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**Datasheet for the decision
of 5 December 2011**

Case Number: T 0634/07 - 3.3.01

Application Number: 03734601.2

Publication Number: 1470196

IPC: C09B 67/00, C09B 29/00,
C09B 29/36

Language of the proceedings: EN

Title of invention:
Aqueous Dye Solutions

Applicant:
Ciba Holding Inc.

Opponent:
BASF Aktiengesellschaft

Headword:
Azodye composition/CIBA

Relevant legal provisions:
EPC Art. 54, 111(1)

Keyword:
"Novelty: yes"
"Remittal: yes, also for consideration of continuation of the
proceedings after withdrawal of the opposition"

Decisions cited:
T 0789/89

Catchword:
-



Case Number: T 0634/07 - 3.3.01

D E C I S I O N
of the Technical Board of Appeal 3.3.01
of 5 December 2011

Appellant: Ciba Holding Inc.
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 20 February 2007
revoking European patent No. 1470196 pursuant
to Article 102(1) EPC 1973.

Composition of the Board:

Chairman: P. Ranguis
Members: G. Seufert
C.-P. Brandt

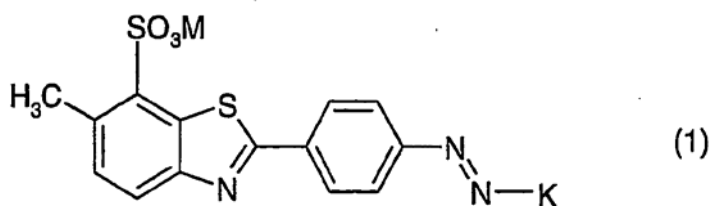
Summary of Facts and Submissions

I. The Appellant (Proprietor of the patent) filed an appeal against the decision of the Opposition Division dated 20 February 2007 revoking European patent no. 1 470 196.

II. The patent was granted on the basis of 12 claims, independent claims 1, 10, 11 and 12 reading as follows:

1. An aqueous dye solution, comprising

a) 5 to 30% by weight of a dye of the formula

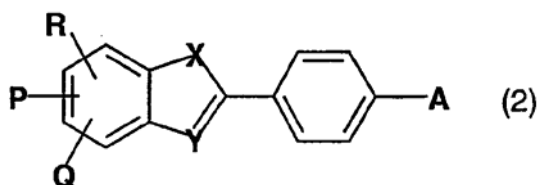


in which

K is a residue of a coupling component of the acetoacetanilide, pyridone, pyrazolone or pyrimidine series and

M is hydrogen, an alkali metal or alkaline earth metal, ammonium or alkylammonium,

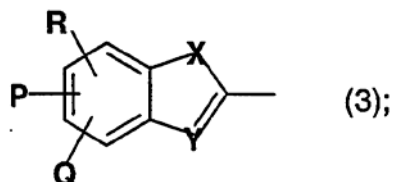
b) 0.05 to 5% by weight of one or more compounds of the formula



in which

A represents $-NR_1R_2$, $-NHCOR_1$, $-CN$, halogen, $-NO_2$, $-OH$, $-OR_1$, hydrogen, C_1 - C_4 alkyl, C_2 - C_4 alkenyl, C_2 - C_4 alkinyl,

$-\text{CO}_2\text{M}$, $-\text{CO}_2\text{R}_1$ or $-\text{CONR}_1\text{R}_2$, wherein R_1 and R_2 each, independently of one another, represent hydrogen or C_1 - C_4 alkyl and M is as previously defined, or a residue of the formula



X represents O , S or NR_1 , R_1 being as previously defined;

Y represents N or CR_1 , R_1 being as previously defined and

P , Q and R each, independently of one another, represent hydrogen, C_1 - C_4 alkyl, $-\text{SO}_3\text{M}$, $-\text{PO}_3\text{M}$, $-\text{CO}_2\text{M}$, $-\text{OH}$, $-\text{NO}_2$ or $-\text{COR}_1$, M and R_1 being as previously defined,

- c) an organic or an inorganic base or mixtures thereof,
- d) if desired, further additives and
- e) water.

10. A process for the preparation of dye solutions, which comprises stirring a dye of formula 1, according to claim 1, with a mixture of water, one or more compounds of formula (2), according to claim 1 and, if desired, further additives.

11. Use of an aqueous dye solution, according to claim 1, for dyeing paper.

12. Paper, which has been dyed with an aqueous dye solution, as defined in to claim 1.

III. In this decision the following numbering will be used to refer to the documents:

- (1) DE 40 30 915 A
- (2) DE 35 11 752 A
- (3) EP 0 553 672 A
- (4) WO 01/90257 A
- (5) DE 34 34 923 A
- (6) WO 01/32786 A
- (7) DE 27 54 486 A
- (8) US 2,927,939

IV. Opposition was filed requesting revocation of the patent in suit in its entirety on the basis of lack of novelty and lack of inventive step (Article 100(a) EPC).

V. The Opposition Division, in view of the "experimental results" provided by the Opponent, held that the patent in suit was not novel over example 7 of document (1).

VI. With the statement of grounds of appeal the Appellant maintained the set of claims on file, namely the claims as granted, and filed document (8). In addition to the substantive issue addressed in the statement of grounds of appeal, the Appellant requested reimbursement of the appeal fee without, however, providing any reasons for this request. Oral proceedings were requested should the Board intend to reject the Appellant's requests.

VII. By letter of 6 May 2009 the Respondent (Opponent), after having originally requested dismissal of the appeal on the basis that the claims as granted lacked novelty, withdrew its opposition.

- VIII. In a communication pursuant to Article 15(1) of the Rules of the Procedure of the Boards of Appeal (RPBA) accompanying the summons to oral proceedings, the Board expressed its preliminary view. In particular, the Board considered the evidence on file to be insufficient to demonstrate that an aqueous dye solution according to claim 1 of the patent in suit was the inevitable result of example 7 of document (1). The Board indicated its intention to remit the case for further prosecution to the department of first instance and informed the Appellant that the sole issue to be discussed during oral proceedings would be the Appellant's request for reimbursement of the appeal fee.
- IX. In reply to the Board's communication, the Appellant with letter of 14 October 2011 withdrew its request for reimbursement of the appeal fee. Subject to the case being remitted to the department of first instance for further prosecution, the Appellant also withdrew its auxiliary request for oral proceedings.
- X. By letter dated 10 November 2011 the oral proceedings were cancelled.
- XI. According to the Appellant the claimed subject-matter was novel over example 7 of document (1). The Appellant did not dispute that the Opponent's experiments led to the reported results, but rather that the conditions under which these experiments were performed did not correspond to the conditions originally employed in the process disclosed in document (1), since no excess of nitrite at the end of the reaction could be observed by the Opponent in any of the experiments. Furthermore, the use of the same quantity of reagents, as in the

Opponent's first experiment, did not guarantee that the same effective amounts were used, since the active content of nitrite may not be the same. In support of its arguments the Appellant referred to document (8), which described the auto degradation of alkyl nitrites. The Appellant considered that exactly this point was demonstrated by the Opponent's second experiment, wherein a higher amount of nitrite was used, which nevertheless was completely consumed resulting in a reduced amount of starting material. The fact that the Opponent's experiments did not give the active content of the reagents and that no remaining dinitrite, contrary to example 7 of document (1), was observed at the end of the reaction, even by using a higher amount than employed in example 7 of document (1), convincingly showed that the conditions of document (1) were not met in the Opponent's experiments. According to the Appellant, this relevant point was incorrectly assessed by the Opposition Division, which apparently was of the opinion that by using identical amounts of reagents the conditions of document (1) were automatically fulfilled.

- XII. The Appellant requested that the decision of the Opposition Division be set aside and that a corresponding patent be granted on the basis of the claims on file.

Reasons for the Decision

1. The appeal is admissible.

2. Procedural matters

In the present case, the Opposition Division had revoked the patent. The withdrawal of the opposition by the Respondent/Opponent has no direct procedural significance other than that the Respondent/Opponent ceases to be a party to the appeal proceedings as far as substantive issues are concerned (see decision T 789/89, OJ EPO 1994, 482).

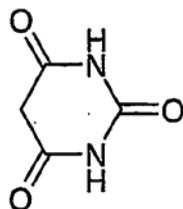
Claims as granted

3. Novelty

- 3.1 In the decision under appeal, the Opposition Division concluded that the patent in suit lacked novelty over example 7 of document (1).

Example 7 of document (1) discloses the reaction of dehydrothio-p-toluidinesulphonic acid (i.e. 2-(4-aminophenyl)-6-methyl-7-benzothiazolesulphonic acid) with neopentyl glycol dinitrite in water resulting in the formation of the corresponding diazonium salt. The remaining nitrite excess is destroyed with amidosulphonic acid and to the resulting suspension there are sequentially added barbituric acid and, within half an hour, lithium hydroxide, an aqueous solution of triethanol amine and an aqueous solution of diethanol amine. Water is then added in an amount to obtain 600g of the aqueous dye solution. The dye according to this example falls within the definition of the formula (1) of the patent in suit, with K being a coupling compound of the pyrimidine series, in

particular a compound according to the following formula



Example 7 does not explicitly disclose the presence of a compound according to formula (2) of the patent in suit. In fact nowhere in document (1) is the presence of such a compound disclosed. It is, however, noted that the starting material, i.e. dehydrothio-p-toluidinesulphonic acid, falls within the definition of formula (2) (i.e. Q, R and P = SO₃M with M = H, C₁-alkyl and H, X = S, Y = N, and A = NR₁R₂ with R₁/R₂ = H). Thus, document (1) would be novelty destroying for the subject-matter of the patent in suit, if it could be established beyond doubt that the presence of the starting material was the inevitable result of example 7 of document (1), as asserted by the Opponent.

- 3.2 The Opposition Division based its conclusion of lack of novelty on "experimental results", i.e. an asserted replication of example 7 of document (1), provided by the Opponent. The Opposition Division considered that it had no reasons to doubt that the Opponent's experimental results were in agreement with the disclosure of example 7 of document (1) or that they were flawed, in particular with regard to the selected reaction temperature during and the presence of nitrite after the diazotization. By providing these data the Opponent, according to the Opposition Division, had discharged himself of the burden of proof.

3.3 The Board, however, notes that the Opponent did not in fact provide any evidence which would allow an objective evaluation of whether or not example 7 of document (1) was correctly replicated, for example by providing the experimental set-up, the batch size, an experimental protocol, preferably by the person who carried out the experiment, describing the exact reaction conditions including information on what temperature or temperature control had been used during each step or whether and how the dinitrite excess had been determined. The notice of opposition merely states that example 7 of document (1) was replicated, which is no more than the subjective assessment by the Opponent himself, and that the reaction product was examined by using analytical HPLC. The conditions of the HPLC analysis were explicitly described in the notice of opposition, but no experimental evidence, for example the chromatogram of the reaction product obtained by replicating example 7 and the reference data for the retention time of the analytes, i.e. the dye and the dehydrothio-p-toluidinesulphonic acid, was provided. In this context, the notice of opposition again merely states that the dehydrothio-p-toluidinesulphonic acid and the dye were identified via their characteristic retention times and that according to this analysis dehydrothio-p-toluidinesulphonic acid was present. It is also stated in the notice of opposition that the amount of dye was 15.8% by weight and the amount of dehydrothio-p-toluidinesulphonic acid was 2.4% by weight without, however, providing information on how these amounts were determined.

3.4 In these circumstances, it is, in the Board's judgement, not possible to assess conclusively and objectively whether or not the Opponent's experiment mentioned in the notice of opposition is in fact a replication of example 7 of document (1), with the consequence that it cannot properly be established whether the aqueous dye solution of the prior art example inevitably comprises a compound according to formula (2) of the patent in suit in the required amount. The Board, therefore, does not agree with the Opposition Division position that the "experimental results", which for the alleged replication of example 7 of document (1) are in fact merely unsupported statements by the Opponent, discharged the Opponent's burden of proof.

3.5 This failure to provide sufficient evidence for its assertion of implicit lack of novelty is not remedied by the Opponent's further "experimental results" provided during the opposition proceedings (see Opponent's letter of 1 December 2006). According to the Opponent an experiment was carried out which differs from example 7 of document (1) in that the amount of neopentyl glycol dinitrite was **increased** in order to demonstrate that even with an increased amount of starting material the diazotization does not proceed to completion. Attached to the Opponent's letter is an "experimental protocol" which is an almost literal copy of the wording used in document (1). Although, according to the Opponent, the disappearance of the dinitrite was controlled, the "experimental protocol" does not contain any information on how or with what frequency this was done, which, in the Board's judgement, raises doubts as to whether this attached "experimental protocol" is in fact the actual

experimental protocol by the person who carried out the experiment or whether it is merely a subjective summary of such an experiment. Here again, no experimental evidence regarding the HPLC analysis or method for the determination of the amount of dye and the allegedly present dehydrothio-p-toluidinesulphonic was provided.

- 3.6 Moreover, it is conspicuous that although the nitrite excess was higher in this second experiment as compared to the asserted replication of example 7 mentioned in the notice of opposition (0.120 mol instead of 0.108 mol), no nitrite excess to be destroyed was apparently found after a reaction time of 3.5 hours. This result appears to be in contradiction to the asserted replication of example 7 of document (1) and thus raises doubts as to the exact replication of example 7 of document (1). If, as alleged, excess nitrite was found and destroyed in the replication of example 7 of document (1) mentioned in the notice of opposition, it is not apparent to the Board why in the second experiment, although it was carried out with a higher amount, no such excess was found. If in the Opponent's replication of example 7 of document (1) no excess to be destroyed was present (as in the second example) at the end of the diazotization, example 7 of document (1) was not properly replicated.

In this context, it is also to be noted that the conditions of example 7 of document (1) are not necessarily fulfilled by using identical amounts of reagents, i.e. 17.5g of neopentyl glycol dinitrite according to document (1). As is shown in document (8) provided by the Appellant, alkyl nitrites upon storage are prone to decomposition (document (8), column 1,

lines 17-61). Thus, despite the use of the same amounts of nitrite as in example 7 of document (1), the active amount may not have been the same.

3.7 In the absence of sufficient evidence that example 7 of document (1) was in fact replicated, it has not been shown to the required strict standard of proof that a compound of formula (2) in the required amount is inevitably present in example 7 of document (1). Hence, the objection of lack of novelty based on this document must fail.

3.8 With regard to documents (2) to (7) provided with the notice of opposition, the Board observes that there is no explicit disclosure of the presence of a compound of formula (2) in the dye solutions disclosed in these documents. Furthermore, the diazotization in these documents is carried out with a different diazotization reagent, namely sodium nitrite, under different reaction conditions. No evidence was provided by the Opponent, who has the burden of proof, that the presently claimed aqueous dyes solutions comprising a compound according to formula (2) are the inevitable result of the diazotization disclosed in documents (2) to (7). Accordingly, the Board considers that none of the documents (2) to (7) anticipates the claimed subject-matter.

3.9 The Board, therefore, concludes that the aqueous dye solution according to the patent in suit as well as the process for its production and its use are novel within the meaning of Article 54 EPC.

4. Remittal

The Board has come to the conclusion that the subject-matter of the claims as granted is novel over the prior art. Since the Opposition Division in the decision under appeal revoked the patent solely for the reason of lack of novelty, the Board considers it appropriate to exercise its discretion according to Article 111(1) EPC and not to examine any further issues for the first time during the appeal proceedings but to remit the case to the department of first instance for further prosecution. However, in view of the withdrawal of the opposition, the Opposition Division will need to decide first whether or not to continue the opposition proceedings of its own motion according to Rule 84(2) EPC. The Board's decision to remit should not be interpreted as an indication of the conclusion to be reached in this respect.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the department of first instance for further prosecution.

The Registrar:

The Chairman:

M. Schalow

P. Ranguis