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Datasheet for the decision of 8 January 2013

Case Number: T 1009/12 - 3.2.07

Application Number: 03078107.4

Publication Number: 1391536

IPC: C23C 18/42, H05K 3/24

Language of the proceedings: EN

Title of invention:
Composition and process for plating silver onto a metallic substrate

Applicant:
MacDermid, Incorporated

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (all requests - no: claims include ineffective concentrations of a component so that this feature not contributing to the solution of a problem cannot be considerd for defining a problem vis-à-vis the closest prior art)"

Decisions cited:
T 0197/86, T 0035/85, T 0190/99, T 0234/03, T 0378/03, T 0206/91

Catchword:
An ineffective concentration of a compound is considered as an arbitrary feature not contributing to the solution of the underlying problem and therefore not further considered (see point 2.7 of the reasons)"

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 1009/12 - 3.2.07

D E C I S I O N
of the Technical Board of Appeal 3.2.07
of 8 January 2013

Appellant: (Applicant)
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 17 November 2011 refusing European patent application No. 03078107.4 pursuant to Article 97(2) EPC.**

Composition of the Board:

Chairman: H. Meinders
Members: H. Hahn
E. Kossonakou

Summary of Facts and Submissions

I. The applicant lodged an appeal against the decision of the Examining Division to refuse the European patent application No. 03 078 107.4 which is a divisional application originating from the earlier European patent application No. 97 301 116.6 (herein designated "parent application").

II. In the present decision the following document from the examining procedure is cited:

D1 = JP-A-03 002379 (Japanese original and English patent abstract)

and the following document was submitted by the appellant in the appeal proceedings:

D2 = Declaration of Ms Toscano (undated)

III. The Examining Division held that the omission of the essential component "imidazole" from the subject-matter of claims 1 (plating solution) and 5 (process for using that solution) and from page 4 of the description of the divisional application according to the then main request contravenes Article 76(1) EPC. Similarly, claims 1 and 5 of the then first auxiliary request were considered to contravene Article 123(2) EPC for not specifying the specific formula of the imidazole while the omission on page 4 of the description was considered to contravene Article 76(1) EPC. The Examining Division further considered that the same holds true with respect to claim 1 and page 4 of the description of the then second auxiliary request, those

being identical with claim 5 and page 4 of the description of the first auxiliary request. Claim 1 of the then third auxiliary request was considered to comply with Articles 76(1) and 123(2) EPC but page 4 of the description to still contravene Article 76(1) EPC. Furthermore, the subject-matter of claim 1 of that third auxiliary request was considered to lack inventive step over D1 since no beneficial effect of the distinguishing feature, i.e. the oxidant (nitro aromatic compound), had been demonstrated.

IV. With a communication dated 14 September 2012 and annexed to summons for oral proceedings the Board presented its preliminary and non-binding opinion with respect to claims 1-9 of the main request and claims 1-7 of the first auxiliary request, both as filed together with the grounds of appeal dated 27 March 2012 and corresponding in substance to the earlier first and second auxiliary requests.

Claims 1 and 5 and page 4 of the divisional application according to the main request appeared not to be formally allowable under Article 76(1) EPC.

The Board further stated amongst others that claim 1 of the first auxiliary request, which does not specify any concentration ranges of the four components at all, is considered to cover embodiments where the presence of the compounds "imidazole" and "oxidant" will not produce any effect at all. Furthermore, the declaration D2 filed with the grounds of appeal does not prove that a technical effect may be achieved "across the breadth of the claim" as alleged by the appellant since D2 discloses only a single example made with a rather high

concentration of the imidazole (compared with the only example in the patent in suit) and a concentration of the nitro aromatic compound within the preferred range mentioned in the patent in suit.

Furthermore, the comparative test according to D2 appeared to have **neither** been made in accordance with the closest prior art D1 **nor** in agreement with the examples of the present divisional application.

In order to demonstrate an alleged surprising effect according to the established jurisprudence (see Case Law of the Boards of Appeal of the European Patent Office, 6th edition 2010, section I.D.9.9) the comparison should have been made on the basis of at least one of the examples of D1. Hence it appeared that a surprising effect vis-à-vis D1 could not be acknowledged.

V. With its letter dated 7 December 2012 the appellant maintained its submissions concerning added subject-matter of the main request and submitted only further arguments with respect to inventive step of claim 1 of the first auxiliary request taking account of the remarks made by the Board in its above mentioned communication.

VI. The independent claims 1 and 5 of the main request read as follows:

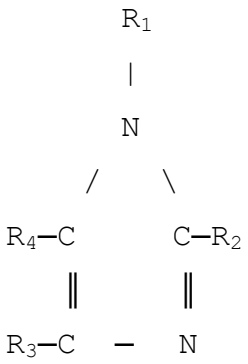
"1. A composition useful in immersion plating silver onto a metallic substrate, which composition comprises:
a) a soluble source of silver ions;
b) an acid;

c) an imidazole; and characterised by
d) an oxidant selected from nitro aromatic compounds."

"5. A process for plating silver onto a surface, wherein the silver is plated from the composition of any one of claims 1 to 4."

VII. Independent claim 1 of the first auxiliary request reads as follows:

"1. A process for plating silver onto a surface, wherein the silver is immersion plated from a composition comprises:
a) a soluble source of silver ions;
b) an acid;
c) an imidazole of the following formula:



wherein R_1, R_2, R_3 and R_4 are independently selected from the group consisting of substituted or unsubstituted alkyl groups, substituted or unsubstituted aryl groups, halogen, nitro groups and hydrogen; and
d) an oxidant selected from nitro aromatic compounds."

VIII. Oral proceedings before the Board were held on 8 January 2013. The issue relating to added subject-matter of the main request was discussed first.

Thereafter the issue of inventive step was discussed with respect to the independent claims of the main and first auxiliary request, particularly in view of document D2 and whether or not its experimental results represent a comparative test with the closest prior art D1 as required by the longstanding jurisprudence of the Boards of Appeal in order to prove the alleged effect and whether this effect is proven over the whole scope of the independent claims.

The appellant requested that the decision under appeal be set aside and that a patent be granted in accordance with either the main or the first auxiliary request as submitted together with its statement setting out the grounds of appeal dated 27 March 2012.

At the end of the oral proceedings the Board announced its decision.

IX. The appellant argued, insofar as relevant for the present decision, essentially as follows:

The declaration D2 of Ms Toscano proves the achievement of an unexpected effect which is plausibly achieved over the scope of the process of claim 1 of the first auxiliary request when using the described composition including the imidazole and the nitro aromatic compound in an effective amount, i.e. when sensibly construed by a skilled person in accordance with the EPC (compare T 190/99, not published in OJ EPO).

Although the declaration is not dated it was received from the applicant in January 2012 and it is believed that it was contemporaneously made and proves the

existence of a technical effect over the state of the art, in particular the closest prior art D1.

According to the experiments A, B and C carried out and described in D2 only one variable was changed to show the influence of the compound in question, i.e. the nitro aromatic oxidant in the form of dinitrosalicylic acid, on the immersion silver plating solutions (see paragraph 3 of D2). Ms Toscano concludes that imidazole is useful to increase the plating rate and to brighten and improve the quality, whereas organic nitro compounds, especially the dinitro compounds, are useful to decrease the plating rate and to brighten and improve the silver deposit. Furthermore, in optimizing an immersion silver plating bath, the presence and concentration of these additives can be manipulated in various ways to achieve good results (see paragraph 4 of D2).

Ms Toscano has optimised the composition of the broadest objective disclosure in D1, i.e. to use an imidazole, in these experiments which are within the claim scope of D1 as well as of the present application. In its communication the Board mentioned several parameters such as silver nitrate concentration, imidazole concentration, etc. which from the Board's point of view may influence the effect but for these postulations it has no factual basis.

According to the decision T 197/86 (OJ EPO 1989, 371) where comparative tests are submitted as evidence of an unexpected effect, there has to be a closest structural approximation in a comparable type of use to the subject-matter claimed (see Case Law of the Boards of

Appeal of the European Patent Office, 6th edition 2010, second paragraph of section I.D.9.9). This requirement is met since the experiments according to D2 are within the claim scope of the present divisional application as well as within the scope of D1. Given the breadth of the disclosure of D1, which is much broader than the specific examples thereof, and the scope of claim 1 it was entirely correct of Ms Toscano to perform the experiments as she did. D2 is a fair comparison and it is not technically credible that this is not so. It is, however, admitted that D1 does not disclose a bath including HEDTA and surfactants but the presence of these components only shifts the base line of the results, not the difference in effect attributed to the nitro aromatic compound.

Taking account of D2 the unexpected technical effect of adding this nitro aromatic compound is to brighten and improve the silver deposit which was not predictable from the disclosures of D1.

D2 admits in the general conclusions of paragraph 4 that the concentrations may be manipulated. This does, however, not mean that a certain minimum threshold of concentrations is necessary. It is plausible that the skilled person would obtain the desired result over the scope of the independent claims when considering a fair construction thereof.

Even if the Board would not accept D2 as comparative example or if the effect over the scope of the claims is not considered proven then a less ambitious problem is to be defined which is the provision of an immersion silver plating bath and/or process which is effective.

The addition of the nitro aromatic compound is not arbitrary since the addition provides a bright coating. It is also not suggested by D1, which nowhere suggests a nitro aromatic compound as an additive. Furthermore, it is not possible to argue that the skilled person would randomly add any solid compound, as argued by the Examining Division. Adding a component increases costs and complexity of a process. An arbitrary addition of the nitro aromatic compound in an ineffective amount as argued by the Board in its communication is not consistent with the could-would test.

Further evidence for proving the effect is not at hand.

It is not known which component(s) of the immersion silver plating bath shall be oxidised by the oxidant (nitro aromatic compound).

Therefore the subject-matter of the independent claims of both requests involves inventive step.

Reasons for the Decision

1. *Allowability of amendments made in both requests (Articles 76(1) and 123(2) EPC)*

Since the Board considers that process claim 1 of the first auxiliary request lacks inventive step (see points 2.1 to 2.7 below), which conclusion equally applies to the subject-matter of the broader independent claims 1 and 5 of the main request (see point 2.8 below) there is no need to consider in this decision whether or not the amendments made in the

divisional application according to the main and the first auxiliary requests comply with Articles 76(1) and/or 123(2) EPC.

2. *Inventive step (Article 56 EPC)*

The discussion of inventive step is more efficient if the Board first turns to the most limited process claim 1 of the first auxiliary request which involves using an immersion silver composition *per se* being restricted to the specific imidazoles (see point VII above), since the said composition is encompassed by the composition of claim 1 of the main request and the process concerned is a specific form of the process of independent claim 5 of the main request (see point VI above).

First auxiliary request

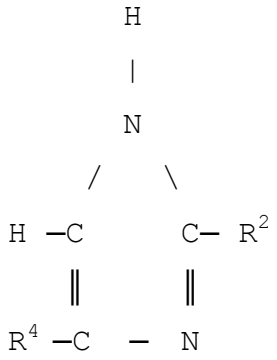
2.1 The Board comes to the conclusion that process claim 1 of the most restricted first auxiliary request lacks inventive step over the teaching of the uncontested closest prior art D1 for the reasons that follow.

2.2 The English abstract of the Japanese application D1 discloses a process for forming a silver plating on the surface of copper or copper alloy by contacting the surface with an aqueous immersion plating solution. This solution according to D1 has a pH of 3.0 to 4.5 and contains 0.01 to 1.0 %, more preferably 0.05 to 0.5 % silver nitrate (this percentage "%" is interpreted as meaning "wt.%" so that these ranges correspond to 0.1-10 g/l and preferably 0.5-5.0 g/l silver nitrate; thus corresponding to 0.06-6.3 g/l or

preferably 0.32-3.2 g/l Ag^+-ions), 2-alkylimidazole (see below) as an alkyl imidazole compound in combination with an organic acid, e.g. acetic acid (preferably in the form of the salt). The treating liquid is usually kept at 20-60°C and is brought into contact with the copper or copper alloy for 30 seconds to 5 minutes to form the silver film (see English abstract of D1).

2.2.1 The aqueous immersion plating silver solution of D1 is thus a simple aqueous composition containing silver nitrate, an organic acid (or salt thereof) and e.g. a 2-alkylimidazole.

Taking account of the structural formula of the imidazole given in D1, which comprises the two substituents R^2 and R^4, and the imidazole description in the English abstract it is clear that the imidazole can be substituted by alkyl groups in the imidazole positions 2 and 4, i.e. R^4 or R^2 can be hydrogen:



2.2.2 The composition described in the English abstract of D1 does **not** contain any surfactant or chelating agent.

2.3 The process for plating silver onto a surface according to claim 1 of the first auxiliary request - wherein the silver is immersion plated from a composition which

comprises a soluble source of silver ions, an acid, an imidazole of the given formula with either R_1, R_2 and R_4 being selected from hydrogen and R_3 being selected from substituted or unsubstituted alkyl groups, or R_1, R_3 and R_4 being selected from hydrogen and R_2 being selected from substituted or unsubstituted alkyl groups, or with R_1 and R_4 being selected from hydrogen and R_2 and R_3 being selected from substituted or unsubstituted alkyl groups (see points VII and 2.2.1 above) - is thus distinguished from the process according to D1 only by an oxidant being selected from nitro aromatic compounds.

2.3.1 The divisional application as originally filed is silent with respect to any effect of this distinguishing feature (see page 7, first paragraph), and in the parent application it is presented only as an optional feature of the composition (see page 5, first paragraph and paragraph bridging pages 6 and 7 and claims 17 and 20), also without any effect.

2.3.2 According to the examples I to III of the divisional application the silver plating solution **consisted of** the four components silver nitrate (1 g/l), **methane sulfonic acid** (20 ml (70%)/l), 3,5-dinitrohydroxy benzoic acid (1 g/l), **l-histidine** (1 g/l) and water and copper was plated with it at 100°F (= **37.8°C**) for **5 minutes** (see examples I-III).

This imidazole compound "L-histidine" (IUPAC name) used in the examples I-III can also be designated "2-Amino-3-(1*H*-imidazol-4-yl) propanoic acid", and thus represents an imidazole of the claimed specific formula wherein R_1, R_2 and R_4 are hydrogen and R_3 is a substituted alkyl group, i.e. $-CH_2-CH(NH_2)-COOH$.

2.3.3 On the other hand, process claim 1 defines a process for plating silver onto a surface which uses a composition which "**comprises**" the four specified components in unspecified amounts.

According to this open definition the presence of a very small amount of e.g. 5 ppm of said oxidant is sufficient to meet the condition of claim 1 of the first auxiliary request. However, the Board considers that such a low concentration will not cause any effect in view of the common general knowledge of a chemist and the intended purpose of an oxidant. Particularly in the light of the most general range of the oxidant of from 0.1-25 g/l (corresponding to 100-25000 ppm) disclosed in the present application (see page 7, first paragraph) it is not reasonable to expect any effect of the nitro aromatic compound in such a low concentration.

Therefore claim 1, which does not specify any concentration ranges of the four components at all, is considered to cover embodiments where the mere presence of the nitro aromatic oxidant compound will **not** produce any effect at all.

The appellant's arguments to the contrary cannot hold for the following reasons.

2.4 First of all, the appellant argued that the skilled person would comprehend the claims as encompassing only effective amounts of the specified components to achieve the technical effects including immersion silver plating as disclosed in the application; he would construe the claims by "building up" and not by

"tearing down", to arrive at an interpretation of the claims which is technically sensible and takes account of Article 69 EPC (T 190/99, *supra*, reasons 2.4).

However, taking account of Article 69 EPC cannot be accepted since that decision relates to an opposition case where an interpretation of the claim which is technically sensible takes into account the whole disclosure of the patent (Article 69 EPC) and therefore does **not** apply to the present **examination** case. In such a case, like the present one, the claims have to make sense as they stand. Since none of the independent claims of both requests defines "an effective amount of the oxidant" it is evident that the scope of these claims is **not** restricted to effective amounts but, to the contrary, encompasses ineffective amounts of said nitro aromatic oxidant.

In this context it is further considered that the immersion silver plating composition *per se* used according to process claim 1 - as described on page 6, second and third paragraphs of the **parent** application as originally filed (the oxidant was an optional component of the immersion silver plating composition of the parent application; see point 2.3.1 above) - even **without the nitro aromatic oxidant** provides brighter, smoother and more cohesive silver deposits than baths not containing imidazoles. The same conclusion would be valid for a bath according to process claim 1 which comprises an ineffective amount of e.g. 5 ppm of the nitro aromatic oxidant. Therefore also the appellant's argument that an effective amount of the oxidant is necessary to obtain a bright silver deposit cannot hold.

2.5 The appellant's arguments that the experiments of D2 would represent acceptable comparative examples with respect to D1 and would prove an effect of the nitro aromatic oxidant - i.e. to decrease the plating rate and to brighten and improve the silver deposit - cannot hold either.

2.5.1 Regarding the declaration D2 of Ms Toscano, the Board considers unusual that it is neither dated nor that it states that the described experiments have been made in agreement with the present application and/or in agreement with the closest state of the art D1. In substance, D2 discloses the examples A, B and C.

Example B repeats example A with the difference that no imidazole was present in the plating composition. Example C repeats example A with the difference that no dinitrosalicylic acid (i.e. the nitro aromatic oxidant) was present (see D2, paragraph 3).

The immersion silver plating bath of example A does not specify the **silver ion concentration** nor that it is **aqueous**. It comprises besides **10 g/l imidazole** (since not further specified it has to be interpreted that all substituents R_1, R_2, R_3 and R_4 are hydrogen), **11.3 g/l of hydroxyethylenediaminetetraacetic acid (HEDTA)**, **5 g/l Chemeen T-15**, **5 g/l Igepal Co-730**, 0.75 g/l of 3,5-dinitrosalicylic acid (which is the trivial name for the compound "3,5-dinitrohydroxy benzoic acid" disclosed in the present application as the preferred oxidant) and said bath has a **pH of 2** adjusted with nitric acid. The copper parts are plated in the bath for **6 minutes** at **35°C** (see D2, page 1, point 3A).

D2 does **not** contain any explanation of Ms Toscano why said chelating agent HEDTA and said two surfactants (Chemeen and Igepal) have been added to the immersion silver plating solution. They clearly do not figure in the plating solution of D1.

2.5.2 Although the definition "comprising" of claim 1 does not exclude such additional components the present application is absolutely silent with respect to an optional addition of such a chelating agent like HEDTA, or of surfactants such as said Chemeen T-15 or Igepal Co-730 (compare point 2.3.1 above), let alone that such components should be added in such considerable amounts.

From a comparison with the components of the composition, temperature and treatment time of example A and the composition and corresponding parameters of examples I-III of the present application (see point 2.3.2 above) it is evident that more than 7 parameters (additional components HEDTA and surfactants, different imidazole in a 10-times higher concentration, lower concentration of nitro aromatic oxidant, longer treatment time and somewhat higher temperature) are modified.

2.5.3 D1 is likewise silent with respect to the addition of the chelating agent HEDTA and surfactants (compare points 2.2.2 and 2.2.3 above) as admitted by the appellant at the oral proceedings. From a comparison between the components of the composition, temperature and treatment time of example A and the composition and corresponding parameters of the abstract of D1 it is evident that at least 3 parameters (additional

components HEDTA and surfactants, different imidazole and longer treatment time) are modified.

2.5.4 Taking account of these modifications, both in comparison with the parent application and the closest prior art D1, it is evident that the experiments A, B and C according to D2 do **not** represent comparative tests as required by the established jurisprudence to demonstrate an alleged effect.

According to this established jurisprudence, if comparative tests are chosen to demonstrate an inventive step on the basis of an improved (or surprising) effect, the nature of the comparison with the closest state of the art must be such that the alleged advantage or effect is convincingly shown to have its **origin in the distinguishing feature** of the invention compared with the closest state of the art (see Case Law of the Boards of Appeal of the European Patent Office, 6th edition 2010, section I.D.9.9; see T 197/86, OJ EPO 1989, 371 reasons point 6; T 234/03; T 378/03 the latter two not published in OJ EPO).

The appellant argued by reference to T 35/85, mentioned in the same section of the "Case Law of the Boards of Appeal", that it should benefit from the qualification that decision applied to this "established jurisprudence". Its variants lay close to the invention so that the advantageous effect attributable to the distinguishing feature was more clearly demonstrated. This cannot be accepted since the comparison remains executed with more than one parameter different from either the examples of the present application or the comparison of the abstract of D1. The decisions cited

in that part all have in common that it is only the distinguishing feature which is different in the "closest variant" as allowed by the cited decisions. Having, 7 respectively, 3 parameters different can hardly qualify as a "closer variant".

2.5.5 Since D2 cannot be accepted as a comparative test the alleged effect of the nitro aromatic oxidant vis-à-vis the closest prior art D1 has **not** been proven and all the appellant's arguments based thereon, including the problem to be solved, need not be considered.

2.6 Furthermore, even if these experiments A, B and C according to the declaration D2 would be considered, they do **not** prove that a technical effect may be achieved "across the breadth of the claim" as alleged by the appellant.

2.6.1 D2 discloses **a single example**, i.e. example A which composition comprises all four components specified in claim 1, made with a concentration of **0.75 g/l** (corresponding to **750 ppm**) of the preferred oxidant, i.e. 3,5-dinitrosalicylic acid (which is the trivial name for the compound "3,5-dinitrohydroxybenzoic acid" disclosed in the present application). This concentration is thus within the preferred range of from 0.5-2 g/l (corresponding to 500-2000 ppm) specified in the first paragraph at page 7 of present application as originally filed.

An example containing 750 ppm of the nitro aromatic oxidant represents, however, no proof at all that a non-*de minimis* concentration of 5 ppm thereof would be effective.

2.6.2 The declaration D2 of Ms Toscano is totally silent with respect to a minimum threshold concentration of the nitro aromatic oxidant.

The appellant's argument that Ms Toscano does not see a minimum threshold as necessary cannot hold since one of her conclusions based on the three experiments A, B and C as drawn in the fourth bullet point of paragraph 4, quoted by the appellant in support, merely and non-specifically states "In optimizing an immersion silver plating bath, the presence and concentration of these additives can be manipulated in various ways to achieve good results". This non-statement with respect to a minimum threshold concentration, however, does not allow to conclusively derive that such a minimum threshold does not exist, particularly when considering her conclusions in the first and second bullet points of paragraph 4 that neither imidazole nor organic nitro compounds are necessary to achieve an acceptable silver plate.

2.7 According to the established case law, features which do not contribute to the solution of the problem set in the description are **not** considered in assessing the inventive step of a combination of features (see Case Law of the Boards of Appeal of the European Patent Office, 6th edition 2010, section I.D.8.4, in particular T 206/91, not published in OJ EPO, point 5.5 of the reasons).

Since process claim 1 includes silver plating compositions including unspecified, therefore also ineffective concentrations of the nitro aromatic

oxidant (see points 2.3.3 and 2.4 above) this feature can only be considered as an arbitrary feature, because it is not credible that it contributes to the solution of the underlying technical problem. The Board therefore does not further consider it. Moreover, in a case like the present one it considers that for the same reason the formulation of a less ambitious problem, i.e. to provide an alternative solution with the same effect, based on this alleged distinguishing feature cannot hold either.

Since there are no further distinguishing features it is not feasible to identify the technical problem to be solved.

Therefore, since no technical problem can be identified which is solved by the subject-matter of process claim 1, the Board considers that claim 1 of the first auxiliary request lacks inventive step (Article 56 EPC). The first auxiliary request is therefore not allowable.

Main request

2.8 Since process claim 1 of the first auxiliary request is narrower in scope than process claim 5 of the main request and includes the use of the immersion silver plating composition comprising the more specific imidazole being included in the more generic composition of claim 1 of the main request (see points VI and VII above) the above conclusion with respect to claim 1 of the first auxiliary request applies *a fortiori* to claims 1 and 5 of the main request.

The Board therefore concludes that the subject-matter of claims 1 and 5 of the main request does not comply with the requirement of Article 56 EPC either. The main request is therefore not allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

G. Nachtigall H. Meinders