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Datasheet for the decision of 16 September 2014

Case Number: T 1686/10 - 3.3.03

Application Number: 05714157.4

Publication Number: 1611175

IPC: C08F255/02

Language of the proceedings: EN

Title of invention:
CATALYST GRAIN SIZE

Patent Proprietor:
TOTAL RESEARCH & TECHNOLOGY FELUY

Opponents:
Ineos Sales (UK) Limited
Basell Poliolefine Italia S.r.l.

Headword:

Relevant legal provisions:
EPÜ Art. 83, 54, 56

Keyword:
Sufficiency of disclosure - (yes)
Novelty - (yes)
Inventive Step (no) - Improvement not credible - Obvious alternative

Decisions cited:

Catchword:

Europäisches Patentamt
European Patent Office
Office européen des brevets

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 1686/10 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 16 September 2014

Appellant: (Patent Proprietor)	TOTAL RESEARCH & TECHNOLOGY FELUY Zone Industrielle C 7181 Seneffe (BE)
Representative:	Bounaga, Sakina De Clercq & Partners Edgard Gevaertdreef 10a 9830 Sint-Martens-Latem (BE)
Appellant: (Opponent 1)	Ineos Sales (UK) Limited Hawkslease Chapel Lane Lyndhurst Hampshire SO43 7FG (GB)
Representative:	Smith, Julian Philip Howard Mathisen & Macara LLP Communications House South Street Staines-upon-Thames Middlesex, TW18 4PR (GB)
Party as of right: (Opponent 2)	Basell Poliolefine Italia S.r.l. Via Pergolesi 25 20124 Milano (IT)
Representative:	Giberti, Stefano Basell Poliolefine Italia S.r.l. Intellectual Property P.le Privato Donegani 12 44100 Ferrara (IT)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted on 7 June 2010 concerning maintenance of the European Patent No. 1611175 in amended form.**

Composition of the Board:

Chairman	B. ter Laan
Members:	F. Rousseau
	R. Cramer

Summary of Facts and Submissions

I. The appeals by the Patent Proprietor and Opponent 01 lie from the interlocutory decision of the Opposition Division posted on 7 June 2010 maintaining European patent No. 1 611 175 (application No. 05 714 157.4) in amended form.

II. Two notices of opposition were filed in both of which the revocation of the patent as granted in its entirety was requested on the grounds of lack of novelty and inventive step (Article 100(a) EPC), as well as insufficient disclosure (Article 100(b) EPC). Inter alia the following documents were submitted in opposition proceedings:

D1: EP-A-0 808 854
D2: JP-A-58-225105 and its translation in English
D3: EP-A-0 703 247,
D7: US-A-6 034 186 and
D8: The Ethylene Polymerization with Ziegler Catalysts: Fifty Years after the Discovery", L. L. Böhm, Angew. Chem. Int. Ed., 2003, 42, pages 5010-5030.

III. The impugned decision was based on the patent as granted (main request) and an auxiliary set of claims submitted during the oral proceedings on 28 April 2010. Claim 1 of the main request read as follows:

" A process for polymerising olefins in two liquid full loop reactors connected in series wherein different molecular weight fractions are produced in the presence of a Ziegler-Natta catalyst system, **characterised in that** the Ziegler-Natta catalyst has a particle size distribution d_{50} of less than 20 µm and greater than 5 µm."

Claim 1 of the auxiliary request corresponded to claim 1 as granted in which the particle size distribution d_{50} had been amended from "of less than 20 µm and greater than 5 µm" to "of less than 20 µm and greater than 8 µm".

IV. According to the decision, the method indicated in the patent in suit for determining the median value d_{50} of the volume distribution of the catalyst led to a significant measuring error regarding the lower value of the range defined in claim 1 of the main request. Therefore, the skilled person reading the specification was not able to carry out the invention in all its essential aspects in a reliable and reproducible manner and did not know when he was working within the forbidden area of the claims. The main request was therefore not allowable for lack of sufficiency of disclosure. This objection was overcome by the first auxiliary request, as it defined a lower limit for the median value d_{50} for which the measuring error was significantly reduced. Novelty was also acknowledged. As regards inventive step, the closest prior art was represented by D1. The problem as formulated in the patent in suit, namely to provide an improved double loop polymerization reaction process for polymodal polyolefins with a reduced number of defects in the products, with an improved unit throughput, with an improved "fluff" bulk density, with an improved comonomer incorporation, with a lower pump power consumption and an improved catalyst/polymer ratio was considered to have been solved by using the Ziegler-Natta catalysts defined in claim 1. The cited documents neither provided any incentive to solve the problem formulated above, nor to use small sized catalyst particles in double loop systems for the synthesis of

polymodal polyolefins. An inventive step was therefore acknowledged.

V. On 9 August 2010 both the Patent Proprietor and Opponent 01 lodged appeals against that decision and paid the prescribed fee on the same day. The statements setting out the grounds of appeal of Opponent 01 and of the Patent Proprietor were submitted on 14 October 2010 and 18 October 2010, respectively. The Patent Proprietor made additional submissions with letter of 27 May 2013 and filed document

D18: WO 2007/138257.

VI. The transfer of the status of opponent concerning Appellant-Opponent 1 was objected to by the Appellant-Patent Proprietor with letter of 7 May 2014. Appellant-Opponent 1 provided additional submissions concerning the above transfer with letter of 8 July 2014.

VII. In preparation of the oral proceedings, the Board issued a communication on 30 July 2014.

VIII. At the oral proceedings before the Board held on 16 September 2014, the Patent Proprietor defended the maintenance of the patent in suit on the basis of the main and auxiliary requests underlying the impugned decision. The objection to the transfer of the status of opponent concerning Opponent 01 was withdrawn.

IX. The arguments of the Patent Proprietor relevant for the present decision can be summarised as follows:

a) There was no problem for the skilled person by using laser diffraction analysis as defined in the patent in suit to measure accurately particle

sizes in the order of magnitude defined in claim 1 of the granted patent. The finding of the opposition division that sufficiency of disclosure was not given for claim 1 of the granted patent was therefore not correct.

b) Regarding inventive step, the closest prior art was constituted by the process disclosed in Example 1 of D1. Concerning the disclosure of that example, the estimation by Opponent 01 of the particle size of the catalyst used, i.e. the catalyst of Example 1 of D2, which had been made on the basis of the resin particle size disclosed to be obtained in D2, could not be relied upon. It was based on an oversimplified model that did not take into account various catalyst properties such as shape, surface area and porosity, and did not consider any dynamic effects such as heat transfer and mass transfer. Document D8 stated that "the particle-forming process is very complicated. It is influenced by the catalyst-particle structure and the reaction conditions for polymerization. Because of the complexity of this particle-forming process, there are different models to describe it". The calculation made by Opponent 01 was however unnecessary, because D2 already disclosed the average size of the support material as ranging from 50 to 200 µm before it was submitted to additional treatment steps. Those steps were not expected to have any significant reducing effect on the catalyst particle size. Thus, the relatively small particle size of the resin obtained in D2 was highly likely to be due to fragmentation of polymer particles during the polymerization process. In this respect reference was made to page 7 of D18 according to which "The

particle size distribution of the powder can be affected by many factors including the type, and particle size distribution, of the catalyst fed to the reactor, the initial and average catalyst activity, the robustness of the catalyst support and susceptibility of the powder to fragment under reaction conditions". In summary, the size of the catalyst used in Example 1 of D1 was above that defined in claim 1 of the granted patent.

c) The problem solved by the claimed subject-matter over the closest prior art was to provide a process for obtaining a polyolefin resin that, during extrusion, exhibited an improved and easier homogenisation, wherein defects in any product that was produced from pellets of that polyolefin resin were prevented or reduced, said process also providing improved unit throughput in the process, lower consumption of comonomer to obtain an equivalent density, lower pump power consumption to circulate the solids in the reactor and higher productivity. That problem was solved by the careful selection of the catalyst size as defined in claim 1. That the problem was successfully solved by the claimed subject-matter was shown by the comparative tests contained in the patent in suit. Those effects resulted from the smaller resin particle size of the resin, shown in Figure 5, which resulted from the smaller particle size of the catalyst. Surprisingly, the size of the resin particles was however not as small as expected.

d) There was no teaching in the cited documents that would have motivated the skilled person to use catalysts as defined in claim 1 in order to solve

said problem. Even if the problem solved by the claimed subject-matter were merely to provide a further process for polymerising olefins, the claimed solution would still not be obvious. D7 dealt with a process for preparing ultra high molecular weight polyethylene which could not be pelletized and D3 did not mention double loop reactors. Therefore an inventive step had to be acknowledged.

e) The same arguments were valid for the presence of an inventive step of claim 1 of the auxiliary request.

X. The arguments of Opponent 01 relevant for the present decision can be summarised as follows:

a) The closest prior was constituted by the process disclosed in Example 1 of D1 using the catalyst prepared in Example 1 of D2. Based on the size of the resin particles obtained in Example 1 of D2 and applying the equation described in D8, one could calculate that the d_{50} of the catalyst used in that example was in the range of 3.6-4.7 µm. The method of calculating d_{50} had been shown to be sufficiently accurate when applied on known systems, as well as on the example and comparative example of the patent in suit. Therefore, the claimed process differed from that of the closest prior art in that the d_{50} of the catalyst was above that disclosed in D1 and the loop reactors were stated to be "liquid full".

b) All the advantages allegedly obtained with the claimed process related to the small resin particle size and were therefore a consequence of

a small d_{50} value of the catalyst. Therefore, the problem meant to be solved by the claimed process had already been solved by the process described in the closest prior art in which even smaller catalyst particles were used. The problem to be solved by the claimed subject-matter was therefore to provide a further process for polymerising olefins.

c) The skilled person wishing to solve that problem would have found it obvious to use Ziegler-Natta catalysts with small particle size disclosed in either D3 or D7, which both related to catalysts for the commercial production of polyolefins.

d) Hence, the process according to the main request lacked an inventive step. The same arguments were valid for the process according to the auxiliary request.

XI. The Patent Proprietor requested that the decision under appeal be set aside and the patent be maintained as granted.

XII. Opponent 01 requested that the decision under appeal be set aside and that European patent No. 1 611 175 be revoked.

XIII. The party as of right (Opponent 02) pursuant to Article 107 EPC, second sentence, did not provide any submissions.

XIV. At the end of the oral proceedings, the decision of the Board was announced.

Reasons for the Decision

1. The appeals are admissible.

Main Request

The meaning of the particle size distribution d_{50}

2. According to paragraph [0018] of the patent in suit the wording "particle size distribution d_{50}" is to be understood as the particle size for which fifty percent by volume of the particles have a size lower than the d_{50}, i.e. d_{50} does not designate a particle size distribution, but rather the median value of the particles size distribution by volume. Although claim 1 does not contain any limitation in this respect, it is sufficient for the purpose of the present decision to adopt the above definition for d_{50} given in paragraph [0018] of the specification as the "median value of the particle size distribution by volume".

Sufficiency of disclosure

3. It is established jurisprudence of the Boards of Appeal that the requirement of sufficiency of disclosure is only met if the invention, i.e. as defined by the terms of the claim(s) (see Rule 43(1) EPC), can be performed by a person skilled in the art without undue burden, using common general knowledge and having regard to further information given in the patent in suit. In view of the reasons for the contested decision, the question to be answered is whether the patent in suit makes available to a person skilled in the art Ziegler-Natta catalysts having a d_{50} of less than 20 µm and greater 5 µm in order to carry out the claimed process.

3.1 In the absence of any method defined in the claims for determining a d_{50} of less than 20 µm and greater 5 µm, the present claims can be read as allowing any method of measurement that can be said to be standard in the art. In this context, it is not disputed that for example the method defined in paragraph [0018] of the patent in suit could be used, namely laser diffraction on a Malvern type analyser (Malvern 2000S), after having suspended the catalyst in cyclohexane.

3.2 The reasoning in the contested decision, based on a significant measuring error at the lower limit of the claimed d_{50} range when determining the catalyst particle size with a Malvern type analyser, does not go beyond the finding that the d_{50} and therefore the limits of the subject-matter of claim 1 as granted are ambiguously defined. An objection regarding the preciseness of the definition of the limits of a claim is however an objection under Article 84 EPC, which cannot be invoked against claim 1 as granted and can therefore not lead to the revocation of the patent in suit.

3.3 Neither of the Appellants, be it with respect to sufficiency of disclosure or inventive step, disputed that Ziegler-Natta catalysts with a d_{50} within the range of less than 20µm and greater than 5 µm as defined in claim 1 were available to the skilled person. The same holds true for any of the catalysts according to dependent claims 2 and 3 which are further defined by restricting the d_{50} range. The Board, despite the fact that the patent in suit does not provide any teaching how such catalysts with the required d_{50} should be obtained, is satisfied that the skilled person in view of his general knowledge and without exercising any

inventive ingenuity would be able to obtain or synthesize such catalysts. In this respect reference is made to D3, D7 and D8. The skilled person could for example use the catalyst of Example 1 of D7 or the synthesis and mean diameter of a known Ziegler-Natta catalyst such as that disclosed in Example 5 of D3, if necessary, after adjustment. Under these circumstances, sufficiency of disclosure is acknowledged.

Novelty

4. The objection on the ground of lack of novelty that had been raised before the first instance was not pursued during the appeal proceedings. On the basis of the parties' submissions, the Board does not see any reason to take the view that the claimed subject-matter lacks novelty.

Inventive step

Closest prior art

5. The patent in suit concerns a process for polymerising olefins in two liquid loop reactors connected in series wherein different molecular weight fractions are produced in the presence of a Ziegler-Natta catalyst and to the use of the polyolefin "obtained by" such a process, to prepare pipes. A similar process providing polyethylene resins suitable for preparing pipes is known from Example 1 of D1. The Board, in line with the impugned decision and the Appellants' submissions, considers that the process described in Example 1 of document D1 represents the closest state of the art and, hence, takes it as the starting point for assessing the presence of an inventive step.

5.1 In Example 1 of D1 it is stated that the Ziegler-Natta catalyst used is that described in Example 1 of Japanese Patent Laid Open No. JP58-225105A (D2 in the appeal proceedings). The d_{50} of that catalyst is neither disclosed in D1 nor in D2. The parties did not agree on the contents of the disclosure of Example 1 of D1. On the basis of an estimation made starting from the size of the polymeric particles disclosed in Example 1 of D2, Opponent 01 argued on the one hand that the d_{50} according to Example 1 of D1 lay below that now being claimed. On the other hand, the Patent Proprietor maintained that it lay above the present range, based on the information provided in D2 concerning the average particle size of the ground support used for the catalyst.

5.2 D2 discloses the preparation of catalysts containing a solid component obtained by grinding an aluminium trihalide, an organic compound containing an Si-O bond and a magnesium alcoholate and bringing it into contact with a tetravalent titanium compound (claim 1). In the general part of the description of D2 (see translation page 9, second and third lines from the bottom) it is stated that the average particle size of the ground material, i.e. before treatment with the titanium compound, is usually 50-200 µm. In Example 1 of D2 the actual average particle size of the ground support is however not given. Nor is there any information concerning the morphology of the ground material, or about the kind of average particle size meant. Whether and how the average particle size of the ground support is influenced by the subsequent treatment steps is also not indicated. Therefore, the general information concerning the usual size of the ground material provided in the general descriptive part of D2 does not allow any conclusion with respect to the d_{50} of the

ground material actually obtained in Example 1 of that document and, as a consequence, about that of the catalyst used in Example 1 of D1.

5.3 Opponent 1 estimated the d_{50} of the catalyst prepared in Example 1 of D2 on the basis of the information concerning the particle size distribution of the copolymer powder obtained with it (D2, Figure 1 and Table 1). That calculation does not take into account various catalyst properties (such as shape, surface area, and porosity) and any dynamic effects (such as heat transfer and mass transfer). According to D8 (page 5017, first column, second paragraph), the polymer particle-forming process is very complicated. It is influenced by the catalyst-particle structure and the reaction conditions for polymerization. The complexity of the resin particle-forming process is also confirmed by D18 (last paragraph of page 7), according to which the particle size distribution of the polyolefin powder can be affected by many factors including the type, and particle size distribution, of the catalyst fed to the reactor, the initial and average catalyst activity, the robustness of the catalyst support and susceptibility of the powder to fragment under reaction conditions.

5.4 On that basis and in the absence of any information in D2 concerning the robustness of the catalyst support and the susceptibility of the polyolefin powder to fragmentation under the reaction conditions used in Example 1 of D2, the estimation of the d_{50} by Opponent 01 - which is based *inter alia* on the unverified assumption that the catalyst is robust and the powder does not fragment- does not constitute convincong evidence of an unambiguous disclosure of the d_{50} of the catalyst particles used in Example 1 of D2.

5.5 Consequently, on the basis of the available evidence the Board concludes that D1, using the catalyst of Example 1 of D2 does not disclose a d_{50} of the Ziegler-Natta catalyst particles as defined in claim 1 of the patent in suit. Nor does D1 disclose that the loop reactors are "liquid full".

Problem solved and solution

6. Having regard to the process for polymerising olefins disclosed in Example 1 of D1, the Patent Proprietor submitted that the technical problem solved by the subject-matter of claim 1 of the main request was to provide a process for obtaining a polyolefin resin that, during extrusion, exhibited an improved and easier homogenisation, wherein defects in any product that is produced from pellets of that polyolefin resin were prevented or reduced, said process also providing improved unit throughput in the process, lower consumption of comonomer to obtain an equivalent density, lower pump power consumption to circulate the solids in the reactor and higher productivity, which was in line with the technical effect mentioned in paragraphs [0011], [0023] and [0024] of the patent in suit,

7. As a solution to that problem, the patent in suit proposes a method according to claim 1 which is characterized by the use of two liquid full loop reactors and a catalyst having a d_{50} of less than 20 µm and greater 5 µm.

8. To demonstrate that the claimed process achieved the alleged benefits, the Patent Proprietor relied upon the comparative tests provided in the patent in suit.

8.1 According to paragraph [0052] of the patent in suit a catalyst having a d_{50} of 23 µm and a catalyst having a d_{50} of 13 µm were used to produce polyethylene in order to demonstrate that a d_{50} of the catalyst of 13 µm reduces the d_{50} of the obtained polyethylene fluff, compared to a d_{50} of the catalyst of 23 µm. According to paragraph [0069] of the patent in suit "*Figure 7 shows that the 13 µm catalyst generates a smaller quantity of large fluff particles (about 1% instead of 6-8 % using 23 pm catalyst) but produces the same quantity of fines (bottom and 63 µm sieves). This fluff morphology should be advantageous, when the product is used in pipe-making, for the improvement of the inner aspect of the pipe product*." Additional advantages, namely improved unit throughput, improved incorporation of comonomer, lower pump power consumption and improvement of catalyst yield (paragraphs [0070] and [0071]) as a result of smaller resin particles being obtained are also in line with paragraph [0024] of the patent in suit.

8.2 Although the process using a catalyst having a d_{50} of 23 µm may serve as a comparative example because that value lies above the range defined in claim 1 of the patent in suit, it was not shown, nor argued, that it represents a reproduction of the process described in the closest prior art, i.e. Example 1 of D1. That this is not the case follows from the debates on the estimation of the median value of the Ziegler-Natta catalyst used in example 1 of D1 (see above points 5.1 to 5.5) and the fact that a split between the two loop reactors for the tests of the patent in suit (reactor ratio in weight of high molecular weight fraction to weight of total product of about 50%) is different from that used in example 1 of D1 (30 wt%). Therefore, the

comparative tests submitted cannot as such constitute a direct proof that the process according to claim 1 of the patent in suit would provide the alleged benefits in comparison to the closest prior art.

8.3 The Patent Proprietor was of the opinion that the experimental tests presented in the patent in suit demonstrated that the alleged benefits resulted from a careful selection of the d_{50}. In this respect, it can be seen from Figure 4 of the patent in suit that both catalysts tested differ not only in the d_{50} (13 and 23 µm), but also in the shape of the particle distribution curve, the curve of the catalyst with a d_{50} of 13 µm being more asymmetrical than the one of the catalyst with a d_{50} of 23 µm. Apart from the graph of the particle size distribution of the two catalysts, expressed in wt.% as a function of particle size, and the indication of the corresponding d_{50} values, the tests do not contain any indication concerning the nature or the structure of the two catalysts tested.

8.4 Furthermore, in view of D8 and D18 (see point 5.3 above), the particle size distribution of the obtained polymer fluff can be affected by many factors including the type and particle size distribution of the catalyst fed to the reactor, the initial and average catalyst activity, the robustness of the catalyst support and the susceptibility of the powder to fragmentation under reaction conditions. It follows from the above that the comparison offered in the patent in suit is not sufficient to demonstrate credibly that a d_{50} of the catalyst as defined in claim 1 would be critical to obtain a d_{50} of the polymer fluff leading to the alleged improvements mentioned in point 6 above. In other words, in the absence of any further structural

elements defining the catalyst, such as its particle size distribution, the mere definition of a d_{50} of the catalyst does not allow the conclusion that the catalyst defined in claim 1 of the patent in suit necessarily provides smaller fluff size and therefore the improvements indicated in the patent in suit are correlated with it. Hence, the criticality of using a d_{50} of the catalyst within the range defined in claim 1 as granted has not been demonstrated under the reaction conditions used in the experimental part of the patent in suit, let alone in a different context such as that of Example 1 of D1, for which no experimental evidence has been presented.

8.5 According to the jurisprudence of the Boards of Appeal, each of the parties to the proceedings carries the burden of proof for the facts it alleges (Case Law of the Boards of Appeal of the European Patent Office, 7th edition, 2013, III.G.5.1 and III.G.5.2). If a party whose arguments rely on these alleged facts does not discharge its burden of proof, this goes to the detriment of that party. Consequently, the allegation that the effects mentioned in paragraphs [0011], [0023] and [0024] of the patent in suit resulted from a careful selection of the d_{50} as defined in claim 1 as granted is not adequately supported by the evidence submitted with the consequence that it cannot be taken into account.

8.6 Since in the present case the alleged improvements lack the required experimental support, the technical problem as defined in point 6 above needs to be reformulated.

8.7 In view of the available evidence, the problem underlying the patent in suit is to be seen as to

provide a further process for polymerising olefins over the closest prior art.

Obviousness

9. It remains to be decided whether or not the proposed solution to the above-defined problem is obvious in view of the state of the art.

9.1 As shown above, the mere indication of a numerical range for the d_{50} to define the catalyst used in present claim 1 is neither critical nor can it be seen as a purposive choice for solving the problem underlying the patent in suit. Moreover, as indicated in above point 3.3, it could be acknowledged that the skilled person, despite the fact that the patent in suit does not provide any teaching how Ziegler-Natta catalysts with a d_{50} within the range of less than 20 µm and greater than 5 µm should be obtained, in view of his general knowledge would be able to obtain or synthesize such catalysts.

9.2 On this basis, the arbitrary selection of a Ziegler-Natta catalyst as defined in present claim 1 can only be seen as lying within the routine activity of the skilled person faced with the problem of providing a further process for polymerising olefins and thus cannot provide the claimed process with any inventiveness.

9.3 The argument of Opponent 01 that the skilled person would be implicitly taught to operate the loop reactors of D1 liquid full, since operating loop reactors that are not liquid full would be of no industrial interest was not contradicted by the Patent Proprietor. Hence,

this feature does not confer any inventive character to the process of claim 1.

10. As a result, the main request of the Appellant-Patent Proprietor is not allowable for lack of an inventive step.

Auxiliary Request

11. The arguments of the Patent Proprietor in support of the claims of the auxiliary request which differ from those of the main request exclusively in that the lower limit of the particle size distribution d_{50} has been amended from "greater than 5 µm" to "greater than 8 µm" are the same as those in support of the main request.

11.1 As concluded for the catalyst defined in claim 1 of the main request and for the same reasons as indicated in points 8.2 to 8.4 above, it is not credible that the selection of a Ziegler-Natta catalyst merely defined by a d_{50} of less than 20 µm and greater 8 µm would bring about the effects mentioned in paragraphs [0011], [0023] and [0024] of the patent in suit. Therefore, the considerations having regard to the assessment of inventive step given in points 8.7 to 9.3 above with respect to claim 1 of the main request, also apply to claim 1 of the auxiliary request.

12. Thus, the auxiliary request is also not allowable for lack of an inventive step pursuant to Article 56 EPC.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar: The Chairman:

E. Goergmaier B. ter Laan

Decision electronically authenticated