BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

Internal distribution code:
(A) [ ] Publication in OJ
(B) [ ] To Chairmen and Members
(C) [X] To Chairmen

D E C I S I O N
of 15 September 1998

Case Number: T 0821/93 - 3.2.2

Application Number: 86302336.2

Publication Number: 0207586

IPC: C23C 18/22

Language of the proceedings: EN

Title of invention:
Sodium permanganate etch baths and their use in cleaning, desmearing and/or etching resinous substrates

Patentee:
Macdermid Incorporated

Opponent:
Schering AG

Headword:
-

Relevant legal provisions:
EPC Art. 52, 54, 56, 84, 123(2), (3)

Keyword:
"Novelty and Inventive step (yes)"

Decisions cited:
-

Catchword:
-

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0821/93 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 15 September 1998

Appellant: (Opponent)
Schering AG
13342 Berlin (DE)

Representative:
Effert, Bressel und Kollegen
Radickestrasse 48
12489 Berlin (DE)

Respondent: (Proprietor of the patent)
Macdermid Incorporated
245 Freight Street
Waterbury
Connecticut 06702 (US)

Representative:
Bankes, Stephen Charles Digby
Baron & Warren
18 South End
Kensington
London W8 5BU (GB)

Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office posted 14 July 1993 concerning maintenance of European patent No. 0 207 586 in amended form.**

Composition of the Board:

Chairman: W. D. Weiß
Members: R. Ries
J. C. M. De Preter

Summary of Facts and Submissions

I. The Appellant (Opponent) lodged an appeal against the interlocutory decision of the Opposition Division of 14 July 1993, by which the European Patent No. 0 207 586 (European patent application No. 86 302 336.2) was maintained in amended form.

II. The opposition was based on Article 100(a) and 100(b) EPC, and was supported by the documents including:

D1: US-A-4 425 380

D2: DE-C-2 550 598

D3: DE-C-3 110 415.

III. The Opposition Division held that the aqueous alkaline liquid permanganate solution claimed according to the first subsidiary request submitted by the Respondent (Patentee) with its letter dated 4 April 1993, was novel and involved an inventive step. In particular with respect to inventive step, the Opposition Division held that the cited prior art taught away from etching resinous substrates with alkaline solutions saturated by K-, Rb- or Cs- permanganate while having excess of MnO_4^- in solution in order to improve the etch rates and to simplify the removal of manganese deposits from the substrate.

Claim 1 according to the first subsidiary request reads as follows:

1. An aqueous, alkaline liquid permanganate solution containing sodium permanganate and sodium

hydroxide, which is made by adding to water 50 to 400 g/l of sodium permanganate, sodium hydroxide in an amount sufficient to provide 1 to 4 mole/l of OH^- and 0.1 to 3.0 moles per mole of permanganate in solution of a co-ion for MnO_4^- selected from K^+, Cs^+, Rb^+ and mixtures thereof, the solution being artificially maintained at the point of saturation of potassium, cesium or rubidium permanganate while having excess MnO_4^- in solution.

IV. Enclosed with its notice of appeal, the Appellant submitted four further documents

D4: EP-B-0 137 981

D5: GB-A-2 083 081

D6: US-A-3 957 059

D7: US-A-3 625 351

V. Oral proceedings were held before the Board of Appeal on 15 September 1998.

VI. The Appellant argued in its written submissions that, in the present case, it was not adequate to formulate claim 1 as a "product-by-process" claim, having in mind the Guidelines for Substantive Examination, CIII-4.7b. The claimed permanganate solution could be unambiguously defined by its components and the elemental ranges thereof. The amended claim 1 was not clear with respect to the technical feature to "maintain the solution artificially at the point of saturation of K-, Cs- or Rb-permanganate" as to whether the solution was saturated either at room temperature

or at the working temperature (about 77°C), or if the saturation status was always given. In the oral proceedings, the Appellant argued that a homogeneous solution in general did not include a precipitate (of the co-ion compound, e.g. $KMnO_4$) which in the claimed solution appeared to be indispensable to guarantee the saturation status. However, claim 1 of the disputed patent did not mention the presence of such a "precipitate". The amended claim 1, therefore, did not comprise all the essential features necessary to define the claimed solution and hence failed to meet the requirements of Article 84 EPC.

Claim 1 was objectionable under Article 123(2) EPC because there was no basis in the original disclosure for the minimum content of 50 g/l $NaMnO_4$.

Moreover, the subject matter of the patent as defined in amended claim 1 did not involve an inventive step in the light of the technical disclosure of document D1 or D4 in combination with the general knowledge of a chemist. Given that the etch rates of permanganate solutions were known to be strongly dependent upon the MnO_4^- concentration in the solution, it was obvious for a skilled person to increase the concentration of MnO_4^--ions in the solution, possibly up to the maximum concentration i.e. the saturation point, if the etching efficiency of the solution should be improved. To this end, permanganate ions could be supplied to the solution in the form of $KMnO_4$ or $NaMnO_4$, both components being equivalent and preferred because of ready availability at reasonable cost and good solubility. $KMnO_4$ or $NaMnO_4$ were found to be favourably present in a concentration varying from 10 to 100 g/l (cf. D4, page 4, lines 9 to 12). As regards the second problem to be solved by the claimed permanganate solution, i.e. the removability of manganese deposits formed during etching, test results presented in annex 1 to the letter dated 8 November 1993, showed no significant distinction between saturated and non-saturated permanganate solutions. The requirement of a "permanganate solution saturated with a co-ion" merely represented a technical feature which was arbitrarily chosen without exhibiting a reproducible effect which could justify an inventive step.

VII. The Respondent argued as follows:

The subject matter of the amended claim 1 was clearly defined and supported by the application as originally filed. The product-by-process terminology was admissible since the composition of the solution was

complicated due to the possible presence of numerous different ionic species and degrees of ionisation and oxidation states varying with temperature. With respect to the objection under Article 123(2) EPC, reference was made to examples 5 to 7 in Table 2 where the minimum content of 50 g/l of sodium permanganate was disclosed. Furthermore, the claimed subject matter of amended claim 1 was novel and involved an inventive step, since nobody had come up with the idea of enhancing the etch rates of sodium permanganate by adding co-ions e.g. in the form of potassium permanganate up to the saturation point.

VIII. The Appellant requested that the decision under appeal be set aside and the patent be revoked.

The Respondent requested that the appeal be dismissed and the patent be maintained according to the documents on which the decision under appeal was based, provided that Figure 2 be replaced by Figure 2 presented at the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments*

2.1 As is apparent from the application as filed, the opposed patent is concerned with "**aqueous**" alkaline solutions comprising water, alkali metal hydroxide, sodium permanganate and a co-ion (cf. the patent application as filed, claim 1, page 10, second paragraph). Consequently, the claimed solution is

prepared by combining **in** water the components enumerated in claim 1. Contrary to the Appellant's view, the definition "adding to water X g/l of component A" in claim 1 does not mean "X grams of A per litre of water". This interpretation of claim 1 is corroborated by the statement on page 14 of the description as filed according to which the term "grams/litre" refers to the amount of the particular component which is in solution at room temperature. As regards the minimum content of 50 g/l $NaMnO_4$, a clear general basis for this figure is given in examples 5 to 7 of Table 2, where 50 g/l $NaMnO_4$ is combined with three different amounts of $KMnO_4$. The Board is, therefore, satisfied that amended claim 1 complies with the requirements of Article 123(2) and (3) EPC.

2.2 Turning to the "product-by-process claim 1" objected to by the Appellant, it is known to a chemist that permanganate stability varies with the concentration of hydroxide ions in the solution (see for instance D4, page 4, line 3). Numerous different ionic species with varying oxidation levels of manganese may be present in the claimed solution. The claimed solution is, therefore, not unambiguously defined by its composition. On the other hand, the present wording of claim 1 gives the clear instruction to maintain the solution (always) at the saturation point of the co-ions. Given this situation, the product-by-process terminology of claim 1 is appropriate in the present case and in alignment with the practice of the EPO as reflected in the Guidelines CIII-4.7b, according to which product-by-process claims are admissible if they fulfil the requirements for patentability.

2.3 Claim 1 is directed to a solution artificially

maintained at the saturation point of $KMnO_4$, $CsMnO_4$ or $RbMnO_4$ without specifying a particular temperature. This implies that at any temperature, e.g. the processing temperature (of about 77°C) or at room temperature, the solution has to be saturated. According to the description of the application as filed, page 12, paragraph 1, the saturated state is accomplished by adding K^+, Rb^+ or Cs^+ ion or a mixture thereof to an aqueous solution of $NaMnO_4$ within specific amounts. The co-ion can be added e.g. in the form of a liquid up to the saturation point or, alternatively, a co-ion precipitate is present to act as a "reservoir", a situation well known to the chemist as saturation.

Consequently, all the amendments made to claim 1 as granted fulfil the requirements of Article 84 EPC.

3. *Cited prior art*

Enclosed with its statement of grounds, the Appellant submitted four new documents D4 to D7 which are late filed in the meaning of Article 114(2) EPC. Given that documents D5 and D6 relate to solutions exhibiting a composition different from that claimed and which are not even remotely concerned with the technical field to which the opposed patent belongs, these documents are disregarded.

4. *Novelty*

None of the cited documents specifically discloses an aqueous alkaline hydroxide - sodium permanganate solution artificially maintained at the saturation point of $KMnO_4$, $CsMnO_4$ or $RbMnO_4$. This technical feature was neither explicitly nor implicitly disclosed in any

of the examples of the cited prior art. This fact was no longer disputed at the oral proceedings.

5. *Inventive step*

5.1 Closest prior art:

Among the cited prior art, document D1 represents the closest state of the art. Like the opposed patent, this document is concerned with an improved process for removing resin smeared on an interior wall of a hole in a resinous substrate and, thereafter, neutralizing essentially all the manganese residues deposited on the substrate. Document D1, in column 7, lines 48 to 52, clearly expresses that the smear removal rate increases with increasing permanganate concentration which effect, however, is associated with an increase of residual manganese left on the surface of the substrate. Good results are obtained with $KMnO_4$ or $NaMnO_4$ in the range of 10 to 60 g/l solution. On the other hand, large amounts of residual manganese were found when alkaline permanganate treating solutions having a pH above 13 were used. Based on these findings, document D1 advocates using alkaline permanganate treating solutions having a pH between 11 and 13 which leave less manganese in the holes (cf. D1, column 4, lines 20 to 43).

5.2 Problem and Solution

Starting from this prior art, the technical problem underlying the opposed patent consists in the provision of an aqueous alkaline permanganate treating solution which exhibits smear removal rates (etching) at least equal or better than those of pure $NaMnO_4$ solutions or

of solutions conventionally used without leaving excessive residual manganese deposits which are difficult to remove.

The patent in suit solves this problem according to claim 1 by an aqueous highly alkaline sodium permanganate solution which is artificially maintained at the saturation point of $KMnO_4$, $CsMnO_4$ or $RbMnO_4$.

Having regard to the examples in the patent, the technical problem defined above has been successfully solved. This is evident, for instance, by comparing the etch rates Δwt of examples 19, 20 and 11 to 13. As can be seen, the etch rate of a "pure" 200 g/l $NaMnO_4$ - 3 molar NaOH solution (example 19) is lower than that of the same solution additionally comprising 0.5 moles/l of co-ion K^+ (example 20) or 0.1 moles/l of K^+ (example 11). Even with an addition of 0.2 or 0.3 moles/l of K^+ (examples 12 and 13), the etch rates are still approximately equivalent to that of the "pure" solution. It is noted that $NaMnO_4$ solutions comprising 1.9 moles/l NaOH (75 g/mole) with lower concentrations of MnO_4^- ions (examples 1 to 4, 6 to 10 of Table 2) exhibit etch rates much higher than that of the "pure" $NaMnO_4$ solution given in example 19.

5.3 Also the combined teaching of documents D1 and D2 does not lead a person skilled in the art to the claimed aqueous permanganate solution. Like D1, document D2 reports an increase of the etch rates with increasing permanganate concentrations (cf. D2, column 4, lines 53 to 56), but nevertheless, potassium permanganate solutions comprising 10 to 75 g/l $KMnO_4$ and having a pH value between 13.4 and 13.5 are preferred (cf. D2, column 3, lines 26 to 31, claim 2). The Appellant pointed to example 8 of D2, comprising 60 g/l $KMnO_4$ and

38 g/l NaOH corresponding to a pH value of 13.40 to 13.45. However, this solution is different from that claimed in that it merely comprises 0.96 moles/l NaOH which quantity is outside the range of 1 to 4 moles/l OH^- defined in claim 1. In addition thereto, the total concentration of Na^+ ions of 0.96 moles/l is below the minimum limit of 1.35 moles/l of Na^+ required in the solution according to claim 1.

The same applies to document D3 which discloses a cleaning solution consisting of 50 g/l $KMnO_4$ (= 0.32 moles/l) and 50 g/l NaOH (1.25 moles/l) (cf. D3, claim 2; column 4, Example). Since in claim 1 of the patent in suit a total Na^+ concentration of 1+0.35=1.35 moles/l and a MnO_4^- concentration of 0.35 moles/l is required, this solution is also outside the concentration ranges defined in claim 1 and would not induce a skilled person to shift the concentration to the ranges claimed.

The Appellant further referred to document D4 which, in common with the opposed patent, deals with a pretreatment of through-hole and multi-layer printed circuit boards using epoxy printed circuit board base material. The solvent pretreatment uses an oxygenated water soluble solvent which is a permanganate solution activated by high pH (cf. D4, page 3, lines 16 to 18). According to document D4, the pH should exceed 13 and should range between 13.1 and 13.6 (cf. page 3, lines 50 to 58; claim 3). It is, however, emphasized on page 4, second paragraph of D4 that the preferred operating pH (=13.1 to 13.6) entails the problem of decreased permanganate stability. Nevertheless, the concomitant increase in peel strength (etching capacity) and the consistency of the results obtained

are held to offset the disadvantage of reduced permanganate stability. Both $NaMnO_4$ and $KMnO_4$ are preferred in the permanganate solution in a concentration ranging from 10 to 100 g/l, preferably between 20 and 50 g/l. In example 1, page 5, of document D4, an oxidising solution comprising 40 g/l $KMnO_4$-30 g/l NaOH was used. The evaluation of the contents of document D4 would lead a skilled person to note the reduced stability of permanganate at high pH and, consequently, this document points away from further increasing the pH closer to 14 as is the case in the disputed patent, i.e. to a NaOH concentration between 1 to 4 moles/l.

Document D7 is more remote from the subject matter of the patent in suit. It discloses an etching pretreatment for electroplatable grades of ABS resins, using an etching composition of 40 to 70 g/l of alkali metal manganate(VI), 400 to 600 g/l of alkali metal hydroxide and 230 to 560 g/l water. Put the other way, this solution comprises 10 moles/l or more OH^- in the case of NaOH or at least 8.7 moles/l OH^- in the case KOH, both concentrations being far outside the composition claimed.

5.4 None of the cited documents leads the skilled person to provide a $NaMnO_4$ solution saturated with a co-ion and exhibiting a NaOH concentration in the range of 1 to 4 moles/l in order to improve the etching ability. On the contrary, in most cases $KMnO_4$-NaOH solutions of pH 13 to 13.6 are preferred for the reasons previously given.

5.5 The Appellant maintained that it was obvious to increase the MnO_4^- concentration in the solution to

enhance its etching efficiency and showed this relationship in the graph (Diagram I) submitted on 22 July 1998. In view of the comparative tests submitted with its grounds of appeal, the feature of maintaining the solution at the saturation point was held to be irrelevant, in particular with respect to the manganese deposits formed on the substrate.

5.6 It is well known that the etching rates depend on the concentration of permanganate in the solution. The patent in suit, however, shows that the etching rate of a pure 200 g/l $NaMnO_4$ solution (example 19) is significantly lower than that of examples 2 or 6, which exhibit much lower concentrations of MnO_4^- ions. Besides, by adding co-ions to "pure" 200 g/l $NaMnO_4$ solution, etching rates first increase or remain almost stable rather than decrease as might have been expected due to the reduced amount of MnO_4^- ions present in the solutions. Although the interactions between the concentrations of $NaMnO_4$, NaOH and the saturation status of the solution with K^+, Rb^+ or Cs^+ acting as a permanent "reservoir" for MnO_4^- ions are not theoretically explained, a pronounced benefit in terms of improvement to the etching efficiency of the claimed permanganate solution is undoubtedly achieved.

5.7 In view of all these considerations, the aqueous highly alkaline permanganate solution defined in claim 1 of the disputed patent involves an inventive step in the meaning of Article 56 EPC.

5.8 Claims 2 to 5 relating to preferred embodiments of the solution claimed in claim 1 and claims 7 to 9 directed to a process for cleaning, etching and desmearing resin from a substrate with the claimed solution are also

allowable.

Order

For these reasons it is decided that:

1. The decision is set aside.

2. The case is remitted to the first instance with the order to maintain the patent in amended form as defined in the decision under appeal, provided that Figure 2 be replaced by Figure 2 as presented at the oral proceedings.

The Registrar: The Chairman:

N. Maslin W. D. Weiß