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# Datasheet for the decision of 26 April 2006

Case Number:	T 0004/03 - 3.3.01
Application Number:	98942556.6
Publication Number:	1000032
IPC:	C07D 211/94

Language of the proceedings: EN

# Title of invention:

Inhibition of pulp and paper yellowing using nitroxides and other coadditives

#### Applicant:

Ciba Specialty Chemicals Holding Inc.

## Opponent:

-

Headword: Inhibition of pulp and paper yellowing/CIBA

# Relevant legal provisions: EPC Art. 56 EPC R. 67

# Keyword: "Inventive step (no) - obvious solution of the technical problem" "Reimbursement of the appeal fee (no)"

# Decisions cited:

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Catchword:

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Beschwerdekammern

Boards of Appeal

Chambres de recours

**Case Number:** T 0004/03 - 3.3.01

#### DECISION of the Technical Board of Appeal 3.3.01 of 26 April 2006

Appellant:

Ciba Specialty Chemicals Holding Inc. Klybeckstrasse 141 CH-4057 Basel (CH)

Representative:

Decision under appeal: Decision of the Examining Division of the European Patent Office posted 6 August 2002 refusing European application No. 98942556.6 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman:	Α.	Nuss
Members:	J.	Jonk
	Ε.	Dufrasne

# Summary of Facts and Submissions

- I. This appeal lies from the decision of the Examining Division refusing the present European patent application 98 942 556.6 (published under number WO 99/05108), which relates to inhibition of pulp and paper yellowing using nitroxides and other co-additives.
- II. The application in suit was refused on the ground that the subject-matter of Claims 1 to 18 of the application as filed lacked inventive step in view of several documents including:

(1) EP-A-0 309 401.

#### III. Claim 1 of the application as filed read as follows:

"1. A composition having reduced loss of brightness and enhanced resistance to yellowing which comprises

- (a) a pulp or paper which still contains lignin, and
- (b) an effective stabilizing amount of a hindered amine compound of formula I or II





where

 $G_1$  and  $G_2$  are independently alkyl of 1 to 4 carbon atoms or are together pentamethylene,

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 $Z_1$  and  $Z_2$  are each methyl, or  $Z_1$  and  $Z_2$  together form a linking moiety which may additionally be substituted by an ester, ether, hydroxy, oxo, cyanohydrin, amide, amino, carboxy or urethane group,

E is oxyl, hydroxyl, hydrogen, alkyl, alkyl substituted by hydroxyl, oxo or carboxy or interrupted by oxygen or carboxy alkenyl, alkynyl, cycloalkyl, cycloalkenyl, bicycloalkyl, alkoxy, alkoxy substituted by hydroxyl, oxo or carboxy or interrupted by oxygen or carboxy, cycloalkoxy, alkenyloxy, cycloalkenyloxy, aralkyl, aralkoxy, acyl, R<sup>(C=0)O-,</sup> R<sup>O(C=0)O-,</sup> R<sup>(N(C=0)O-</sup> or chloro, where R<sup>(C=0)O-,</sup> R<sup>(O(C=0)O-,</sup> aromatic moiety,

X is an inorganic or organic anion, and

where the total charge of cations h is equal to the total charge of anions j, and with the proviso that the compound of formula I is not bis(2,2,6,6tetramethylpiperidin-4-yl)sebacate or the polycondensation product of 1-(2-hydroxyethyl)-2,2,6,6tetramethyl-4-hydroxypiperidine and succinic acid."

Claim 6 of the application as filed concerned compositions according to Claim 1 wherein the hindered amine compound of component (b) was selected from the compounds of a number of formulas, for example, formula (A)



wherein E can be, for example, hydroxyl, R can be, for example, hydrogen, and, when n is 1,  $R_1$  can be, for example, hydrogen and an alkyl of 1 to 18 carbon atoms,

and

formula (B)



#### wherein

E can be, for example, hydroxyl, R can be, for example, hydrogen, and when m is 1,  $R_2$  can be, for example, alkyl of 1 to 18 carbon atoms, or when m is 2,  $R_2$  can be, for example, alkylene of 1 to 12 carbon atoms.

Furthermore, according to Claim 9 of the application as filed the hindered amine compound of component (b) was, for example,

(b) bis(1-hydroxy-2,2,6,6-tetramethylpiperidin-4yl)sebacate, or

(u) 1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidine.

- IV. The Examining Division considered that the technical problem underlying the application in suit was the prevention of loss of brightness and enhancing the resistance to yellowing in pulp or paper which still contained lignin and the provision of novel compounds useful to this end. The solution of this problem by applying a hindered amine compound of formula I or II according to Claim 1 did not involve an inventive step, since it could be expected in view of the cited prior art that hindered amine compounds falling under the scope of Claim 1 would be effective in stabilising lignin containing pulp or paper.
- V. Oral proceedings before the Board were held on 26 April 2006.
- VI. The Appellant defended the patentability of the subject-matter of the present application on the basis of the claims as originally filed as main request and Claims 1 to 5 submitted during the oral proceedings as auxiliary request.

Claim 1 of the auxiliary request read as follows:

"A composition having reduced loss of brightness and enhanced resistance to yellowing which comprises

(a) a pulp or paper which still contains lignin, and

(b) an effective stabilizing amount of a hindered amine compound

wherein the compound of component (b) is

bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)sebacate;

1-hydroxy-2,2,6,6-tetramethyl-4-acetoxypiperidinium citrate;

1-oxyl-2,2,6,6-tetramethyl-4-acetamidopiperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-acetamidopiperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-acetamidopiperidinium bisulfate;

1-oxyl-2,2,6,6-tetramethyl-4-oxo-piperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-oxo-piperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-oxo-piperidinium
acetate;

1-oxyl-2,2,6,6-tetramethyl-4-methoxy-piperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-methoxy-piperidine;

1-hydroxyl-2,2,6,6-tetramethyl-4-methoxy-piperidinium
acetate;

1-oxyl-2,2,6,6-tetramethyl-4-acetoxypiperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-acetoxypiperidine;

1-oxyl-2,2,6,6-tetramethyl-4-propoxy-piperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-propoxy-piperidinium
acetate;

1-hydroxy-2,2,6,6-tetramethyl-4-propoxy-piperidine;

1-oxyl-2,2,6,6-tetramethyl-4-(2-hydroxy-4oxapentoxy)piperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-(2-hydroxy-4oxapentoxy)piperidinium acetate;

1-oxyl-2,2,6,6-tetramethyl-4-hydroxypiperidine;

1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidinium chloride;

1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidinium
acetate;

1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidinium bisulfate;

1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidinium
citrate;

bis(1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidinium)
citrate;

tris(1-hydroxy-2,2,6,6-tetramethyl-4hydroxypiperidinium) citrate;

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tetra(1-hydroxy-2,2,6,6-tetramethyl-4-
hydroxypiperidinium) ethylenediaminetetraacetate;
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tetra(1-hydroxy-2,2,6,6-tetramethyl-4-
acetamidopiperidinium) ethylenediaminetetraacetate;
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tetra(1-hydroxy-2,2,6,6-tetramethyl-4-oxopiperidinium)
ethylenediaminetetraacetate;

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penta(1-hydroxy-2,2,6,6-tetramethyl-4-
hydroxypiperidinium) diethylenetriaminepentaacetate;
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penta(1-hydroxy-2,2,6,6-tetramethyl-4-
acetamidopiperidinium) diethylenetriaminepentaacetate;
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penta(1-hydroxy-2,2,6,6-tetramethyl-4-oxopiperidinium)
diethylenetriaminepentaacetate;
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tri(1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidinium)
nitrilotriacetate;
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tri(1-hydroxy-2,2,6,6-tetramethyl-4-
acetamidopiperidinium) nitrilotriacetate;
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tri(1-hydroxy-2,2,6,6-tetramethyl-4-oxopiperidinium)
nitrilotriacetate;
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penta(1-hydroxy-2,2,6,6-tetramethyl-4-
hydroxypiperidinium)
diethylenetriaminepentamethylenephosphonate;
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penta(1-hydroxy-2,2,6,6-tetramethyl-4-
acetamidopiperidinium)
diethylenetriaminepentamethylenephosphonate;
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penta(1-hydroxy-2,2,6,6-tetramethyl-4-oxopiperidinium)
diethylenetriaminepentamethylenephosphonate."

- VII. Concerning the assessment of inventive step, the Appellant accepted that the prior document
  - (A) Pulp Bleaching Principles and Practise (1996),
     C.W.Dence, D.W.Reeve, eds., TAPPI Press, Atlanta,
     Section III, Chapter 5, C. Heitner: "Chemistry of
     Brightness Reversion and Its Control", pages 183-212,

which was acknowledged in the application in suit, represented the closest prior art. He argued that from this document it could be derived that light or oxygen induced yellowing of lignin containing pulp or paper was attributable to chemical changes in the lignin and that document (1) did not relate to the stabilisation of lignin at all. In fact, this last mentioned document was concerned with the stabilisation of hydrophobic organic materials rendering it necessary that the hindered amine compounds being used as stabilisers were soluble in organic solvents. Consequently, it rather tended to deter the skilled person from utilising such stabilising compounds for the application in hydrophilic pulp and paper. He emphasised that the hindered amine compounds use according to the application in suit and in particular those indicated in Claim 1 of the auxiliary request were surprisingly effective in reducing yellowing of pulp and paper as shown in the examples of the present application.

VIII. The Appellant requested that the decision under appeal be set aside, and that a patent be granted on the basis of the originally filed claims or, in the alternative, on the basis of Claims 1 to 5 of the auxiliary request filed during the oral proceedings.

He also requested the reimbursement of the appeal fee.

IX. At the conclusion of the oral proceedings the Board's decision was pronounced.

#### Reasons for the Decision

1. The appeal is admissible.

Main request

- 2. Inventive step
- 2.1 The sole issue with respect to this request consists in deciding whether or not the subject-matter of the claims of the application as filed involves an inventive step.
- 2.2 Article 56 EPC states that an invention is held to involve an inventive step if, having regard to the state of the art (in the sense of Article 54(2) EPC), it is not obvious to a person skilled in the art.
- 2.3 For deciding whether or not a claimed invention meets this criterion, the Boards of Appeal consistently apply the problem and solution approach, which involves essentially identifying the closest prior art,

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determining in the light thereof the technical problem which the claimed invention addresses and successfully solves, and examining whether or not the claimed solution to this problem is obvious for the skilled person in view of the state of the art.

According to the established jurisprudence of the Boards of Appeal the "closest prior art" for assessing inventive step is normally a prior art document disclosing subject-matter conceived for the same purpose as the claimed invention and having the most relevant technical features in common.

2.4 In agreement with the Appellant the Board considers therefore that Document (A) represents the prior art closest to the claimed invention and, hence, the starting point in the assessment of inventive step.

> This document discloses that light-induced yellowing of lignin containing papers and pulps occurs by photooxidation of lignin and that the lignin can be stabilised by quenching free radicals using scavengers, in particular ascorbates, thiols, thioethers, formates, dienes and aldehydes (see page 205, under "Free radical scavengers"; and pages 209 and 210, under "Summary", in particular page 209, right column, first paragraph, and page 210).

2.5 As indicated in the application in suit, the technical problem to be solved in the light of this closest prior art consists in providing a further way of inhibiting undesirable light-induced yellowing of lignin containing pulp and/or paper (see page 3, penultimate paragraph, last sentence).

- 2.6 The application in suit suggests as the solution to this technical problem the provision of pulp or paper compositions comprising an effective stabilising amount of hindered hydroxyl amines, hindered oxyl amines, or their salts as defined in Claim 1, and more particular compounds such as listed in the dependent Claim 9.
- 2.7 Having regard to the technical information provided in the examples of the application in suit the Board finds it plausible that the technical problem as defined above has been solved.
- 2.8 The next question to be answered is whether a skilled person starting from document (A) and by following the suggestions made in the cited prior art as a whole, when trying to solve the technical problem as defined above, would arrive at a pulp or paper composition falling within the scope of the present claims.
- 2.9 Document (A) discloses as indicated above under point 2.4 - that lignin containing pulps or papers can be stabilised against light-induced yellowing by adding free radical scavengers selected from several different classes of compounds. However, this document does not give any pointer to the skilled person that the technical problem underlying the application in suit could also be solved by using hindered amines as defined in the present claims.
- 2.10 When aiming at providing a further way of inhibiting undesirable light-induced yellowing of lignin containing pulp and/or paper, it is a matter of course that the person skilled in the art would turn his

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intention to that prior art in the field of stabilising organic materials against the deleterious effects of light and oxygen.

2.11 As a skilled person he would then be struck by document (1) which not only aims at improving the stabilisation of ambient curable or acid catalysed thermosetting coating systems against the deleterious effects of light and oxygen and at reducing loss of gloss and yellowing of the cured coatings, but also teaches that hydroxyl substituted hindered amines containing a group of the formula



where R is hydrogen or methyl (see page 2, lines 3 to 35),

are particularly effective in stabilising organic materials including a large variety of polymeric materials, such as natural cellulose, against the degradative effects of actinic stimuli (see page 2, lines 3 to 17 and page 23, line 35 to page 25, line 4, in particular page 24, line 63).

Suitable hydroxyl substituted hindered amines are various groups of compounds having, for instance, formula A



wherein

R can be, for example, hydrogen, and,

when m is 1,

 $R_1$  can be, for example, hydrogen, an alkyl of 1 to 18 carbon atoms, a monovalent acyl radical of an aliphatic carboxylic acid having 2 to 18 carbon atoms (see page 5, lines 22 to 28), or

when m is 2,

 $R_1$  can be, for example, a divalent acyl radical of an aliphatic dicarboxylic acid having 2 to 18 carbon atoms (see page 5, lines 55 to 58).

This group of hindered amines having formula A represents suitable compounds in the sense of the present application in that it overlaps with the variety of compounds defined in Claim 1 of the application as filed and also with the more particular group of compounds defined in Claim 6 of the application as filed with respect to the formulas (A) and (B) (see under point III above).

Moreover, this document (1) specifies as suitable hydroxyl substituted hindered amines the compounds

1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidine (see page 9, line 1, compound nr. 1)

and

bis(1-hydroxy-2,2,6,6-tetramethylpiperidin-4yl)sebacate (see page 9, line 5, compound nr. 5),

which are also preferred stabilising compounds of the application in suit (see Claim 9, compounds (u) and (b) respectively; and Examples 1 and 6).

In view of these considerations the Board concludes that the cited prior art, in particular document (1) teaching that hydroxyl substituted hindered amines as claimed in the application in suit can be used to stabilise a large variety of organic materials including lignin containing natural cellulose, gives the skilled person an incentive of how to solve the problem underlying the present application with a reasonable expectation of success.

2.12 It is true, that document (1) does not explain the mechanism leading to the stabilising effects, let alone the stabilising activity of the hindered amines with respect to lignin. However, in view of the fact that it has been explicitly taught in said document that natural cellulose, i.e. cellulose which necessarily contains lignin, can be stabilised, it is the Board's position that merely not explicitly stating this implied technical information does not deter the skilled person from applying the teaching of document (1) to pulp or paper compositions as claimed in the application in suit. Furthermore, the Appellant's contentions that according to document (1) the suitable hydroxyl substituted hindered amine stabilisers should be soluble in organic solvents normally used in coating applications such as methyl amyl ketone, xylene, n-hexyl acetate, alcohol and the like (see page 11, lines 31 and 32) and that this technical information would deter the skilled person from applying the teaching of document (1) is not relevant, since the present claims do not exclude the use of such organic solvents (see also page 66, first paragraph, of the application as filed).

2.13 As a result, the Appellant's main request is not allowable as the subject-matter of the present Claims 1,6 and 7 lacks inventive step pursuant Article 56 EPC.

Auxiliary request

3. Amendments (Article 123(2) EPC)

In claim 1 according to the auxiliary request the subject-matter has been limited to that of Claim 9 of the application as filed, whereby the compounds

1-hydroxy-2,2,6,6-tetramethyl-4-hydroxypiperidine (former compound (u))

and

bis(1-hydroxy-2,2,6,6-tetramethylpiperidin-4yl)sebacate (former compound (b)) have been deleted. Thus that amendment complies with the requirements of Article 123(2) EPC.

4. Inventive step

4.1 Since present Claim 1 comprises compositions wherein the compound of component (b) is

1-hydroxy-2,2,6,6-tetramethyl-4-methoxy-piperidine,

1-hydroxy-2,2,6,6-tetramethyl-4-acetoxy-piperidine, or

1-hydroxy-2,2,6,6-tetramethyl-4-propoxy-piperidine,

i.e. compounds indicated in Claim 9 as originally filed as (k), (n) and (q), respectively, the considerations of the Board with respect to the issue of inventive step for the main request indicated above (see point 2.11 above indicating suitable compounds according to document (1) of formula A, wherein m is 1 and  $R_1$  is alkyl or acyl) essentially also apply to the present auxiliary request.

- 4.2 In view of these considerations, a skilled person faced with the technical problem defined above would arrive at compositions as presently claimed without any inventive activity.
- 4.3 Thus, the Board concludes that the subject-matter of Claim 1 of the auxiliary request does not involve an inventive step either.

- 5. Reimbursement of the appeal fee
- 5.1 According to Rule 67 EPC, reimbursement of the appeal fee shall be ordered where the Board of Appeal deems an appeal to be allowable and if such reimbursement is equitable by reason of a substantial procedural violation.
- 5.2 In the present case, the Appellant has not been successful on appeal to the extent requested. Thus, already for this reason the reimbursement of the appeal fee has to be refused.

# Order

# For these reasons it is decided that:

- 1. The appeal is dismissed.
- The request for reimbursement of the appeal fee is refused.

The registrar:

The Chairman:

N. Maslin

A. Nuss