catalyst having a ratio of specific surface area of the support to titanium content of the titanated catalyst ranging from 5000 to 20000 m²/g Ti."
 - "2. A process according to claim 1 , wherein if the support has a specific surface area of from at least $250~\text{m}^2/\text{g}$ and of less than $380~\text{m}^2/\text{g}$, the ratio of

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specific surface area of the support to titanium content of the titanated catalyst ranges from 5000 to $20000 \text{ m}^2/\text{g}$ Ti, and if the support has specific surface area of from at least $380 \text{ m}^2/\text{g}$ and of less than $400 \text{ m}^2/\text{g}$, the ratio of specific surface area of the support to titanium content of the titanated catalyst ranges from $5000 \text{ to } 8000 \text{ m}^2/\text{g}$ Ti."

- III. An opposition against the patent was filed, in which the revocation of the patent was requested on the grounds of Article 100(a) EPC (lack of novelty and lack of an inventive step) and Article 100(b) EPC.
- IV. The following documents were *inter alia* cited in the opposition division's decision:

E2: ASTM D3663-03 (Reapproved 2008)

E3: ASTM D4567-03 (Reapproved 2008)

E4: National Institute of Standards & Technology,
Report of Investigation, Surface Area Reference
Materials 8570-8572, 1994

In that decision, which was based on the granted patent as sole request, the opposition division held that the sole information contained in the patent in suit regarding the determination of the specific surface area of the silica-based support mentioned in granted claim 1 was that it was to be measured by N_2 adsorption using the well-known BET technique. Considering that it had been shown with documents E_2 to E_3 that different results in terms of specific surface areas were obtained depending on the technique and the sample preparation used and that other determination methods than E_3 or E_3 could also be used, the opposition division concluded that the requirements of sufficiency of disclosure were not satisfied.

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- V. The patent proprietor (appellant) appealed the above decision and requested that the decision of the opposition division be set aside and the patent be maintained as granted or, alternatively, that the case be remitted to the opposition division for deciding on novelty and inventive step. Also the following document was filed:
 - E8: E.F. Vansant et al., Characterisation and chemical modification of the silica surface, Studies in Surface Science and Catalysis, Vol. 93, 1995
- VI. With its rejoinder to the statement of grounds of appeal the opponent (respondent) requested that the appeal be dismissed.
- VII. With letter of 20 February 2017 the appellant submitted
 - E9: Aerosil® 300, product information, Evonik
- VIII. In a first letter dated 21 February 2017 the respondent mentioned documents ASTM D1993-03(2008) (E11) and ISO 9277 (E12), whereby E11 had already been cited in the notice of opposition. In a second letter dated 21 February 2017 the respondent submitted
 - E10: Aerosil for High Solid-Coatings, Technical information 1197, Evonik
- IX. During the oral proceedings before the Board, which were held on 21 March 2017 in the presence of both parties, the appellant requested that the decision of the opposition division be set aside and that the case be remitted to the first instance for further

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prosecution.

X. The appellant's arguments, insofar as relevant to the present decision, may be summarised as follows:

Granted patent - Sufficiency of disclosure

- (a) The patent in suit taught that the specific surface area was to be measured by N_2 adsorption using the BET technique, which was well-known in the art as shown by E2, E3 and E8. Although it was shown in E4 that the methods of determination by BET of E2 and E3 gave different results, those differences were not significant in view of the repeatability and reproducibility reported therein. Besides, the method of E3 was based on an approximation of the BET equation and therefore led to less accurate results than E2. Also, considering that the patent taught to use the BET technique, E3 would not be considered since it obviously deviated from the BET equation. In both E2 and E3 the sample pretreatment included a preheating at 300°C, which was known from e.g. E8 to be appropriate. In that respect, it was further derivable from the wording of granted claim 1 and from the patent in suit that the surface area referred to was that of the support without chromium.
- (b) There was no evidence on file that the BET determination method according to E2 led to wrong results in terms of specific surface area. Nor had any evidence been submitted showing that other standard methods would provide different results, in particular as compared to E2.

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- (c) E4 merely showed a slight discrepancy between the specific surface area determined with E2 or E3. It had not been shown that the ambiguity was such that it amounted to a lack of sufficiency.
- (d) All the skilled person had to do to carry out the process step a) according to granted claim 1 was to use a silica-based support having a specific surface area within the range indicated therein. Such supports were described in the patent in suit and commercially available as shown by e.g. E9 and E10.
- (e) The respondent's arguments were related to the definition of the claim boundaries, which was at most an issue of clarity but not of sufficiency of disclosure as derivable from several decisions cited in Case Law of the Boards of Appeal of the EPO, 8th edition, 2016. In particular it had not been shown that the alleged ambiguity was such as to amount to a lack of sufficiency of disclosure.

Remittal

- (f) Considering that the issues of novelty and inventive step had not been addressed at all by the first instance, the case should be remitted to the first instance for further prosecution.
- XI. The respondent's arguments, insofar as relevant to the present decision, may be summarised as follows:
 - Granted patent Sufficiency of disclosure
 - (a) The patent in suit contained no indication which determination method of the surface area should be

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used and further failed to provide any information regarding the sample pretreatment and/or measurement technique.

It was derivable from the reproducibility data contained in E2 that that method itself was not very accurate and led to discrepancies of e.g. 23 % between different laboratories.

It was indicated in E4 that the temperature used for the sample pretreatment was crucial. In that respect, the pretreatment taught in E2 was not mandatorily conducted at 300°C and it was indicated in E2 that any results should be given together with the pretreatment temperature, which was not done in the patent in suit. Contrary to the appellant's conclusion, E8 did not show that the specific surface area was constant when pre-treated at high temperatures. Besides, considering that E8 was related to three specific materials and did not explicitly specify which method of determination was used, no conclusion could be drawn therefrom.

(b) E4 exemplified the discrepancy in surface area measurements obtained when using two well-defined measurement methods according to E2 and E3.

The fact that E3 was an approximation method of E2 was not relevant since the BET technique itself was based on approximations as clearly indicated in E8.

(c) Other methods than the one of E2, such as those of E11 or E12, could also be used and lead to similar conclusions. As shown by E10 the specific surface area of the product according to E9 was for instance determined using E12.

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In that respect, in Ell the temperature used for the sample pretreatment was 160°C, which was much lower than the temperature of 300°C which could be used in E2. Therefore, both methods could not lead to similar results. It was to be noted that Ell was specifically directed to precipitated silica, which was a suitable support according to the patent in suit. This further showed that different standards and/or different pretreatment temperatures should be used depending on the nature of the support, which was not indicated in the patent in suit.

- (d) The loadings of chromium and titanium indicated in granted claim 1 were defined in relation to the specific surface area. Therefore, the lack of accuracy of the determination of the specific surface area further led to the same deficiency for those features. Consequently it was neither possible to determine the scope of granted claim 1 nor to know whether the claimed benefits of the surface area and loadings could be obtained.
- (e) It was not clear from the wording of granted claim 1 whether the relevant surface area was that of the catalyst comprising chromium on silica from step a) or that of the silica support per se. In that respect, the dehydrating step b) of granted claim 1 could imply that the surface area was determined in the presence of water, in which case no pre-treatment by heating was required.
- (f) In the absence of any indications related to specific commercial products having known specific surface area, the ambiguity in terms of specific surface area could in the present case not be

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removed by reworking the examples of the patent in suit.

- (g) In view of the above the information contained in the patent in suit did not allow to determine with accuracy the specific surface area parameter mentioned in granted claim 1, which was a crucial feature of the invention according to the patent in suit. The ambiguity was so severe that it amounted to a lack of sufficiency as held in several decisions cited in Case Law of the Boards of Appeal of the EPO, 8th edition, 2016.
- (h) The deficiencies identified above were even more severe for granted claim 2, which contained narrower ranges of specific surface area.

Remittal

- (i) The remittal of the case to the first instance was not objected to.
- XII. The appellant requested that the decision under appeal be set aside and the case be remitted to the opposition division for further prosecution.

The respondent requested that the appeal be dismissed.

Reasons for the Decision

Granted patent - Sufficiency of disclosure

1. The objections of lack of sufficiency of disclosure raised by the respondent are directed to granted

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claims 1 and 2.

- 2. In order to meet the requirements of sufficient disclosure, an invention has to be disclosed in a manner sufficiently clear and complete for it to be carried out by the skilled person, without undue burden, on the basis of the information provided in the patent specification, if needed in combination with the skilled person's common general knowledge. This means in the present case that the skilled person should be in particular able to carry out the process for preparing a supported chromium-based catalyst according to claims 1 and 2.
- 2.1 In that respect, the only point in dispute between the parties is whether or not the method of determination of the specific surface area mentioned in granted claims 1 and 2 is sufficiently disclosed.
- 2.2 The respondent argued that the patent in suit did not disclose any details for the method of determination of the specific surface area specified in claim 1 and, considering that BET methods were not sufficiently accurate and that different standard methods would lead to different results, one did not know the boundaries of the claims, which amounted to a lack of sufficient disclosure.
- 2.2.1 Although there are no specific details of the determination method of the specific surface area mentioned in claim 1, it is indicated in paragraph 23 of the patent in suit that it is measured "by N_2 adsorption using the well-known BET technique". It was not contested by the respondent that the BET technique is a usual method for determining the specific surface area, as confirmed by E2, E3 and E8 (page 34, first

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sentence of section 2).

2.2.2 The respondent argued that the reproducibility of the BET method according to E2 was so poor that it amounted to a lack of sufficiency.

However, it may be seen from the reproducibility values indicated in the Table on page 5 of E2 that the lowest reproducibility (95% reproducibility interval of 23%) is achieved for the sample with the smallest specific surface area $(10.7 \text{ m}^2/\text{g})$, whereby said value is significantly lower than the one specified in granted claim 1 for the support (250 to 400 m^2/g). Those data further show that the reproducibility increases with increasing specific surface area to reach a 95% reproducibility interval of 4.1% for a sample having a specific surface area of 289 m^2/q i.e. in the range according to granted claim 1. In the Board's view, the fact that those reproducibility data are disclosed in an accepted international standard method (ASTM) for determining the surface area of catalysts and catalyst carriers shows that such a variability is acceptable by the skilled person working in that technical field. Besides, in the range of specific surface area which is relevant for the subject-matter of granted claim 1, the 95% reproducibility interval is of 4.1%, which in relation to an experimental method is not held to constitute an inacceptable lack of accuracy as argued by the respondent. In particular it cannot be considered that the accuracy of the determination method leads to an ambiguity in terms of specific surface area which is such as to amount to a lack of sufficiency.

2.2.3 The respondent argued that the patent in suit also lacked sufficiency because it failed to disclose any

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details for the method of determination of the specific surface area specified in claim 1 and E4 showed that different standard methods such as E2 and E3 led to different results.

a) Although E2 and E3 are two different ASTM standards for determining the specific surface area of catalysts and catalyst carriers (see titles), it is explicitly indicated in E3 that the multiple point BET method according to E2 is preferred to the single point determination method of E3 because the latter is based on an approximation of the BET equation. It is also indicated that the method of E3 is to be used when rapid specific surface area determinations are desired (sections 1.1, 1.2 and 11.1 of E3).

That statement is confirmed by the data related to the precision of the methods indicated in section 12.2 of E2 and section 13 of E3, which show that the most accurate method is the one of E2.

Therefore, on the basis of that information, the skilled person desiring to measure accurately the specific surface area of a support by the BET technique specified in paragraph 23 of the patent in suit, would consider E2 and not E3. Also, in view of the above, it is not surprising that it was found in E4 that the methods of E2 (based on a BET method) and E3 (based on an approximation of a BET method) led to somewhat different results. In that respect, it may be seen from the comparison of the data of the three materials studied in E4 that the difference in surface area is of maximum 5.5 % for materials having a surface area of 10.9, 158 and 291 m²/g (according to E2). Therefore, the variability between E2 and E3 is, in the Board's view, not so large that the skilled person would

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conclude that the methods of E2 and E3 lead to significantly different results.

b) Regarding the sample pretreatment of the materials to be tested, it is correct that it is indicated in E4 (pages 1 and 2: paragraphs starting with "Caution:") that the sample pretreatment is critical and that the indications given therefor in E2 and E3 must be followed.

In that respect it is indicated both in E2 (section 7.8) and E3 (sections 9.6 and 9.7) that the pretreatment should be carried out at 300 °C. Although it may be derived from "Note 3" of E2 (page 3, between sections 7.9 and 7.10) and "Note 2" of E3 (page 3, between sections 9.6 and 9.7) that lower temperatures may be used for materials which decompose at 300°C, the general teaching of E2 and E3 is that the pretreatment is usually carried out at 300°C. In that respect, the indication at section 11.2 of E2 that the pretreatment temperature should be indicated is understood as being relevant when the operator deviates from the usual temperature of 300°C. That conclusion is confirmed by the indication in E4 that a temperature of 300°C was used as taught in E2 and E3 (pages 1 and 2: paragraphs starting with "Caution:").

Therefore, when carrying out a specific surface area determination by BET technique according to either E2 or E3, the skilled person would perform a degassing (pretreatment) at 300 °C. For those reasons it cannot be concluded that it was shown that the sample pretreatment used in E2 and E3 is related to an issue of insufficiency of disclosure, as argued by the respondent.

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- 2.2.4 In view of the above the respondent's objection based on E2 to E4 is not persuasive.
- 2.2.5 The respondent further argued that the skilled person could contemplate using different BET methods than the one of either E2 or E3 to determine the specific surface area of a support, e.g. E11 or E12 and that it was to be expected that different methods led to different results.

However, there is no evidence on file that any of those other methods than E2/E3, in particular E11 or E12, effectively leads to significantly different results than E2.

In that respect, the respondent's argumentation according to which the method of E11 required a sample pretreatment at only 160°C (as compared to 300°C in e.g. E2) was not contested by the appellant. However, there is, also in that regard, no evidence on file that the method of E11 effectively leads to different results in terms of specific surface area as compared to e.g. E2. There is also no evidence that the ambiguity in the determination method would be so severe that the skilled person would not be in a position to carry out the process according to granted claim 1.

Therefore, that objection, which is not supported by facts, cannot be adhered to.

2.2.6 The respondent's objections addressed in above sections 2.2.2, 2.2.3 and 2.2.5 are all related to the issue whether the skilled person is in a position to determine unambiguously if he is working within or outside the scope of the claims. The question whether

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or not that issue effectively amounts to a lack of sufficiency or if it is an issue of clarity was the object of many decisions as indicated in Case Law of the Boards of Appeal of the EPO, 8th edition, 2016, II.C.4.5, 5.6.5, 5.6.8 and 7.2, some of which have been relied upon by the parties during the oral proceedings before the Board. As indicated therein, an ambiguity of a parameter in the claims is not enough in itself to deny sufficiency of disclosure and the question whether said ambiguity leads to insufficiency of disclosure is to be decided on a case-by-case basis (see e.g. reference to T 2403/11 and T 608/07 on pages 338 and 359 in the Case Law, supra). Rather, with respect to sufficiency the relevant question is whether the patent in suit provides sufficient information which enables the skilled person when taking into account common general knowledge to reproduce the invention (see e.g. reference to T 466/05 on page 359 in the Case Law, supra).

In the present case, it is concluded in view of the considerations given in above sections 2.2.2 and 2.2.3 that it was not shown that the skilled person would have any difficulty to identify silica-based supports having a suitable specific surface area in order to carry out the process of granted claim 1, whereby said specific surface area is determined according to any suitable BET method known in the art. Therefore the alleged ambiguity relied upon by the respondent was, in the present case, not shown to amount to a lack of sufficiency of disclosure. Rather, that issue could at most be related to a matter of clarity pursuant to Article 84 EPC, which however cannot be addressed at the present stage of the proceedings since the parameter in question is already present in the granted

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claims (see G 3/14, OJ EPO 2015, A102: catchword).

- 2.3 The question whether or not "the claimed benefits of the surface area and Cr/Ti loadings can be obtained", which was put forward by the respondent, is at most related to the question whether a technical effect is present over the whole scope of the claims, which in the absence of any effect mentioned in granted claim 1 is at most an issue of inventive step pursuant to Article 56 EPC rather than an issue of sufficiency of disclosure (G 2/03, OJ EPO 2004, 448: section 2.5.2 of the reasons).
- 2.4 The respondent also argued that it was not clear from the claims whether the "relevant" surface area was that of the catalyst comprising chromium on silica from step a) of claim 1 or that of the silica support per se.
- 2.4.1 However, it is derivable from the wording of claim 1 itself that for the ratios specified in claim 1 use is made of the specific surface area of the support per se (i.e. without chromium and/or titanium: see wording of granted claim 1 "specific surface area of the support"). That reading of claim 1 is further supported by the patent specification (see e.g. paragraphs 22, 23, 28, 33 and 58). It is further clear from paragraph 58 in combination with column 9 of Table 4 of the patent in suit that the expression "Surface area starting catalyst" indicated in each of Tables 1-4 of the patent in suit is related to the specific surface area of the neat silica support, not the chromium containing support obtained after step a) of claim 1.
- 2.4.2 The fact that step b) of claim 1 is a dehydrating step is not incompatible with that reading. In particular,

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there is no reason to consider that the specific surface area measurement should be made in the presence of water. In that respect, it is in particular derivable from paragraph 53 of the patent in suit that the deposition of chromium requires wetness impregnation and thus calls for a subsequent dehydrating step b).

- 2.4.3 Therefore, that argument is not persuasive.
- 2.5 The respondent's objection raised against granted claim 2 is based on the same arguments as those identified above in respect of granted claim 1. They are, for the same reasons, not convincing.
- 3. For those reasons, the respondent's objections pursuant to Article 100(b) EPC are rejected.

4. Remittal

The issues of novelty and inventive step were not addressed in the contested decision. Further considering that the appellant requested remittal to the first instance, which was not objected to by the respondent, it is appropriate to remit the case to the department of first instance for further prosecution (Article 111(1) EPC).

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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:



G. Rauh D. Semino

Decision electronically authenticated