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D E C I S I O N
of 5 June 2000

Case Number: T 0202/96 - 3.3.5

Application Number: 89904674.2

Publication Number: 0409879

IPC: C09C 1/36

Language of the proceedings: EN

Title of invention:

Process for producing durable titanium dioxide pigments

Patentee:

KERR-MCGEE CHEMICAL LLC

Opponent:

Bayer AG, Leverkusen Konzernverwaltung RP Patente Konzern

Headword:

-

Relevant legal provisions:

EPC Art. 56
EPC R. 67(1)

Keyword:

"Inventive step (no): obvious modification"
"Reimbursement of appeal fee (no)"

Decisions cited:

-

Catchword:

-



Case Number: T 0202/96 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 5 June 2000

Appellant: KERR-MCGEE CHEMICAL LLC
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(Opponent) Konzernverwaltung RP
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Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 22 December 1995
revoking European patent No. 0 409 879 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: A.-T. Liu
J. H. van Moer

Summary of Facts and Submissions

- I. The appeal was from the decision of the Opposition Division revoking European patent No. 0 409 879.
- II. The impugned decision was based on claims 1 to 11 as granted. Claim 1 read as follows:

"A batch process for preparing a coated titanium dioxide pigment having deposited thereon a dense amorphous inner coating of silica and an outer coating of alumina comprising:

forming an aqueous slurry of a non-coated titanium dioxide pigment, said slurry having a pH adjusted to a value of at least 9.8 and heating said slurry to an elevated temperature from 75°C to 90°C;

adding a water-soluble silicate compound to said heated slurry while maintaining said heated slurry at said elevated temperature for from 5 to 40 minutes to initiate deposition and cure of said dense amorphous inner coating of silica on said pigment, the amount of said water-soluble silicate added being such as to provide said coating in a weight from 0.5 to 5.0 weight percent; based on the total weight of coated titanium dioxide pigment, of silica;

rapidly adjusting the pH of said heated slurry to a value of from 9.2 to 9.4 and maintaining said heated slurry at said elevated temperature for from 5 to 40 minutes to complete the deposition and cure of said inner coating of silica;

further rapidly adjusting the pH of said heated slurry to a value of from 2.8 to 3.2 and thereafter commencing addition of an amount of a

water-soluble aluminium containing compound sufficient to raise the pH of said heated slurry to a value of from 5.5 to 6.5 and to initiate a deposition of an outer coating of alumina on said silica coated pigment;

continuing the addition of said water-soluble aluminium containing compound to said heated slurry in an amount sufficient to provide an outer coating of said alumina of from 1.5 to 5.0 weight percent, based on the total weight of the coated titanium dioxide pigment, on said silica coated pigment while maintaining the pH of said heated slurry at said value of from 5.5 to 6.5; and

further adjusting the pH of said heated slurry to a value of from 6.5 to 8.5 and recovering said coated titanium dioxide pigment substantially as produced."

III. The opposition division held that the subject-matter of claim 1 did not involve an inventive step, in particular in view of the following documents:

D1: DE-A-2 740 561

D3: EP-A-73 340

IV. In summary, it was indicated in the reasons for the decision that the process according to the patent in suit was distinguished from the closest prior art D1 only in that the alumina coating was precipitated onto the silica coating in an acidic medium, instead of from an alkaline medium as in the process of D1. However, it was known from D3 that acidic alumina precipitation yielded titania pigments with advantageous properties. The opposition division held that it was therefore

obvious for the skilled person to choose the acidic precipitation route, to obtain a titania pigment with improved properties as expected.

V. With the statement of the grounds of appeal, the appellant (patentee) submitted the following reasoning in respect of inventive step:

- The object of the patent in suit was to provide a process with a relatively short processing time for preparing a coated titanium dioxide pigment having good optical and durability properties.
- The claimed process was distinguished from D1 at least in the acidic alumina precipitation and in the two-step silica curing.
- D3 was not directed to a batch process.
- Even if the skilled person had combined the teaching according to D3 with that of D1, he would not have arrived at the process of claim 1.
- The careful control of the present process parameters resulted in an improved product compared to D1.
- The enclosed comparative data showed that processing time for the claimed process was shorter than that for the process of D3.

Further to the above, the appellant criticised the fact that the weathering data filed 1 September 1995 by the opponent were not valid since they were based on only a few weeks of exterior exposure.

VI. The following affidavits were enclosed with the letter setting out the grounds of appeal:

A1: Affidavit signed 27 September 1994 by
Dr John R. Brand

A2: Affidavit signed 28 March 1996 by Dr John R. Brand

A3: Affidavit signed 5 April 1996 by Dr Jürgen Braun

A4: Affidavit signed 12 April 1996 by
Mr Garmond G. Schurr

The appellant also made reference inter alia to a new document, GB-A-1 589 070 (D1'), in support of his arguments. This document was a British patent specification corresponding to the German patent application D1.

VII. Further to the above, the appellant submitted that the opposition division had committed a substantial procedural violation since it had failed to take account of several arguments put forward by him.

VIII. With the response to the grounds of the appeal the respondent refuted the arguments of the appellant. He maintained that the process of claim 1 was clearly derivable from D1 in combination with D3 and that the improvement of the resulting product was to be expected in view of D3. The validity of accelerated tests for assessing the long term weatherability of paints was also commented upon.

IX. The opposition was withdrawn with the respondent's letter of 9 June 1998.

X. In response to the summons to oral proceedings dated 24 September 1999, the appellant filed with his letter of 23 February 2000 four new sets of claims as basis for auxiliary requests I to IV.

XI. Claim 1 of auxiliary request I had been revised to specify that the claimed process consisted of the process steps as stipulated. Claims 2 to 11 were as granted.

XII. Auxiliary request II consisted of an amended claim 1 and claims 2 to 11 as granted. Claim 1 of this request was worded as follows:

"A batch process for preparing a coated titanium dioxide pigment having deposited thereon a dense amorphous inner coating of silica and an outer coating of alumina comprising:

(a) forming an aqueous slurry of a non-coated titanium dioxide pigment, said slurry having a pH adjusted to a value of at least 9.8 and heating said slurry to an elevated temperature from 75°C to 90°C;

(b) a two-stage silica deposition and cure of the inner coating of silica, consisting of the steps of:

(i) adding a water-soluble silicate compound to said heated slurry while maintaining said heated slurry at said elevated temperature for from 5 to 40 minutes to initiate deposition and cure of said dense amorphous inner coating of silica on said pigment, the amount of said water-soluble silicate added being such as to provide said coating in a weight from 0.5 to 5.0 weight percent; based on the total weight of coated titanium dioxide pigment, of silica; and

(ii) rapidly adjusting the pH of said heated slurry to a value of from 9.2 to 9.4 and maintaining

said heated slurry at said elevated temperature for from 5 to 40 minutes to complete the deposition and cure of said inner coating of silica; then

(c) further rapidly adjusting the pH of said heated slurry to a value of from 2.8 to 3.2 and thereafter commencing addition of an amount of a water-soluble aluminium containing compound sufficient to raise the pH of said heated slurry to a value of from 5.5 to 6.5 and to initiate a deposition of an outer coating of alumina on said silica coated pigment; then

(d) continuing the addition of said water-soluble aluminium containing compound to said heated slurry in an amount sufficient to provide an outer coating of said alumina of from 1.5 to 5.0 weight percent, based on the total weight of the coated titanium dioxide pigment, on said silica coated pigment while maintaining the pH of said heated slurry at said value of from 5.5 to 6.5; and then

(e) further adjusting the pH of said heated slurry to a value of from 6.5 to 8.5 and recovering said coated titanium dioxide pigment substantially as produced."

XIII. Claim 1 of auxiliary request III had been amended with respect to claim 1 of the main request by incorporation of the subject-matter of claim 10 as granted. Claims 2 to 10 corresponded to claims 2 to 9 and 11 as granted. The relevant part of claim 1 of this request read (added feature emphasised by the Board):

".... continuing the addition of said water-soluble aluminium containing compound to said heated slurry in an amount sufficient to provide an outer coating of said alumina of from 1.5 to 5.0 weight percent, based on the total weight of the coated titanium dioxide

pigment, on said silica coated pigment while maintaining the pH of said heated slurry at said value of from 5.5 to 6.5 by simultaneously adding an acid; and further adjusting ..."

XIV. The revisions made in the previous auxiliary requests had all been incorporated in auxiliary request IV. Claim 1 of this request read as follows:

"A batch process for preparing a coated titanium dioxide pigment having deposited thereon a dense amorphous inner coating of silica and an outer coating of alumina consisting of the following steps:

(a) forming an aqueous slurry of a non-coated titanium dioxide pigment, said slurry having a pH adjusted to a value of at least 9.8 and heating said slurry to an elevated temperature from 75°C to 90°C;

(b) a two-stage silica deposition and cure of the inner coating of silica, consisting of the steps of:

(i) adding a water-soluble silicate compound to said heated slurry while maintaining said heated slurry at said elevated temperature for from 5 to 40 minutes to initiate deposition and cure of said dense amorphous inner coating of silica on said pigment, the amount of said water-soluble silicate added being such as to provide said coating in a weight from 0.5 to 5.0 weight percent; based on the total weight of coated titanium dioxide pigment, of silica; and

(ii) rapidly adjusting the pH of said heated slurry to a value of from 9.2 to 9.4 and maintaining said heated slurry at said elevated temperature for from 5 to 40 minutes to complete the deposition and cure of said inner coating of silica; then

(c) further rapidly adjusting the pH of said

heated slurry to a value of from 2.8 to 3.2 and thereafter commencing addition of an amount of a water-soluble aluminium containing compound sufficient to raise the pH of said heated slurry to a value of from 5.5 to 6.5 and to initiate a deposition of an outer coating of alumina on said silica coated pigment; then

(d) continuing the addition of said water-soluble aluminium containing compound to said heated slurry in an amount sufficient to provide an outer coating of said alumina of from 1.5 to 5.0 weight percent, based on the total weight of the coated titanium dioxide pigment, on said silica coated pigment while maintaining the pH of said heated slurry at said value of from 5.5 to 6.5 by simultaneously adding an acid; and

(e) further adjusting the pH of said heated slurry to a value of from 6.5 to 8.5 and recovering said coated titanium dioxide pigment substantially as produced."

XV. In the letter of 3 March 2000, the appellant withdrew the request for oral proceedings, requesting instead that the appeal be decided on the basis of the written submissions.

XVI. The appellant requested that the appeal fee be refunded, the decision under appeal be set aside and the patent maintained as granted (main request). Auxiliarily, the patent was to be maintained on the basis of the amended sets consisting of claims 1 to 11, or claims 1 to 11, or claims 1 to 10 or claims 1 to 10 submitted 23 February 2000 as auxiliary requests I to IV, respectively.

Reasons for the Decision

1. The Board can only set aside the decision under appeal and maintain the patent in suit if the latter meets the requirements of the EPC. Therefore, even though the respondent has withdrawn his opposition, the Board has the right, of its own motion (Article 114(1) EPC), to examine if said requirements are met. For its findings, the Board shall only take into account the submissions of the opponent prior to his withdrawal of the opposition, on which the appellant has had an opportunity to present his comments (Article 113(1) EPC)

Main request

2. *Inventive step*
 - 2.1 The process as stipulated in claim 1 is a batch process for producing a coated titania pigment comprising the following essential steps in sequence:
 - (i) precipitation of silica at a pH of at least 9.8 and a temperature from 75°C to 90°C followed by a first silica cure of 5 to 40 minutes;
 - (ii) pH adjustment to a value of from 9.2 to 9.4 and a second silica cure of 5 to 40 minutes;
 - (iii) pH adjustment to a value of from 2.8 to 3.2;
 - (iv) commencement of alumina precipitation at a pH between 2.8 and 3.2 and
 - (v) continuation of the alumina precipitation at a pH

between 5.5 to 6.5.

2.2 Selection of the closest prior art document

The decision under appeal is based on the conclusion that the subject-matter of claim 1 does not involve an inventive step with respect to D1 and D3 (see points III and IV). D1 has been introduced into the proceedings at the opposition stage whilst D3 is already acknowledged in the patent in suit (column 2, lines 7 to 34). The Board infers from the submissions of the parties, both in opposition and in appeal, that the other documents cited in the patent in suit as starting point for the claimed invention are no longer considered to be relevant in this respect. The Board shares this view and shall therefore focus on documents D1 and D3 in the following discussion.

As is noted earlier (point VI), D1 and D1' are of the same patent family. Since the latter is in the language of the proceedings, the Board, for the purpose of this decision, shall cite the relevant passages in D1' rather than citing from D1.

- 2.2.1 It is undisputed that both documents D1' and D3 relate to the same subject-matter as the patent in suit, namely the preparation of a durable titanium dioxide pigment having deposited thereon discrete layers of silica and alumina (compare patent in suit, column 1, lines 7 to 10 and D1', page 1, lines 86 to 90 and D3, page 1, first paragraph). Furthermore, both these prior art teachings as well as the patent in suit tackle the problem of long processing times of batch processes (compare patent in suit, column 1, lines 48 to 50; D1', page 1, lines 81 to 83; and D3, page 3, paragraph 2).

2.2.2 In order to achieve the above goal, D1' teaches a process wherein the silica coating is cured in a single step and the alumina coating is precipitated from an alkaline medium.

D3 in contrast discloses a process wherein the silica coating is cured in three incremental steps and the alumina precipitated from an acidic medium (see patent in suit, column 2, lines 7 to 34).

2.2.3 As is correctly pointed out by the appellant, the claims in D3 are directed to a continuous process (see letter of 29 April 1996, page 5, last paragraph). The Board also concurs with the appellant in that D3 proposes a continuous process to solve the above-mentioned problem of long processing times involved with batch processes. However, the disclosure of D3 is not limited to a continuous process. On the contrary, 17 of the 18 examples given in D3 relate to batch processes (see D3, page 11, paragraph 1 and page 12, paragraph 3). This is not refuted by the appellant.

2.2.4 In document D3, it is already recognised that an acidic precipitation of the alumina coating results in a titania pigment which has better properties than one obtained with alumina precipitation from an alkaline medium (see page 12, Table 12 and page 13, lines 1 to 3). Further, taking into consideration the fact that D3, published on 9 March 1983, is of a more recent date than D1' (or D1), published on 7 May 1981 (or 16 March 1978), the Board holds D3 to be more representative of the true state of the technical development in the field of TiO₂ pigment production at the priority date of the patent in suit than D1' and D1.

2.2.5 For the above reasons, the Board chooses document D3, with particular reference to the batch processes exemplified therein, as the most realistic starting point for discussing inventive step.

2.3 Problem and solution

The problem to be solved with respect to D3 is seen by the appellant in the reduction of processing time (see Grounds of Appeal dated 29 April 1996, page 8, paragraph 2). As factual basis for this submission, he relies on the comparative examples filed as part of the affidavit A2. These data are in conformity with those submitted on 25 August 1995 in the course of the opposition proceedings.

2.3.1 In these experiments, the retention time of each silica cure step is 20 minutes and the products have the same durability and optical properties. The appellant has then concluded that the process of claim 1, which provides pigments with only two cure steps, thus in a shorter time, represents an advance in technology over the process of D3 (see A2, point 6 and Table I).

The Board, however, holds that the patent in suit is not compared with the closest prior art as disclosed. It is true that, according to D3, it is preferable to cure the slurry for 20 to 30 minutes after each neutralisation step. However, a cure time of 5 minutes is also considered efficient (page 8, paragraph 2). In fact, in the worked Examples 1 to 14, the cure steps last only 5 minutes each, adding up to 15 minutes in total (see page 11, paragraph 1). Since this explicit disclosure is not reflected in the comparative examples submitted by the appellant, the data obtained are not

suitable for demonstrating that the processing times according to the claimed process are shorter than those of the processes disclosed in Examples 1 to 14 of D3.

By comparison, the retention time for the only example in the patent in suit is 20 minutes at each step, thus adding up to 40 minutes. This is also in conformity with the claim which stipulates a duration of up to 40 minutes for each step. In the Board's judgment, the evidence on file therefore does not justify the contention that the processing time for the process as claimed is shorter than that for the process of D3.

2.3.2 Concerning the question of product quality, the appellant has submitted results of acid solubility tests, asserting that this test is accepted by the pigment industry to predict the contribution of silica-encapsulated TiO₂ pigments to the weatherability of paint films (see Grounds of Appeal, page 7, paragraph 2 and A3, point 2). Whilst this assertion is not being challenged, the Board remarks that none of these experiments has been conducted with pigments obtained in five minutes cures as in Examples 1 to 14 of D3 (see A2, Tables I and II). The data are therefore not suitable for comparing the durability of the products made according to the patent in suit with that of D3.

2.3.3 In the course of the opposition proceedings, comparative test data have also been filed by the respondent to demonstrate that the durability of the products obtained according to D3 is as good as that of products according to the patent in suit (see letter dated 1 September 1995). The validity of these tests has been contested by the appellant on the basis that outdoor durability data obtained from a few weeks of

exposure are not reliable (see Grounds of appeal, page 6, last three paragraphs; A1, point 7; A2, point 6 and A4, point 2). However, the appellant has not argued, let alone submitted evidence that a cure time of 5 minutes as in Examples 1 to 14 of D3 does not provide a pigment with a durability comparable to that obtained according to the patent in suit. On the contrary, the declarations in A1 and A2 appear to confirm the respondent's finding that the pigments made according to the process of D3 are as good as those produced by the process as claimed (see A1, point 10; A2, points 7 and 8).

2.3.4 Under these circumstances the Board considers that, with respect to Examples 1 to 14 of D3, the problem underlying the invention is to be seen in the provision of a further process for preparing titania pigment of comparable quality.

2.4 The Board notes that the process of claim 1 and that of D3 have the following features in common:

- silica being precipitated at a pH above 9.8;
- the precipitated silica being cured first and last at a pH level of above 9.8 and between 9.2 and 9.4, respectively;
- alumina being precipitated at a pH between 5.5 and 6.5.

(see point 2.1 and present claim 1 versus D3, page 5, paragraph 1; page 9, lines 1 to 5 and Table 1, Examples No. 1, 5, 7, and 9).

The solution as proposed in claim 1 is distinguished from the closest prior art only in that:

- (a) the pH of the heated slurry is adjusted from above 9.8 directly to a value of from 9.2 to 9.4, without the intermediate curing at pH between 9.6 and 9.8 as in D3; and
- (b) the addition of a water-soluble aluminium compound commences at a pH value between 2.8 and 3.2 and not after the pH adjustment to a value between 5.5 and 6.5.

2.5 The Board is satisfied that the process according to claim 1 actually results in a titania pigment of a quality comparable with that obtained according to D3. This has never been contested. It remains to be decided whether the proposed solution to the problem of long processing times is obvious to a person skilled in the art in view of the available prior art documents.

2.6 Re distinguishing feature (a)

2.6.1 It is well known in the art that the precipitation of silica is to form a dense protective barrier around the TiO_2 particle (see D3, page 1, paragraph 2; D1', page 1, lines 52 to 59). The skilled person is further aware that such silica coating can be obtained not only in incremental curing steps as recommended in D3 (see point 2.4) but also in a single cure step as in D1' (see Example 1) or also as in Examples 15 to 17 of D3.

2.6.2 In addition to the specific example, the general teaching of D1' is that the silica coating should be cured at a pH between 9 and 10.5 and a temperature

between 80 and 100°C for 15 to 60 minutes. In principle, the lower the temperature, the higher a pH is required to achieve an impervious silica coating (see D1', page 2, lines 89 to 96). It is further taught that the exact duration of the cure step depends on the pH and temperature selected (see D1', page 2, lines 103 to 105). It is thus known that the duration of the cure step, the slurry pH and temperature are tightly interrelated with respect to the efficiency of the silica cure. The skilled person thus immediately knows that, if the slurry pH is varied within the prescribed range (between 9 and 10.5), the other two parameters (in this case, the temperature and retention time) are to be adjusted accordingly.

- 2.6.3 The Board holds that, against this background, the skilled person is fully aware that the curing method used in Examples 1 to 14 of D3 is open to modification, and in particular, that the number of curing steps is not critical. He would thus envisage modifying the process of D3 by skipping the intermediate silica curing step (at pH between 9.6 and 9.8) in order to arrive at another process for obtaining a product of about the same quality. Further adjustments of temperature and retention time that may be entailed by this modification are merely a matter of trial and error, within the limits disclosed in D1'. In this respect, it is noted that the ranges of temperature (75° to 95°C) and total retention time (from 10 to 80 minutes for both cure steps) as stipulated in claim 1 largely overlap the teaching of the prior art (D3: 85° to 95°C and a total of 15 to 95 minutes for all three cure steps; D1: 80° to 100°C and 15 to 60 minutes in a single step). The appellant, in fact, has not argued that these ranges are significant for

solving the present technical problem.

2.6.4 The appellant has advanced the argument that there is a clear technical prejudice in favour of maintaining slow neutralisation and slow silica deposition (Grounds of Appeal, page 9, paragraph 2). However, the processing times for neutralisation and silica deposition stipulated in claim 1 are fully within the framework disclosed and exemplified in the prior art (see points 2.3.1 and 2.6.2). Thus, the Board cannot see in which way the present modification should be considered to go against a technical prejudice.

2.6.5 The appellant has also drawn the Board's attention to the difference between a silica coating precipitated slowly at high pH and temperature and one which is precipitated quickly at neutral to slightly acid pH (Grounds of Appeal, page 9, last two paragraphs and Figure at page 10). However, this information is irrelevant for the assessment of inventive step in the present case since neither of the silica coatings under discussion is precipitated at neutral to slightly acid pH.

2.7 Re distinguishing feature (b)

2.7.1 The Board remarks that the wording in D3 is such that sodium hydroxide is only mentioned as an example of a base which can be used to raise the pH of the slurry from about 3 to between 5 and 6.5 (see page 8, last paragraph to page 9, line 4). Thus, the use of another base such as aluminate for raising the pH is also encompassed by this disclosure. Since aluminate is also used in D3 for the subsequent alumina precipitation, the Board holds that this compound - which is also a

base - is an obvious substitute for sodium hydroxide for raising the pH at this stage. The Board further notes that there is neither argument nor evidence on file that the use of aluminate instead of sodium hydroxide is significant in any aspect.

- 2.8 The appellant has not argued, let alone proved, that the distinguishing features (a) and (b) interact with each other or with the remaining technical features stipulated in the claim. The Board therefore finds that, in the light of the general disclosure in D3 and D1', the process as claimed is the predictable result of routine modification of the processes according to Examples 1 to 14 of D3. The subject-matter of claim 1 thus does not involve an inventive step within the meaning of Article 56 EPC.

Auxiliary request I

3. Claim 1 of this request differs from claim 1 of the main request only in that it is now directed to a process "consisting" of the steps as expressly stipulated.

However, the above reasoning for claim 1 of the main request is made without considering the possibility of additional steps (being comprised in the process). The finding in point 2.8 therefore applies mutatis mutandis to claim 1 of the present request.

Auxiliary request II

4. Claim 1 of this request differs from claim 1 of the main request only in that it clearly stipulates that the silica deposition and cure step is a two-stage step

and that the remaining steps follow in sequence. However, this interpretation is already the basis for the above reasoning concerning claim 1 of the main request.

The finding in point 2.8 therefore also applies to claim 1 of the present request.

Auxiliary request III

5. Claim 1 of this request differs from claim 1 of the auxiliary request II in that it specifies that the maintenance of the pH at from 5.5 to 6.5 is achieved by simultaneous addition of an acid. This is, however, also the case in D3 (see page 9, paragraph 1)

The present amendment therefore does not introduce any new aspect that could change the Board's finding concerning the previous requests.

Auxiliary request IV

6. Claim 1 of this request incorporates all amendments made in earlier requests.

The appellant has not submitted that the separate amended features interact in any unexpected way. The amendment to claim 1 thus does not involve any new aspect which has not been discussed. The finding in points 2.8, 3, 4 and 5 therefore also applies to claim 1 of the present request.

Request for reimbursement of the appeal fee

7. The Board has taken into account all the arguments and

submissions of the appellant. For the reasons expounded above, the Board has come to the conclusion that the appeal cannot be allowed. Therefore, the prerequisite for ordering the reimbursement of the appeal fee according to Rule 67 EPC is not met.

Order

For these reasons it is decided that:

1. The appeal is dismissed.
2. The request for refunding the appeal fee is rejected.

The Registrar:

The Chairman:

S. Hue

R. Spangenberg