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**Datasheet for the decision  
of 7 December 2017**

**Case Number:** T 0577/14 - 3.3.10  
**Application Number:** 08862288.1  
**Publication Number:** 2231586  
**IPC:** C07C215/82, C07C251/22,  
C07D233/64, C07D295/073,  
A61K8/41, A61Q5/10, C09B55/00  
**Language of the proceedings:** EN

**Title of invention:**

AZOMETHINE DIRECT DYES OR REDUCED PRECURSORS OF AZOMETHINE  
DIRECT DYES OBTAINED FROM 2-ALKYLRESORCINOLS, AND HAIR DYEING  
PROCESS USING THESE DYES OR PRECURSORS

**Patent Proprietor:**

L'Oréal

**Opponent:**

The Procter & Gamble Company

**Headword:**

AZOMETHINE DIRECT DYES/L'OREAL

**Relevant legal provisions:**

EPC Art. 54  
RPBA Art. 13(1), 13(3)

**Keyword:**

Main request and auxiliary requests 1 to 4 - Novelty - (no)  
Late-filed auxiliary request 5 - procedural economy

**Decisions cited:**

**Catchword:**



**Beschwerdekammern**  
**Boards of Appeal**  
**Chambres de recours**

Boards of Appeal of the  
European Patent Office  
Richard-Reitzner-Allee 8  
85540 Haar  
GERMANY  
Tel. +49 (0)89 2399-0  
Fax +49 (0)89 2399-4465

Case Number: T 0577/14 - 3.3.10

**D E C I S I O N**  
**of Technical Board of Appeal 3.3.10**  
**of 7 December 2017**

**Appellant:** L'Oréal  
(Patent Proprietor) 14, rue Royale  
75008 Paris (FR)

**Representative:** Fevrier, Murielle Françoise E.  
L'Oréal  
Service DIPI  
9 Rue Pierre Dreyfus  
92110 Clichy (FR)

**Respondent:** The Procter & Gamble Company  
(Opponent) One Procter & Gamble Plaza  
Cincinnati, Ohio 45202 (US)

**Representative:** Herzog, Fiesser & Partner Patentanwälte PartG  
mbB  
Isartorplatz 1  
80331 München (DE)

**Decision under appeal:** **Decision of the Opposition Division of the  
European Patent Office posted on 20 January 2014  
revoking European patent No. 2231586 pursuant to  
Article 101(3) (b) EPC.**

**Composition of the Board:**

**Chairman** P. Gryczka  
**Members:** J.-C. Schmid  
T. Bokor

## Summary of Facts and Submissions

I. The Appellant (Proprietor of the patent) lodged an appeal against the decision of the Opposition Division revoking the European patent No. 2 231 586.

II. Notice of opposition had been filed by the Respondent (opponent) requesting revocation of the patent in suit in its entirety on the grounds of lack of novelty and inventive step (Article 100(a) EPC), based inter alia on document

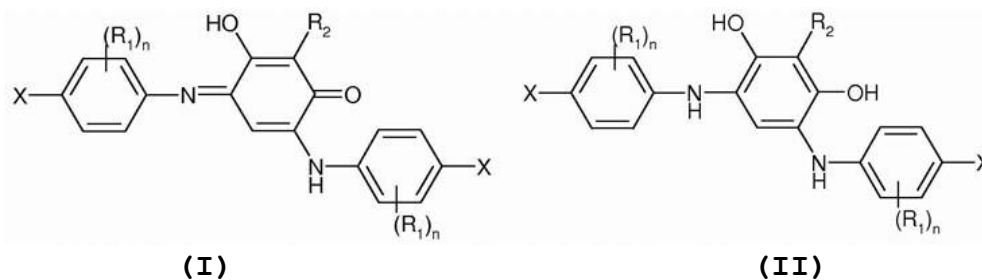
(1) C. Zviak "Oxidation Coloring", The Science of Hair Care, Edited by Charles Zviak, Marcel Dekker, INC. New York and Basel, 1986, Chapter 8, pages 263 to 286.

In the decision under appeal, the Opposition Division found that the subject-matter of claim 8 of the then pending main request lacked novelty and rejected the the then pending auxiliary request for lack of clarity.

III. At the oral proceedings before the board held on 7 December 2017, the Appellant defended the maintenance of the patent in suit on the basis of a main request filed with the grounds of appeal dated 28 May 2014, four auxiliary requests filed with a letter dated 31 August 2017 and a fifth auxiliary request filed during the oral proceedings before the Board.

Claim 1 of the main request reads as follows:

"1. Compound of formula **(I)** or **(II)** :



salts thereof with an organic or mineral acid, geometrical isomers thereof, tautomers thereof, and solvates thereof such as the hydrates; in which formula **(I)** or **(II)** :

- **R<sub>1</sub>**, which may be identical or different, represent:
  - a chlorine atom;
  - a (C<sub>1</sub>-C<sub>3</sub>) alkyl radical optionally substituted with one or more hydroxyl groups;
  - a (C<sub>1</sub>-C<sub>3</sub>) alkoxy radical optionally substituted with one or more hydroxyl groups;
- **R<sub>2</sub>** represents a (C<sub>1</sub>-C<sub>3</sub>) alkyl radical;
- **X**, which may be identical or different, represent:
  - a hydroxyl radical;
  - a radical -NR<sub>3</sub>R<sub>4</sub> with R<sub>3</sub> and R<sub>4</sub> representing, independently of each other:
    - (i) a hydrogen atom;
    - (ii) a C<sub>1</sub>-C<sub>5</sub> alkyl radical optionally substituted with one or more groups chosen from hydroxyl, (C<sub>1</sub>-C<sub>3</sub>) alkoxy, amino, (C<sub>1</sub>-C<sub>3</sub>) alkylamino, di(C<sub>1</sub>-C<sub>3</sub>) alkylamino, aminocarbonyl, carboxylic -COOH, sulfonic -SO<sub>3</sub>H, tri (C<sub>1</sub>-C<sub>3</sub>) alkylammonium and (C<sub>1</sub>-C<sub>3</sub>) alkylimidazolium;
  - a pyrrolidinyl radical optionally substituted with a group chosen from hydroxyl, (C<sub>1</sub>-C<sub>3</sub>) alkoxy, amino, (C<sub>1</sub>-C<sub>3</sub>) alkylamino, di (C<sub>1</sub>-C<sub>3</sub>) alkylamino, tri (C<sub>1</sub>-C<sub>3</sub>) alkylammonium and (C<sub>1</sub>-C<sub>3</sub>) alkylimidazolium;
  - a piperidine radical optionally substituted with a group chosen from hydroxyl, (C<sub>1</sub>-C<sub>3</sub>) alkoxy, amino, (C<sub>1</sub>-C<sub>3</sub>) alkylamino, di (C<sub>1</sub>-C<sub>3</sub>) alkylamino, tri (C<sub>1</sub>-C<sub>3</sub>)

alkylammonium and (C<sub>1</sub>-C<sub>3</sub>) alkyimidazolium;

- n represents an integer between 0 and 3 inclusive;  
and n is zero for the compound of formula (I);

it being understood that when X and/or R<sub>3</sub> and/or R<sub>4</sub> comprise a cationic group, the electrical neutrality of the compound of formula **(I)** or **(II)** is achieved with an anionic counterion or a mixture of anionic counterions that are cosmetically acceptable, for instance chlorides, bromides and sulfates."

Claim 1 of auxiliary request 1 differs from claim 1 of the main request in that the compounds of formula (II) have been deleted.

Claim 1 of auxiliary request 2 is identical to claim 1 of the main request.

Claim 1 of auxiliary request 3 is identical to claim 1 of auxiliary request 1.

Claim 1 of auxiliary request 4 differs from claim 1 of auxiliary request 3 in that R<sub>3</sub> and R<sub>4</sub> can no longer represent hydrogen.

Claim 1 of auxiliary request 5 is directed to a process for dyeing keratin fibres in which a suitable dye composition is applied to the material, the suitable composition having a pH between 6 and 11 comprising, in a suitable cosmetic medium at least one compound of formula (I) or (II) according to claim 1 of the main request wherein n represents 0.

- IV. According to the Appellant, the subject-matter of claim 1 of the main and auxiliary requests 1 to 4 was novel with respect to document (1), since this document

related to oxidation dyes and there was no proof that a tri-nuclear compound according to claim 1 was formed by mixing one equivalent coupler with one equivalent base. Furthermore, there was no absolute certainty that the mass spectra shown in document (12) corresponded to a tri-nuclear compound. Claim 1 of auxiliary request 5 was restricted to a process for dyeing hair with a direct dye and was based on claim 9 of the main request. Auxiliary request 5 should therefore be admitted into the proceedings.

V. According to the Respondent, the subject-matter of claim 1 of the main and auxiliary request 1 to 4 lacked novelty with respect to document (1). This document disclosed a dyeing composition prepared by mixing equimolar quantities of coupler and base. A tri-nuclear compound of formula (1) according to claim 1 of the main and auxiliary request 1 to 3 was inevitably formed by mixing equimolar quantities of para phenylenediamine and 2-methylresorcinol. This was confirmed by analysing the products formed, as shown in the experimental report filed with the letter dated 15 December 2014 (document (12)). Document (1) also disclosed dyes formed mixing 2-methyl resorcinol with para aminophenol or with N,N-bis-(B-hydroxyethyl)-p-phenylenediamine. The subject-matter of claim 1 of auxiliary request 4 thus also lacked novelty with respect to document (1).

VI. The Appellant (patent proprietor) requests that the decision under appeal be set aside, and the patent be maintained in an amended form on the basis of the main request filed with the grounds of appeal dated 28 May 2014, or on the basis of any of the 1<sup>st</sup> to 4<sup>th</sup> auxiliary requests filed with letter dated 31 August 2017, or on the basis of the 5<sup>th</sup> auxiliary request filed in the oral proceedings before the Board.

- VII. The Respondent (opponent) requested that the appeal be dismissed. He also requests that auxiliary requests 1, 2, 4 and 5 should not be admitted into the proceedings.
- VIII. At the end of the oral proceedings the decision of the Board was announced.

### **Reasons for the Decision**

1. The appeal is admissible.

#### *Novelty*

2. *Main request*

Document (1) relates to oxidation colouring obtained by mixing the so called "oxidation dyes" or "dye precursors" with an oxidizing composition. The oxidation dyes comprise primary intermediates or bases and couplers. Examples of bases are disclosed in table 1 at page 266, including p-phenylenediamine (base B<sub>1</sub>), p-aminophenol (base B<sub>4</sub>) and N,N-bis-(β-hydroxyethyl)-p-phenylenediamine (B<sub>9</sub>), and the couplers are disclosed in table 2 at page 266 including resorcinol (coupler C<sub>9</sub>) and 2-methylresorcinol (coupler C<sub>14</sub>).

Document (1) discloses that upon mixture with an oxidizing composition these dye precursors produce di-, tri- and polynuclear dyes (see chapter II, starting at page 268). The mechanism of the cooxidation of p-phenylenediamine (base B<sub>1</sub>) with resorcinol (coupler C<sub>13</sub>) is disclosed in chapter II C, at pages 270 and 271, and reveals that a tri-nuclear dye [VI] is formed by mixing p-phenylenediamine with resorcinol.



Table 3 discloses the shade of hair obtained by applying for 30 minutes dyeing compositions prepared by mixing an equimolecular mixture of various pairs of bases (B) and couplers (C) with an oxidizing composition prior to use (see page 279, bottom).

In particular, document (1) discloses a dyeing composition obtained by mixing equimolar quantities of base B<sub>1</sub> (para phenylenediamine) and coupler C<sub>14</sub> (2-methylresorcinol) with an oxidizing agent (see page 284, first entry). According to the mechanism of oxidation disclosed in document (1), a tri-nuclear dye is produced, which is a compound of formula (I) of claim 1 of the main request, wherein both X represent NH<sub>2</sub>, R<sub>2</sub> represents methyl and n is 0, henceforth referred to as compound (1a) (see patent-in-suit, example 1, and first compound of dependent claim 7 of the main request).

According to the Appellant, there was no absolute certainty that the tri-nuclear compound (1a) was formed, since document (1) envisaged further couplings to form poly-nuclear dyes. Furthermore only one equivalent of the base was used, while the formation of the tri-nuclear compound (1a) required two equivalents.

However, the teaching of document (1) applies to dyeing compositions made from the mixtures of equimolar quantities of bases and couplers (see third and fourth full paragraph of page 272). Furthermore, the formation of the tri-nuclear dye was confirmed by the experiment filed by the Respondent with the letter dated 15 December 2014 reproducing the dyeing composition prepared by mixing equimolar quantities of para phenylenediamine and 2-methylresorcinol with an oxidizing agent (document (12)). The presence of the

tri-nuclear compound (1a) in the dyeing composition was identified by liquid chromatography coupled with mass spectrometry using electrospray ionization.

During the oral proceedings the Appellant stated that there was no absolute certainty that the peak of the mass spectra at  $m/z=335$  was the molecular ion  $(M+H)^+$  of the expected tri-nuclear compound, as argued by the Respondent, because this peak could also be a fragment of a higher poly-nuclear compound. However, electrospray ionization is a soft ionization technique which is used for production of ions without fragmentation of thermally labile large molecules. Furthermore, document (1) unambiguously foresees the production of the tri-nuclear compound. As the Appellant did not provide evidence to the contrary, namely that compound (1a) is not produced by the oxidative dyeing composition comprising equimolar quantities of para phenylenediamine and 2-methylresorcinol, the Appellant's argument is rejected by the Board as unfounded.

The Appellant further alleged that the composition of document (1) comprising equimolar quantities of base phenylenediamine and 2-methylresorcinol did not comprise compound (1a), since the shade obtained with the composition of document (1) was medium yellow-brown, whereas the shade obtained with the composition comprising compound (1a) of the patent-in-suit was pale yellow.

However, the dyeing conditions are not the same in document (1) and in the patent-in-suit (pH, support, quantities of dye, presence of other dyes). Hence the fact that a different shade is obtained with composition (A) of the patent-in-suit comprising

compound (1a) does not permit to conclude that compound (1a) is not present in the oxidative dye composition of document (1).

The Appellant's arguments being rejected, the Board comes to the conclusion that the dye composition prepared by mixing equimolar quantities of base B<sub>1</sub> and C<sub>14</sub> with an oxidizing agent (first entry of table 3 of document (1)) comprises compound (1a). Accordingly, the subject-matter of claim 1 of the main request lacks novelty over document (1).

3. *Auxiliary requests 1 to 3*

Compound (1a) is encompassed by claim 1 of auxiliary requests 1 to 3 (see also dependent claim 5 of auxiliary requests 1 and 3 and dependent claim 7 of auxiliary request 2). The Appellant did not dispute this.

Therefore, these requests must be rejected for lack of novelty too.

4. *Auxiliary request 4*

Document (1) discloses further dyeing compositions, in particular such compositions which are obtained by mixing a composition comprising equimolar quantities of p-aminophenol (base B<sub>4</sub>) and 2-methyl resorcinol (coupler C<sub>14</sub>) with an oxidizing agent (see table 3 on page 294, fourth entry), thus producing a compound of formula (I) wherein both X represent a hydroxyl radical, R<sub>2</sub> represents methyl and n is 0, which is compound (1c) according to the patent-in-suit (see example 3 of the patent-in-suit, dependent claim 7 of auxiliary request 4).

Hence, auxiliary request 4 therefore is rejected for lack of novelty.

5. The Respondent requested that late filed auxiliary requests 1, 2 and 4 not be admitted into the proceedings. However, since these requests are rejected for lack of novelty, an answer to the question whether or not these requests can be admitted into the proceedings has no impact on the decision. Therefore, there is no need to take a formal decision on the admission of these requests into the proceedings.

6. *Auxiliary request 5 - Admission*

This request was filed by the Appellant after the Board had indicated its conclusion that the subject-matter of claim 1 of the main request was not novel. The admission of this late-filed request is a matter for the Board's discretion which should be exercised in view of the current state of the proceedings and the need for procedural economy (Article 13(1) RPBA). Furthermore, the request was filed after oral proceedings have been arranged, so that Article 13(3) RPBA also applies.

Claim 1 of auxiliary request 5 has been limited to a process for dyeing keratin fibres involving the compounds of formula (I) and (II) of claim 1 of the main request, wherein n represents 0, and is based on process-claim 19 of the main request. The Appellant argues that this composition no longer concerns an oxidative dyeing process.

However, the Appellant was well aware of the novelty objection against the product-claims from the written

opposition and appeal proceedings, but chose not to file amended process-claims aiming to overcome this objection.

Admitting this auxiliary request shortly before the end of the oral proceedings would have been contrary to a fair and efficient conduct of the proceedings, since one party provided its objections in due time whereas the other waited until the last minute before filing a request aiming to overcome a long-standing objection. In addition, the Respondent would have had to present for the first time his observations and possible objections against this request at the oral proceedings. In light of the arguments presented by the Appellant, it appears that the chemical character of the dyeing process changed significantly as compared to the previously discussed processes, and this new process would have to be discussed essentially without any preparation either by the Respondent or the Board. Thus the Board deems that the subject-matter of this request raises issues with respect to novelty and inventive step which neither the Respondent nor the Board can be reasonably expected to deal with in the oral proceedings. For this reason alone, the request cannot be admitted pursuant to Article 13(3) RPBA.

In *inter partes* proceedings the parties are expected to play an active role and to provide requests at an early stage. This requires *inter alia* that the Appellant (patent proprietor) carefully studies the objections raised by respondent (opponent) and reacts to those which it deems to be particularly relevant, e.g. by filing amended claims. Therefore, the Appellant should not have delayed the filing of auxiliary request 5 until the Board announced its conclusion on novelty with respect to the main request.

Hence, the late-filed auxiliary request 5 is not admitted into the proceedings under Articles 13(1) and 13(3) RPBA.

**Order**

**For these reasons it is decided that:**

The appeal is dismissed.

The Registrar:

The Chairman:



C. Rodríguez Rodríguez

P. Gryczka

Decision electronically authenticated