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Datasheet for the decision
of 14 June 2019

Case Number: T 1596/16 - 3.3.05
Application Number: 09762550.3
Publication Number: 2322322
IPC: B24B37/00, C09K3/14, H01L21/304, C01F7/02
Language of the proceedings: EN

Title of invention:
ALUMINUM OXIDE PARTICLE AND POLISHING COMPOSITION CONTAINING
THE SAME

Patent Proprietor:
FUJIMI INCORPORATED

Opponent:
Sasol Germany GmbH

Headword:
Aluminum oxide/Fujimi

Relevant legal provisions:
EPC Art. 100(b)

Keyword:
Sufficiency of disclosure (yes)
Decisions cited:
T 2399/10, T 1164/11, T 1329/11, T 0797/14, T 2220/14

Catchword:
Case Number: T 1596/16 - 3.3.05

DECISION
of Technical Board of Appeal 3.3.05
of 14 June 2019

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Decision under appeal: Decision of the Opposition Division of the European Patent Office posted on 6 May 2016
revoking European patent No. 2322322 pursuant to Article 101(3)(b) EPC.
Composition of the Board:

<table>
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<tr>
<th>Chairman</th>
<th>E. Bendl</th>
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<td>Members:</td>
<td>S. Besselmann</td>
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<td>A. Jimenez</td>
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Summary of Facts and Submissions

I. The present appeal lies from the decision of the opposition division to revoke European patent No. EP 2 322 322. The patent in suit concerns aluminum oxide particles and a polishing composition containing the same.

II. In its decision, the opposition division dealt with the ground for opposition pursuant to Article 100(b) EPC and found that the invention was not sufficiently disclosed because how to obtain the claimed aluminum oxide particles was not known. This finding applied to the patent as granted, as well as to the patent in amended form according to then pending auxiliary requests 1-5.

III. In the decision under appeal the following documents, inter alia, were referred to:

D11a machine translation of D11

IV. The patent proprietor (appellant) lodged an appeal against this decision. With its grounds of appeal, the appellant maintained the claims as granted as its main request and filed a new auxiliary request 1, which contained, in addition to the claims of the main request, an amended page 6 of the description. The five auxiliary requests previously on file were modified by including the same amended description page and were
maintained as auxiliary requests 2-6.

The following additional documents were submitted with the grounds of appeal:


D14    Römpp Chemie Lexikon, 9. Auflage, p. 4871

The statement of grounds of appeal also included experimental results (pages 6-7; pages 12-13).

V.    A further document was filed by the appellant on 30 August 2017:


VI.    The opponent (respondent) replied to the statement of grounds of appeal and requested that the appellant's experimental results be disregarded because they were incomplete and could not therefore be verified.

In addition to objections under Article 100(a) and (b) EPC, the respondent raised objections under Rule 80 and Article 123(2) and (3) EPC against the auxiliary requests.

VII.   On 13 May 2019, in reply to the summons to oral proceedings, the respondent filed document D16:

The respondent also requested that D12-D15 be disregarded.

VIII. The only independent claim of the patent as granted (main request) relates to aluminum oxide particles and reads as follows:

"1. Aluminum oxide particles characterized by primary particles each having a hexahedral shape and an aspect ratio of 1 to 5."

Claims 2-9 define preferred embodiments.

IX. It was common ground between the parties that the claimed aluminum oxide particles could be produced by calcining a hydrated alumina having the claimed morphology and aspect ratio, the morphology and aspect ratio of said hydrated alumina used as raw material being maintained during the calcination (paragraph [0037] of the patent in suit). The central question with regard to sufficiency of disclosure was the provision of a hydrated alumina useful as raw material.

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

The teaching provided in paragraph [0038] of the patent was sufficient to enable the skilled person to produce the hydrated alumina useful as raw material, namely boehmite, having the desired hexahedral shape and aspect ratio. Each of the various process conditions
considered essential in the first-instance decision either was not relevant or was taught by the patent in suit.

XI. The respondent's arguments, as far as relevant to the present decision, may be summarised as follows:

The supplementary experiments filed by the appellant were incomplete. In the absence of a reproducible working example, the burden of proof rested on the appellant to demonstrate sufficiency of disclosure. The conditions of the hydrothermal treatment, including the purity of the starting material, the pH and the use of a nucleating agent, were essential for obtaining boehmite (hydrated alumina) having the required properties for use as raw material, but were not taught in the patent in suit. In support of its arguments, the respondent relied in particular on D11/D11a, D13, and D16, all published after the priority date of the patent in suit and treated as supplementary experimental evidence.

XII. The appellant requested that the decision under appeal be set aside and the opposition rejected, or, alternatively, that the patent be maintained in the form of one of auxiliary requests 1-6 filed with the statement of grounds of appeal.

The respondent requested that the appeal be dismissed.
Reasons for the Decision

1. Admissibility of experimental results and documents

1.1 Experimental results filed by the appellant (admitted)

1.1.1 The provision of experimental data in the statement of grounds of appeal is to be seen in the present case as a reaction to the decision of the opposition division with a view to supporting the appellant's arguments on sufficiency of disclosure. This fact on its own may in the present case support admissibility into the proceedings.

1.1.2 The respondent additionally requested that said experiments be disregarded due to them being incomplete, in that for example the precise starting materials were unknown, making it impossible to repeat and verify the experiments. It also pointed out that the resulting aspect ratio had not been indicated.

1.1.3 Indeed, no detailed experimental protocol has been provided. However, there is no reason why it would not be possible to carry out the steps, per se, indicated on page 6 and pages 12-14 of the statement of grounds of appeal, namely providing a gibbsite material having a primary particle size less than 10 µm, preparing an aqueous slurry containing 10% by mass, respectively 18.75% by mass, of the gibbsite particles, and subjecting it to hydrothermal treatment at 200 °C for 4 hours in an autoclave. The respondent's objection regarding the unknown nature or source of the gibbsite used concerned the assumed relevance of its properties (such as purity) for the morphology of the resulting boehmite. The respondent did not raise doubts that
gibbsite having a primary particle size less than 10 µm was available.

1.1.4 The arguments put forward by the respondent thus concerned the question of the success of the method steps described in the statement of grounds of appeal, and not difficulties to repeat the indicated method steps as such.

1.1.5 There is no prima facie evidence to deny the success of these method steps. Specifically, it does not derive from the results presented (see the figure on pages 7 and 13 of the statement of grounds of appeal) that the resulting particles, described to be hexahedral, would not exhibit the required aspect ratio, in contrast to the very purpose of these experiments.

1.1.6 The respondent's arguments concern the question of the significance of the experimental data and therefore need to be considered when assessing the objection of sufficiency of disclosure as to its merits, but in the present case they do not represent a reason to disregard the experiments from the outset for being irreproducible.

1.1.7 The experimental results are admitted into the proceedings (Article 12(4) RPBA).

1.2 D12-D15 filed by the appellant (admitted)

1.2.1 The filing of D12-D14 in the statement of grounds of appeal is also to be seen as a reaction to the decision of the opposition division.

1.2.2 D12 and D14 are excerpts from standard textbooks and thus merely reflect common general knowledge.
1.2.3 D13 is a later application by the appellant. It does not constitute prior art but may be considered as supplementary experimental data. D13 is similar to the patent in suit in that it also aims at the provision of boehmite having hexahedral shape, see paragraph [0005]. In fact, both the appellant and the respondent considered it relevant and referred to D13 when presenting their arguments.

1.2.4 D15 is cited to show the phase diagram of the Al₂O₃ - H₂O system. It has been filed in reply to the respondent's objection that the pressure was an essential feature of the hydrothermal treatment process, and thus constitutes a reaction to the course of the proceedings.

1.2.5 In summary, D12-D14 are admitted into the proceedings (Article 12(4) RPBA), as is D15 (Article 13(1) RPBA).

1.3 The appellant requested that D16 be disregarded because it was late filed and did not constitute prior art.

1.3.1 However, D16 is relied upon as supplementary experimental data. It is relevant for the assessment of sufficiency of disclosure in that it describes the hydrothermal synthesis of boehmite crystals of various shapes. It does not raise any complex new issues and is considered as complementing the experimental results provided by the appellant.

D16 is therefore also admitted into the proceedings (Article 13(1) and (3) RPBA).
Main request

Sufficiency of disclosure

2. Sufficiency of disclosure, burden of proof

2.1 The respondent acknowledged that the burden of proof in opposition proceedings is generally on the opponent but argued that this did not apply to the present case where the patentee claimed a result, without providing sufficient details to repeat the synthesis, apparently relying on the Case Law of the Boards of Appeal of the EPO, 8th edn. 2016, III.G.5.1.2(c). The respondent also argued, with reference to T 1329/11, that this was a special case where the application as filed did not include a single example or other technical information from which it was plausible that the claimed invention could be carried out.

2.2 The board does not view this case as such a special case where no relevant teaching is provided. The claimed invention relates to a product, namely aluminum oxide particles having a specific shape. The patent in suit does provide instructions on how the claimed invention may be carried out, by describing - albeit in general terms - the synthesis of these aluminum oxide particles in paragraphs [0037] and [0038]. In analogy to the considerations set out under point 1.1 in relation to the supplementary experimental results, there is no reason to disregard or doubt from the outset this description of a synthesis method.

2.3 A successful objection of lack of sufficiency of disclosure presupposes that there are serious doubts substantiated by verifiable facts (Case Law of the Boards of Appeal of the EPO, 8th edn. 2016,
III.G.5.2.2). There is no reason why this requirement would not apply to the present case. The mere argument that the description of the synthesis of the boehmite used as raw material is too general to have an expectation of success does not substantiate such serious doubts.

3. Sufficiency of disclosure, teaching of the patent in suit

3.1 As indicated, the central question with regard to sufficiency of disclosure is the provision of a hydrated alumina useful as raw material, namely exhibiting the required primary particle shape and aspect ratio.

3.2 The patent in suit describes a hydrothermal synthesis step to obtain boehmite (a hydrated alumina) useful as raw material, see paragraph [0038]. According to this paragraph, a slurry containing 1-30% by mass gibbsite or bayerite particles having an average primary particle size of 10 μm or less is subjected to hydrothermal treatment at 200 °C for 4 hours.

This paragraph conveys the understanding that there are no further essential method steps, other than routine operations, and that there is, for instance, no need for a pH adjustment step or for the addition of a nucleating agent.

3.3 According to Table 1 and Figures 1-2 of the patent in suit, aluminum oxide particles having the required shape and aspect ratio have been produced.
Furthermore, the appellant has provided the indicated supplementary experimental results illustrating a hydrothermal treatment according to the method of paragraph [0038].

Following the considerations made above (see point 1.1), the available experimental results in the present case lead to the initial conclusion that the claimed aluminum oxide particles may be produced based on the teaching of paragraphs [0037] and [0038], even if no detailed experimental protocol has been provided.

3.4 No counter-experiments are available that would overturn this initial conclusion.

3.5 This initial conclusion is neither overturned by the respondent's argument that it was no trivial task to grow boehmite crystals having a specific morphology.

The reasons are the following:

3.5.1 This argument of the respondent is indeed supported by the general explanations that the hydrothermal synthesis step involves the dissolution and crystallisation of the material, and that crystal growth normally occurs preferentially along the c-direction, leading to flat particles (see Figure 15 of D16).

3.5.2 However, these general explanations as such do not allow any conclusion as to the necessary level of detail. In particular, they do not establish that further details or additional steps of the synthesis method, not described in the patent in suit, would have been essential to reproduce the invention, i.e. to
provide aluminum oxide particles within the scope of claim 1.

4. Sufficiency of disclosure, alleged gaps in the disclosure

4.1 The respondent held that the absence of details of the hydrothermal treatment step constituted gaps in the disclosure, which the skilled person would only have closed by exercising inventive skill.

The specific method details invoked by the respondent are the nature (such as purity) of the gibbsite used as starting material, the temperature, the time, the pressure, the solids concentration, the pH, the addition of a nucleating agent, and the need for mixing (shear rate).

Each of them will be individually addressed.

4.2 As regards the specific gibbsite used as starting material, the following considerations are made.

4.2.1 The board shares the appellant's view that the terms "gibbsite" and "bayerite" in the present case define the aluminum hydroxide form, namely $\alpha$-Al(OH)$_3$ (bayerite) and $\gamma$-Al(OH)$_3$ (gibbsite), and thus synthetic materials, not a natural mineral. This derives from the context, which is their transformation to alumina hydrates and aluminum oxides. In addition, even the passage on page 10 of D7, cited by the respondent on page 3 of its reply to the grounds of appeal, states that gibbsite was produced by precipitation from a potassium aluminate solution.
4.2.2 D7 has been relied upon by the respondent to show that gibbsite may contain a high level of impurities, for instance a high level of sodium (page 10, "2.21 Gibbsite"). D13 also states that powder of aluminum hydroxide often contains sodium as impurities, see paragraph [0032].

The general reference to "gibbsite particles" in paragraph [0038] of the patent in suit thus encompasses various degrees of purity of the gibbsite, in particular various sodium levels.

4.2.3 However, the mere observation in D13 that sodium ions are not desirable (paragraph [0032] of D13), and the presence of washing steps in the examples of D13, may show that a low sodium content is desirable, but these do not prove that a specific washing step or starting material purity would have been essential for obtaining hexahedral particles.

4.2.4 On the other hand, the appellant argued that the precise nature, namely the purity, of the gibbsite used as starting material would not have been relevant. In support of these arguments, the appellant performed the hydrothermal synthesis step using two different starting materials having different values of electrical conductivity, said to be indicative of different sodium content (page 6 of the statement of grounds of appeal).

Both materials have been found to be suitable.

4.2.5 Hence, even though the specific properties and in particular the sodium levels of the gibbsite materials used by the appellant are not known, these examples are nevertheless consistent with the appellant's arguments
that the selection of a certain purity (i.e. sodium content) of the gibbsite would not have been essential in order to reproduce the invention.

4.2.6 The present case is not comparable to T 797/14 or T 2399/10, cited by the respondent in view of the need to disclose the starting material.

T 797/14 concerned a case where the key element of the invention was the use of a specific coating composition. The board found that sufficiency of disclosure was lacking because the preferred and unique coating composition disclosed was a commercial product, but the composition and method of production thereof were not publicly known.

T 2399/10 concerned a case where a starting material could not be provided, not only because no method was described, but also because it was characterised by an undefined parameter and could not therefore be identified.

4.3 Temperature and time

4.3.1 The temperature and time of the hydrothermal treatment step are explicitly and precisely mentioned in the patent in suit, namely a temperature of 200 °C and a duration of 4 hours (paragraph [0038]).

4.4 Pressure

4.4.1 There is no indication of the pressure during the hydrothermal treatment step. However, this would not have prevented the skilled person from carrying out the hydrothermal treatment step; rather, it implies in the first place that the skilled person would not have been
concerned with the selection of a specific value of the pressure. As stated by the appellant, the skilled person would have normally performed the synthesis under autogenous pressure. The board concurs with the appellant that the pressure of the autoclave would not have been considered essential for the conversion taking place in the liquid/solid phase.

4.4.2 The respondent argued with reference to the phase diagram in Figure 3.1 on page 36 of D7 that, at pressures of 200 bar or above, a mixture of boehmite and diaspore would have been obtained, given the synthesis temperature of 200 °C. However, this diagram is not detailed enough to allow a clear distinction of pressure-dependent regions of the formation of only boehmite, only diaspore, and a mixture of boehmite and diaspore. Moreover, the phase diagram submitted by the appellant (Figure 1 of D15) supports its argument that, at a temperature of 200 °C, only boehmite is formed.

4.4.3 Hence, there is no evidence that the desired hexahedral particles would not have been obtained at a pressure of, for example, 200 bar.

4.5 Solids content

4.5.1 As regards the solids content, the patent in suit instructs the skilled person to select a solids content of 1-30 mass%, see paragraph [0038]. Therefore, it is not relevant that higher solids contents, such as 40 or 50 mass% Al(OH)₃, allegedly do not provide the desired hexahedral shape. Moreover, the experimental results provided by the appellant with the statement of grounds of appeal illustrate solids contents of 10 and 18.75 mass%, respectively, and thus support the range described in paragraph [0038].
4.6 pH value

4.6.1 The general method description in paragraph [0038] does not contain any pH adjustment step. The board shares the appellant's view that this would, in the first place, have been regarded as an implicit teaching that no such pH adjustment step would have been required (see also point 3.2).

4.6.2 In support of its arguments that a selection of a specific pH value would have nevertheless been essential, the respondent cites D11, D13 and D16.

4.6.3 However, D11 is not representative of the hydrothermal treatment described in paragraph [0038] of the patent in suit. D11 neither relates to the same starting material as taught in the patent in suit, nor the same temperature and duration.

Specifically, D11 describes a hydrothermal reaction of an aluminum hydroxide containing aqueous solution. In the examples of D11, said aluminum hydroxide containing aqueous solution is prepared by adding sodium hydroxide aqueous solution to aluminum sulfate aqueous solution. The aluminum hydroxide solution of D11 thus inevitably contains at least sodium and sulfate, in addition to aluminum hydroxide. Moreover, D11 does not mention that gibbsite or bayerite particles are present. The aluminum hydroxide solution of D11 thus differs from a slurry obtained by mixing gibbsite or bayerite particles with water.

The formation of different morphologies in D11, namely needle-like particles at pH < 10.5, therefore does not prove that the selection of a specific pH value would
have been essential for obtaining hydrated alumina having hexahedral shape when carrying out the hydrothermal treatment step taught in paragraph [0038] of the patent in suit.

4.6.4 D13 is similar to the patent in suit in that it discloses the hydrothermal treatment of a slurry containing gibbsite in order to prepare boehmite particles having a low aspect ratio. D13 teaches that the slurry preferably has a pH of 8 or lower, more preferably 7 or lower, and further preferably 6 or lower, see paragraphs [0007] and [0026]. D13 states that at these pH values, particles having a low aspect ratio are particularly easily obtained.

4.6.5 However, these statements do not amount to a teaching that the desired aspect ratio would not have been obtained, if there had been no adjustment of the pH value. Furthermore, while the specific results given in Table 1 of D13 show this effect of the pH value on the aspect ratio, the aspect ratio remains within the claimed range of 1-5 across the entire range of pH values examined.

4.6.6 The respondent argued that the specific results of Table 1 were not illustrative of the patent in suit, because the synthesis slurry contained an alumina sol as nucleating agent. In any event, the general teaching in D13 regarding preferred pH values does not lead to the conclusion that the selection of a specific pH value would have been essential to obtain the desired aspect ratio.

4.6.7 Similar considerations apply to D16. Figures 5 and 13, specifically highlighted by the respondent, describe hydrothermal treatments in presence of sodium hydroxide
(Figure 5) and acetic acid (Figure 13). The addition of this base or acid is not contemplated in paragraph [0038] of the patent in suit. Irrespective of the question of the influence of the significantly longer synthesis time applied in D16, namely 72 hours (see the paragraph "2.1. Method") as opposed to 4 hours taught in the patent in suit, the respondent could not identify a hydrothermal treatment step in D16 that directly reproduced the teaching of paragraph [0038] of the patent in suit but did not provide the desired result.

4.7 Nucleating agent

4.7.1 The general method description in paragraph [0038] does not mention any additives such as nucleating agents either.

4.7.2 The respondent argued in view of D13 that the presence of a nucleating agent was nevertheless essential, but that it would have required inventive skill to recognise the need for a nucleating agent, and to select a suitable one.

4.7.3 The appellant disagreed, stating that the presence of a nucleating agent merely accelerated the crystallisation reaction; it was not essential for obtaining the desired shape and aspect ratio.

4.7.4 D13, a later application by the appellant, teaches the presence of a nucleating agent as an essential feature of the invention disclosed in D13, which is a method for producing boehmite particles, see claim 1. According to paragraph [0019] of D13, the nucleating agent facilitates the control of the particle size.
4.7.5 This teaching of D13 suggests that the presence of a nucleating agent is beneficial for the claimed method of D13. However, it does not demonstrate that instructions to add a nucleating agent would have been required in the patent in suit in order to enable the skilled person to provide the claimed aluminum oxide particles.

4.7.6 The respondent's observation that the - unexplained - label "5000 ppm" on the micrographs provided in the statement of grounds of appeal (see page 7) indicated the amount of nucleating agent remains speculation. In any event, this on its own would not show that the addition of a nucleating agent would have been essential.

4.8 Mixing (shear rate)

4.8.1 The examples of D13 mention stirring of the slurry and the rotation speed applied. However, this observation on its own does not lead to the conclusion that the selection of a specific stirring step would have been essential to produce the claimed aluminum oxide particles. Moreover, it would have been a routine measure to carry out a stirring step when preparing a slurry.

4.9 In summary, the respondent has invoked the absence of various details of the hydrothermal synthesis reaction. For the above reasons, these details have either been disclosed (for instance the time, the temperature and the solids content), or have not been shown to be essential for the success of the hydrothermal treatment step.
4.10 The respondent has not attempted to conduct the hydrothermal treatment described in paragraph [0038].

Nor has the respondent identified any example in the cited documents that could have been seen as a reproduction of the method of paragraph [0038] but did not yield boehmite having the required properties.

Documents D11 and D16, which have been specifically highlighted by the respondent, differ from the relevant method description, see point 4.6.3 regarding D11 and point 4.6.7 regarding D16.

D13 is a further development of the patent in suit. In the case of D13, the desired hexahedral particles having an aspect ratio of 1-5 are obtained.

The respondent mentioned during the oral proceedings that the priority document underlying the patent in suit contained a relevant experiment where an aspect ratio of 30 was obtained, but has neither provided any further substantiation nor identified or filed the relevant part of the priority document.

4.11 Hence, there is no evidence of any gap in the disclosure, which the skilled person would only have closed by exercising inventive skill.

4.12 For the same reasons, there is no indication that the reproduction of the claimed invention would not merely have required routine experiments, as argued by the respondent with reference to T 2220/14 and T 1164/11.

4.13 Without any attempt to carry out the described synthesis step, there is also no basis to conclude that additional instructions would have been necessary to
enable the skilled person to direct the synthesis towards a desired result. Nor is there any indication that the successful reproduction of the hydrothermal synthesis would have depended on chance.

5. Sufficiency of disclosure with regard to alternative synthesis methods

5.1 The respondent also argued that paragraph [0037] described alternative raw materials for use in the calcining step, namely gibbsite, bayerite and nordstrandite, but was silent as to how these materials, having the required shape and aspect ratio, could have been obtained.

5.2 However, the claimed invention is directed to the aluminum oxide particles, not to a preparation method. In the present case, it is not relevant for sufficiency of disclosure of this claimed invention whether the general description of the preparation method includes additional variants which possibly may not be carried out, as long as the instructions enable the skilled person to produce the claimed aluminum oxide particles

6. Sufficiency of disclosure, further considerations

6.1 The question whether boehmite (AlO(OH)) is to be regarded as an aluminum oxide, as mentioned in paragraph [0020] of the patent in suit, is not seen to be relevant for sufficiency of disclosure, because boehmite as the product of the hydrothermal reaction step of paragraph [0038] may exhibit the properties required in claim 1.
6.2 The respondent also objected to the "alpha conversion rate" as being an undefined parameter.

6.2.1 The board concurs with the appellant that the "alpha conversion rate" (see claim 3) would have been understood as referring to the ratio of conversion to α-alumina, and thus corresponds to the "degree of conversion to α-Al2O₃" (corundum), mentioned in section 2.412 of D7. This understanding is supported by the relevant part of the patent in suit entitled "[w]ith respect to the alpha conversion rate" (see paragraph [0020]).

6.2.2 In the present case, the apparently missing description of the detailed measuring method in the patent in suit might therefore be a question of clarity but would not have prevented the skilled person from reproducing the invention.

7. Sufficiency of disclosure, conclusion

7.1 For these reasons, the invention as defined by the main request has been disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Article 100(b) EPC).

Remittal

8. The appealed decision solely concerned arguments with regard to sufficiency of disclosure. To give the parties the opportunity to present their case to the departments of first and second instance, the board exercises its discretion in accordance with Article
111(1) EPC and remits the case to the opposition division for continuation of the opposition proceedings, as also requested by both parties.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the opposition division for further prosecution.

The Registrar: The Chairman:

C. Vodz E. Bendl

Decision electronically authenticated