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D E C I S I O N
of 8 July 1997

Case Number: T 0472/93 - 3.3.1

Application Number: 84303542.9

Publication Number: 0127456

IPC: C09B 67/24

Language of the proceedings: EN

Title of invention:
Reactive dye composition

Patentee:
SUMITOMO CHEMICAL COMPANY, LIMITED

Opponent:
Bayer AG, Leverkusen
HOECHST Aktiengesellschaft

Headword:
Reactive dye composition/SUMITOMO

Relevant legal provisions:
EPC Art. 54(1), 56, 123(2)

Keyword:
"Inventive step - no"
"Amendment inadmissible - no clear basis in application as
filed"

Decisions cited:
-

Catchword:
-



Case Number: T 0472/93 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 8 July 1997

Appellant: SUMITOMO CHEMICAL COMPANY, LIMITED
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Representative: -

Respondent: HOECHST Aktiengesellschaft
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Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 4 March 1993
revoking European patent No. 0 127 456 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: P. Krasa
S. C. Perryman

Summary of Facts and Submissions

- I. This appeal lies from the Opposition Division's decision revoking European patent No. 0 127 456 relating to reactive dye compositions. The patent as granted contained 10 claims.
- II. Two oppositions were filed on the grounds that the subject-matter of Claim 1 of the patent was not inventive. In one opposition it was also argued that the subject-matter of Claim 10 as granted (relating to fibre materials dyed by contacting these materials with a composition of Claim 1 under conditions as specified) was not novel. The oppositions were based on a number of documents, among them:

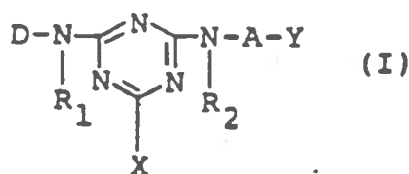
- (1) DE-A-2 417 255
- (2) DE-A-3 102 287
- (4) GB-A- 838 337
- (7) DE-A-2 207 758

The patent proprietor cited, inter alia,

- (13) Journal of the Society of Dyers and Colourists **84**, 413-422 (1968).

- III. The decision was based on an amended set of 8 claims filed on 8 October 1990. Claim 1 read as follows:

"A reactive dye composition comprising (1) a dye having the formula,



wherein D is an organic dye residue having at least one sulfo group, R_1 and R_2 are independently a hydrogen atom or an unsubstituted or substituted lower alkyl group, A is an unsubstituted or substituted phenylene or naphthylene group, X is a halogen atom and Y is a group, $-SO_2CH=CH_2$ or $-SO_2CH_2CH_2Z$, in which Z is a group capable of being split by the action of an alkali, and (2) a buffer which is inert to both the fibre-reactive groups, selected from alkali metal salts of phosphoric acids, acetic acid, boric acid, oxalic acid and phthalic acid, the amount of the buffer being from 3 through 80% by weight based on the weight of dye, and the pH value of an aqueous solution prepared from the dye composition and water of 20 times as much as the weight of the dye composition being from 4.5 to 6.5."

The Opposition Division found that the claimed subject-matter was novel, but did not involve an inventive step. It was held that document (2) represented the closest state of the art, that the patent in suit concerned the technical problem of improving storage stability of the dye composition disclosed in document (2) and that the solution of that technical problem was obvious in view of the disclosure of, inter alia, documents (1), (7), and (13).

IV. During the appeal proceedings, the Appellant (patent proprietor) introduced, inter alia, the following documents:

(16) The Chemistry of Synthetic Dyes, volume 6, page 1 (1972)

(17) EP-A-0 059 782.

V. Oral proceedings took place on 8 July 1997. At these proceedings the duly summoned Respondent I (Opponent I, Bayer AG) was not represented. He had submitted in writing that he fully concurred with the requests and submissions made by Respondent II (Opponent II, Hoechst AG).

VI. The Appellant submitted in writing and orally essentially

- that it was the object of the patent in suit to provide a **solid** dye composition with improved storage stability containing a **difunctional** reactive dye (hereinafter called **DRD**) **having both a halogen triazinyl (HT) group and a vinyl sulfonyl type (VS) group** as defined, which technical problem was not mentioned in the state of the art;
- that a person skilled in the art, knowing that stability problems of solid compositions of DRD during storage were caused by the presence of minor amounts of water in these compositions, would have considered, first of all, to use drying agents for overcoming such stability problems and would not have suggested the claimed compositions to that end;
- that document (2), which disclosed the most relevant state of the art, mentioned the possible use of a buffer only in connection with the isolation of the DRD after their synthesis and contained neither an indication of the amounts and types of buffer nor any suggestion that the pH of

the dye after dissolution in water may influence the storage stability of a solid composition comprising such DRD and that, therefore, document (2) did not suggest the claimed solution of the technical problem;

- that it was well known that dyes containing **either** HT or VS groups belonged to classes of dyes and had storage stabilities different from DRD, as further demonstrated by citation (16);
- that it was only an unproven speculation that the disclosure of document (1) relating to aqueous solutions of dyes containing **either** HT or VS groups could be applied to powdery compositions of DRD;
- that, furthermore, the Appellant had demonstrated by way of an experimental report the insufficient stability of the liquid dye composition according to example 14 of document (1), a fact that would have discouraged the skilled person from using a slightly acidic buffer as employed in the said example 14;
- that the data on dye-stability disclosed in document (13) confirmed that a reactive dye containing a HT group was less stable in the acidic pH region of the patent in suit than in the neutral pH region of about 7; and
- that, furthermore, document (17) required a pH in the range of 7 to 8 for the stabilisation of liquid dye compositions comprising a reactive dye containing a HT group, so that the prejudice existed that HT groups were not stable under slightly acidic conditions; and

- that document (7) related to a class of dyes different from that of the DRD and, therefore, would not have been considered by a skilled person searching for a solution of the technical problem.

VII. The Respondents, while no longer contesting the novelty of the claimed subject-matter, submitted in essence

- that the classification of dyes in document (16) was based on structural features and did in no way relate to storage stability properties;
- that it was not common general knowledge that the storage stability of DRD was significantly different from the storage stability of reactive dyes containing either a HT or a VS group;
- that, on the contrary, according to document (1) dyes containing a VS group and dyes containing a HT group were both stabilised in a slightly acidic pH range rendering obvious thereby the stabilisation of a difunctional reactive dye comprising both groups at a pH below 7, which conclusion was corroborated by document (13) relating to the kinetics of the hydrolysis of the HT group;
- that, furthermore, example 2b of document (2) showed that DRD and, therefore, also the HT group were sufficiently stable under slightly acidic conditions and that the disclosure of document (2) was sufficient to inform the skilled person on the type, amount and function of the buffer used;
- that, having regard to the disclosure in documents (4), (13) and (17), no prejudice existed against the stabilisation of a HT group in the acid pH region;

- that powdery compositions of DRD contained minor amounts of water and that therefore stability problems of such compositions were closely related to the storage stability properties of aqueous solutions of dyes containing HT or VS groups; and
- that the application of drying agents was not feasible in practice.

VIII. The Appellant submitted during the oral proceedings a set of eight claims (main request) and two further sets of eight claims each (first and second auxiliary request). Claim 1 of the main request was identical with that quoted in point III above, whereas in the respective claims of the auxiliary requests the pH range of from "4.5 to 6.5" was replaced by "4.5 to 6.0" (first auxiliary request) or by "4.5 to 5.5" (second auxiliary request).

He requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request or first or second auxiliary requests submitted at the oral proceedings on 8 July 1997.

- IX. The Respondents requested that the appeal be dismissed.
- X. At the end of the oral proceedings, the chairman announced the Board's decision to dismiss the appeal.

Reasons for the Decision

1. The appeal is admissible.

Main request

2. *Amendments*

The Board agrees with the finding of the decision under appeal that Claims 1 to 8 comply with the requirements of Article 123(2) and (3) EPC and, thus, are admissible. Since this is not in dispute, no comments of the Board are required.

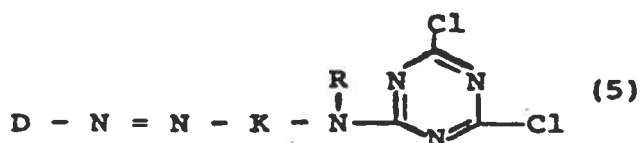
3. *Novelty*

The Board further agrees with the finding of the decision under appeal that the subject-matter of these claims is novel. Since this was no longer in dispute in the appeal proceedings, no comments of the Board are necessary on this issue either.

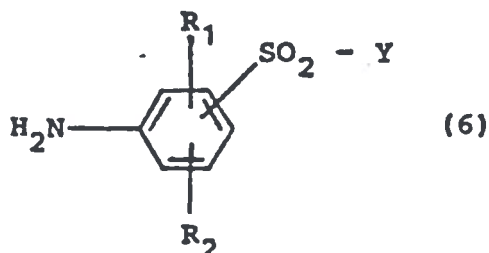
4. *Inventive step*

- 4.1 The patent in suit relates to reactive dye compositions comprising DRD and a buffer as specified in Claim 1 in amounts of from 3 to 80% by weight based on the weight of the DRD. An aqueous solution prepared from the DRD composition and an amount of water, which is 20 times as much by weight as that of the DRD, has to have a pH (hereinafter referred to as solution-pH) of from 4.5 to 6.5.

- 4.2 From document (2), which discloses the most relevant state of the art, DRD are known and in particular their manufacture, e.g. by reacting a dichlorotriazinylamino compound of formula (5)



wherein the meanings of R, D and K include those leading to compounds of the formula in present Claim 1, with an amino compound of formula (6)



wherein the meanings of R₁, R₂ and Y include those leading to compounds of the formula in present Claim 1, preferably in an aqueous medium at temperatures of from 10 to 60°C and at a pH value of from 2 to 9, in particular of from 3 to 8 (document (2), the paragraph bridging pages 11 and 12, in combination with the formulae (5) and (6) on page 9). For completing the reaction, the reaction mixture is stirred for about 10 hours at 40 to 45°C and at a pH-value between 5 and 6.5 according to example 1, and for 12 hours at 40 to 50°C at a pH-value between 4.5 and 6.5 according to example 2 (page 18, lines 26 to 29 and the sentence bridging pages 19 and 20). According to these examples the DRD are recovered from the neutral reaction solution, e.g. by spray-drying without the addition of any buffer.

However, document (2) also states that these DRD may be recovered by evaporation of the reaction solution, e.g. by spray-drying, whereby a buffer may be added to this reaction solution (page 14, lines 23 to 25).

Thus, taking into account its whole content, document (2) discloses DRD dye compositions containing a buffer.

4.3 The patent in suit aims at an improvement of the storage stability of DRD compositions (see page 2, lines 21 to 24, in combination with lines 33 to 37) for which those disclosed in document (2) are representative, as acknowledged by the parties. Thus, the technical problem underlying the patent in suit can be defined as improving the storage stability of DRD compositions known from citation (2).

4.4 No experimental data are available which directly compare the storage stability of the DRD compositions of document (2) and of the patent in suit, since the former is silent on the nature and the amounts of the buffer to be used and on the solution-pH. However, the Appellant submitted on 8 October 1990, in reply to the notices of opposition, a so-called "Comparative Experiment III". The experimental data compiled there demonstrate that DRD compositions with a solution-pH of 5 and containing 4% by weight of NaH_2PO_4 (based on the weight of the dye) as a buffer, i.e. compositions according to Claim 1 of the patent in suit, display a superior storage stability than compositions containing only 2% by weight of NaH_2PO_4 and/or a solution-pH of 2.5 or of 8. The Board accepts these data as a strong indication that the underlying technical problem is solved by the subject-matter of Claim 1.

4.5 There is no dispute that stability problems of the DRD are due to the hydrolysis of their HT and/or VS group in the presence of even small amounts of water which may be encountered as residual humidity in solid dye compositions or as atmospheric humidity (see e.g. the patent in suit, page 2 lines 5 to 7 and also document (1) schemes 1 and 2 on page 11, reactions (2) and (5), respectively). These hydrolysis reactions

are pH-dependent: According to the patent in suit, it was well known that the HT group was easily hydrolysed in an acidic region (whereas being stable under neutral or basic conditions) and that the VS group was easily hydrolysed in a basic region (whereas being stable under acidic conditions; see the patent in suit, page 2, lines 8 to 16).

- 4.6 This stability problem is addressed in document (1) relating to liquid aqueous compositions containing reactive dyes having one or more reactive groups such as, inter alia, HT and VS groups. These compositions have advantageously a pH between 5.5 and 6.5 and contain 1 to 5% by weight of a buffer substance, preferably sodium borate and sodium hydrogen phosphate. These neutral or weakly acidic, buffered dye solutions are stable for prolonged periods of time (document (1), page 1, last paragraph and page 9, last sentence; page 3, lines 1 to 8 and 25 to 26, and page 6, lines 27 to 28, in combination with page 12, lines 12 to 19). Examples 1 to 13 deal with compositions of dyes containing VS groups and having pH values ranging of from 5.9 to 6.6 (examples 4 and 13, respectively, pages 17 and 26); examples 14 and 15 deal with compositions of dyes containing a HT group. According to example 14, 5 parts by weight of Na_2HPO_4 are added to 245 parts by weight of an aqueous solution comprising 20.4% by weight of pure dye, resulting in a composition with a **stabilised pH** value of 6.1; according to example 15, 9 parts by weight of Na_2HPO_4 are added to 284 parts by weight of an aqueous solution comprising 23.3% by weight of pure dye, resulting in a composition with a **stabilised pH** value of 6.0 (pages 27 and 28).

4.7 The Appellant submitted that a person skilled in the art would have disregarded document (1) when looking for a solution of the existing technical problem, because,

- firstly, this person would have known that in reactive dyes containing **both** a HT **and** a VS group, these two groups **could** influence each other and would, therefore, have assumed that the results disclosed in document (1) could not be applied to the DRD compositions of the patent in suit,
- secondly, document (1) was concerned with aqueous solutions, which could not be compared with the solid preparations according to the patent in suit, in particular having regard to the fact that the pH conditions in a solid dye composition were unknown, and
- thirdly, the technical information contained in that document was unreliable and in contrast to other information belonging to the state of the art, such as documents (13) and (17).

These arguments are not convincing.

4.7.1 Concerning the first one, the Respondent disputed at the oral proceedings the existence of the alleged common general knowledge, and the Appellant conceded on inquiry that he could not provide any evidence supporting his allegation other than document (16), which provides the table of contents of a review article concerning reactive dyes, and shows that "dyestuffs containing several reactive groups" are dealt with in a separate chapter. However, the mere fact that certain classes of dyestuffs are treated in different chapters of a review article does not even

render it likely, let alone prove, that the reactive groups of such dyestuffs influence the reactivity of each other in the way alleged by the Appellant. Under these circumstances, the Board cannot agree with this line of argument. While it is true that in principle there may be cases where the reactivities of two reactive groups may be influenced by each other, the Board cannot find that a skilled person, in the absence of any tangible indication to such an interfering action, would have been prevented, merely by a vague theoretical possibility of failure, from reasonably expecting to succeed in solving the present technical problem by applying the teaching of document (1).

- 4.7.2 The second line of argument is not convincing either. Taking into account the undisputed fact that the stability problems of the reactive dye compositions concerned are caused by the presence of water (see point 3.6, above), it is clear that the pH (and also the amount) of the residual water, which is in contact with the solid dye, **may** have a bearing on the dye composition's storage stability. However, even if the Board would accept in the Appellant's favour that this residual water may have a different, perhaps also a somewhat lower pH than the reaction solution from which the dye is isolated, e.g. by spray-drying, it considers that a skilled person would have expected that any theoretically possible differences in the stability requirements for aqueous and for solid reactive dye compositions would only be a matter of degree and not of principle, so that he would not have been prevented from considering the technical teaching of document (1) as a promising way for improving the stability of solid dye compositions.

4.7.3 Concerning the third line of argument, the Appellant submitted that a skilled person would have been discouraged from applying a buffer under slightly acidic conditions for stabilising the reactive dye compositions concerned, since he would have found that a composition according to example 14 of document (1) had an insufficient storage stability as demonstrated by the experimental report submitted with the letter of 20 March 1992. However, this experimental report does not form part of the state of the art and, in the Board's judgment, a skilled person would only have considered verifying the technical teaching of document (1), if there were convincing reasons to consider the published technical teaching to be incorrect. In this connection the Appellant, relying, inter alia, on documents (13) and (17), submitted that a skilled person would have called in question the correctness of the finding in document (1) regarding the stability of a HT-group under slightly acidic conditions (see point 4.6 above), since this finding was contrary to an existing prejudice in the art.

4.7.4 However, the existence of such a prejudice is not in accordance with the facts of the case. Thus, document (4), relied upon by the Respondents, discloses inter alia compositions comprising HT group containing dyes and a phosphate buffer which maintains in aqueous solution a pH of not less than 6 and not more than 8 (page 1, lines 40 to 48). This pH range extends into the acidic region and overlaps with that of present Claim 1. Likewise, document (17) does not support the Appellant's submission, since it discloses a pH range of from 3 to 8.5 for HT group containing dye compositions, a range of from 7 to 8 being only **preferred** (see page 11, first paragraph in combination with the second paragraph on page 6). It is generally accepted that a so-called technical prejudice must

reflect the common opinion of the majority of those skilled in the art. The above evaluation of documents (4) and (17), however, shows that, whereas admittedly a neutral to weakly basic pH-range was preferred for the HT group, it was not the unanimous opinion of those skilled in the art that HT group containing dye compositions were instable under slightly acidic conditions.

4.7.5 The Appellant further submitted that document (13) demonstrated that a HT group was less stable in the acidic region than in the neutral pH region of about 7. Table 2 in the left hand column on page 416 lists for a number of pH-values the rate constants of the reaction of Procion Brilliant Red H-3B, a dye containing an HT group with water at 50°C (see page 413, left hand column, the first sentence of the second paragraph). The following figures are taken from this table.

pH	$k \cdot 10^4$ [min ⁻¹]
1.96	5.00±0.10
2.22	4.01±0.13
4.41	0.90±0.06
4.49	0.73±0.08
6.95	0.21±0.04
8.33	0.49±0.07
9.12	1.43±0.06
10.07	4.80±0.10
10.79	10.19±0.20

These figures show that, whereas the rate of hydrolysis runs through a broad minimum with the lowest rate at pH 6.95, the minimum is not symmetrical in respect to this lowest value. The increase of the hydrolysis rate is steeper when the pH is increased into the basic region than when the pH is lowered into the acidic

region. Therefore, the Board cannot see in this information any basis for a technical prejudice against the stability of reactive dyes at weakly acidic pH values. The fact that solutions of the dye with a 1000 fold excess of buffer were used for the measurements from which the figures of the above table resulted, is of no relevance in this connection, since, at least in the pH range of from 2 to 8, a buffer of phosphoric acid and its sodium salts was used, which is inert to the HT group (see page 414, right hand column, last paragraph and page 415, left hand column, first paragraph). In this context the Board observes that the present Claim 1 also covers rather high concentrations of buffer salts, in particular having regard to the fact that in the solid compositions only minor amounts of water are present.

- 4.7.6 Therefore, being confronted with the technical problem of improving the stability of solid DRD compositions a skilled person would not test the reliability of the technical information contained in document (1) before considering applying the technical teaching of this document with a view to solving his problem. The fact that, from a hindsight position, i.e. knowing the test results obtained after the problem was solved, the correctness of that technical teaching may be questionable, is of no relevance.
- 4.8. Thus the Board holds that a skilled person searching for a solution of the existing technical problem would have considered document (1) as a relevant and important source of information. Being aware of the respective pH dependencies of the hydrolysis of the HT and of the VS group as discussed in point 4.5 above, he would have looked for a pH range in which both groups were sufficiently stable and for measures how to maintain the pH of the DRD composition in this range. He would have learnt from document (1) that both

reactive groups are sufficiently stable at a weakly acidic pH around 6 and that this pH could be stabilised by the addition of an alkali metal salt of phosphoric acid (see point 4.6 above). By applying this teaching to that of document (2), he would have arrived at the solution of the technical problem as suggested in Claim 1, apart from the fact that the claimed compositions are characterised by their solution-pH. However, as the Appellant confirmed in the oral proceedings, the solution-pH will not differ very much from that of the buffered dye solution prior to the spray-drying step. Thus, in the Board's judgement, the solution-pH is nothing more than an indicator that sufficient amounts of buffer were incorporated into the DRD compositions to secure the maintenance of the weakly acidic environment of a pH value of about 6. To establish the appropriate buffer amounts for this purpose was, however, a mere question of routine optimisation within the ordinary skill of a skilled person.

- 4.9. The Appellant also submitted that, apart from document (7), no citation disclosed the purpose of the addition of a buffer substance to the dye compositions and, in particular, not its stabilising effect for these compositions. On the other hand, document (7) related to dye compositions different from those of the patent in suit and, so he argued, would not have been considered by a skilled person. However, this submission does not take into account that Examples 14 and 15 of document (1) refer to the **stabilised** pH of the buffered solution (see point 4.6 above). Since this citation is concerned with the stabilising of aqueous dye compositions, the purpose of stabilising the pH - and, therefore, the purpose of the addition of buffer to the respective solutions - can only be to stabilise the composition. Furthermore, document (7) relates to

water soluble azo-dyes which may contain a HT group (page 6, lines 7 to 10, in combination with the sentence bridging pages 1 and 2). These dyes may be recovered from the reaction solutions in which they were formed, e.g. by spray-drying; if desired, **stabilising agents**, e.g. mixtures of alkali metal hydrogen phosphates, may be added to the dyes during or after their recovery (page 42, paragraph 2). Therefore, while document (7) is indeed not concerned with DRD, it confirms that alkali metal hydrogen phosphates, which are uncontestedly buffers, are used as stabilising agents for dyes containing a HT group. For these reasons, the Board considers that a skilled person knew that buffers were normally used for stabilising dye compositions, among them such ones having HT groups, and would have had no difficulties in understanding that the buffers mentioned in document (2) were intended to serve that purpose.

4.10 Further, the Board is unable to agree with the Appellant's submission that, since the technical problem underlying the patent in suit was not mentioned in the state of the art, the mere identification of it required inventive skill. In the Board's judgement, a technical problem which would become obvious to a skilled person in the normal and routine use of a known chemical product, cannot normally contribute to the inventive step of the solution suggested for this problem. In the present case, the Appellant has not substantiated any reason why deficiencies in storage stability of DRD compositions would not have been encountered automatically by a skilled person as soon as he applied such a stored composition.

4.11. The Appellant's further argument that a skilled person would have tried, first of all, to solve the existing problem by removing residual water by the application

of drying agents is also not convincing. Without going into the details of possible practical problems mentioned by the Respondent, the Board finds that in the present case document (1) provided a clear and unmistakable incentive for solving the present stability problem, so that the skilled person had no reason at all to undertake experiments based on an idea on which no information at all was available in the state of the art. So much the more, as the skilled person would have been aware that drying is always an equilibrium process and that, therefore, a sufficiently complete removal of humidity could be difficult and troublesome.

- 4.12. For all the above reasons, the Board comes to the conclusion that the subject-matter of Claim 1 of the main request was rendered obvious by the combined disclosure of documents (1) and (2) and does, therefore, not involve an inventive step as required by Articles 52(1) and 56 EPC.

Auxiliary requests

5. *Admissibility of the amendments*

- 5.1 The Appellant conceded at the oral proceedings, that the feature in Claim 1 of the first auxiliary request, according to which the pH value of an aqueous solution prepared from the dye composition and water of 20 times as much as the weight of the dye composition must be from 4.5 to 6.0, was not disclosed as such in the application documents as originally filed. He argued, however, that the buffered DRD solution to be spray dried had a pH of 6.0 according example 3 of the application as filed and that the deviation of the

concentration of this solution from that required by Claim 1 as the solution-pH would be of no practical importance. He concluded that, therefore, the above-quoted feature was implicitly disclosed in the application as filed.


- 5.2 The Board cannot agree to this argument. Even if one would accept that the presence of a buffer would only allow a small shift of the pH, this could lead to a pH value outside of the range given in Claim 1 for the solution-pH. This implies that example 3 as filed does not support the suggested amendment. Already for this reason, the amendment leading to Claim 1 of the first auxiliary request is not admissible, since it violates the requirements of Article 123(2) EPC. Moreover, no evidence was submitted that the pH of 6.0 could be considered to be independent of the particular dye used in the said example 3 and could therefore be used for formulating a generic feature in Claim 1.
- 5.3 Claim 1 of the second auxiliary request, which differs from that of the first auxiliary request only by replacing the figure 6.0 by 5.5, does not meet the requirements of Article 123(2) EPC for the same reasons, mutatis mutandis, as given in point 5.2, above.
- 5.4 For these reasons the auxiliary requests must also fail.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:



E. Gorgmaier

The Chairman:



R. Spangenberg