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**Datasheet for the decision
of 2 March 2010**

Case Number: T 0879/08 - 3.3.10

Application Number: 99923094.9

Publication Number: 1077921

IPC: C07C 209/36

Language of the proceedings: EN

Title of invention:

Process for preparing 4-aminodiphenylamines

Patentee:

FLEXSYS AMERICA L.P.

Opponent:

LANXESS Deutschland GmbH

Headword:

Process for preparing 4-aminodiphenylamines/FLEXSYS

Relevant legal provisions:

EPC Art. 56

Relevant legal provisions (EPC 1973):

-

Keyword:

"Inventive step (yes) - unexpected improvement, fair comparison"

Decisions cited:

T 0099/85, T 0525/90, T 0270/90, T 0954/93, T 0355/97,
T 0506/99, T 0836/02, T 0176/04, T 0762/04

Catchword:

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Case Number: T 0879/08 - 3.3.10

D E C I S I O N
of the Technical Board of Appeal 3.3.10
of 2 March 2010

Appellant: FLEXSYS AMERICA L.P.
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Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 28 March 2008
revoking European patent No. 1077921 pursuant
to Article 101(3)(b) EPC.

Composition of the Board:

Chairman: R. Freimuth
Members: J.-C. Schmid
F. Blumer

Summary of Facts and Submissions

- I. The Appellant (Proprietor of the patent) lodged an appeal on 24 April 2008 against the decision of the Opposition Division posted on 28 March 2008 revoking European patent No. 1 077 921.
- 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) and of insufficient disclosure (Article 100(b) EPC). *Inter alia* the following documents (1), (2) and (6) were cited in the opposition proceedings.
- (1) EP-A-0 784 049,
- (2) Jerry March: "Advanced Organic Chemistry; Reactions, Mechanisms, and Structure", John Wiley & Sons, New York, USA, Third Edition, 1985, pages 906 and 907,
- (6) J. Walker and J. Johnson, J. Chem. Soc., Trans. Vol. 16, (1905), pages 955 to 961,
- III. The Opposition Division held that the European patent disclosed the invention in a manner sufficiently clear and complete for it to be carried out by a skilled person since the skilled person was able to monitor the conversion of nitrobenzene till the end of the coupling reaction, i.e. the point in time at which at most 2.0% of the nitrobenzene remains unreacted in the batch, and to control the distillation conditions as to maintain a molar ratio of water to the base in step (i) of not less than 0.6:1 at this point of time. The subject-matter of claim 1 as granted was novel with respect to

document (1), since neither a step of controlling the value of the molar ratio of water/base of not less than 0.6:1 at the end of the coupling reaction nor the specific molar ratio of water/base of 0.6:1 were disclosed in document (1). The problem to be solved with respect to that document (1) which represented the closest prior art was the provision of an improved process with regard to the hydrogenation step (ii) and to a better recovery of the base. The proposed solution was to control the molar ratio of water/TMAH to be not less than 0.6:1 at the end of the coupling reaction. However, this problem was not solved over the whole scope claimed since claim 1 encompassed the possibility of long reaction time (nitrobenzene addition plus hold time) which led to decomposition of the base. Hence, the subject-matter of claim 1 as granted and of auxiliary request 1 included non inventive embodiments and accordingly lacked an inventive step.

IV. At the oral proceedings before the Board, held on 2 March 2010, the Appellant withdrew its request for reimbursement of the appeal fee and defended the maintenance of the patent in suit in amended form on the basis of a main and an auxiliary request, thus superseding any previous requests. Claim 1 of the main request reading as follows:

"1. A process for preparing 4-aminodiphenylamine comprising:

(i) reacting aniline and nitrobenzene in the presence of water and a base wherein said base is tetramethylammonium hydroxide or carbonate salt thereof while controlling the amount of water in relation to the base so as to ensure a molar ratio of water to the

base charged in the range of not less than 4: 1 at the start of the coupling reaction and not less than 0.6: 1 at the end of the coupling reaction to produce 4-nitrodiphenylamine and/or 4-nitrosodiphenylamine and/or salts thereof, and the elapsed time from the start of nitrobenzene addition to the completion of the reaction in step (i) does not exceed 3.5 hours;

(ii) hydrogenating the reaction product of step (i) in the presence of a hydrogenation catalyst and added water so as to ensure a molar ratio of total water to base of at least 4: 1 at the end of hydrogenation;

(iii) separating the hydrogenation catalyst from the reaction mixture; and

(iv) obtaining an aqueous phase and organic phase from the reaction mixture, separating the organic phase from the aqueous phase and isolating the 4-aminodiphenylamine from the organic phase; and

(v) reusing the aqueous phase from step (iv) containing recycle base in step (i)."

V. According to the Appellant, document (1) represented the closest prior art. The technical problem to be solved was to provide an improved process of preparing 4-aminodiphenylamine, i.e. reducing tetramethylammonium hydroxide (henceforth referred to as TMAH) decomposition and improving the hydrogenation step. The problem relating to the improvement of the hydrogenation step was directly linked to the problem relating to the reduction of the decomposition of TMAH since a smooth running of the hydrogenation step was hindered by the impurities occurring from the TMAH decomposition. The improvement of the hydrogenation step was thus already achieved by reducing TMAH decomposition in step (i). The solution proposed to

this problem was to control the amount of water in the coupling reaction such as to obtain a molar ratio water:TMAH of not less than 0.6: 1 at the end of the coupling reaction and to shorten the time of step (i) to not exceed 3.5 hours. The results of annex I filed on 24 July 2008 (see below) showed a beneficial effect on the TMAH degradation by maintaining a high ratio water:TMAH during the coupling reaction and by shortening the hold time. This effect was nowhere suggested in document (1). Furthermore, the prior art did not teach that TMAH was not thermally stable under the conditions used in the process disclosed in example 1 of document (1), i.e. in solution in aniline at 80°C. The claimed subject-matter involved therefore an inventive step. The Appellant submitted in the appeal proceedings, *inter alia*, annexes I to IV to support its position.

- I "Procedure for effect of Hold Period on Base Decomposition" and results,
 - II John Tanaka et al., "Thermal decomposition of Quaternary Ammonium Hydroxides. Synthesis and Properties of Tri(²H₃-methyl)amine", J. Org. Chem, 31, (1966), pages 3431 to 3433,
 - III Musker W. K., "A Reinvestigation of the Pyrolysis of Tetramethylammonium Hydroxyde" J. Am. Chem. Soc., vol. 86, (1964), pages 960 to 961,
 - IV Musker W. K., "Nitrogen Ylides from Tetramethylammonium salts", J. Chem. Educ., vol. 45, (1968), pages 200 to 202.
- VI. The Respondent did not maintain its objection of insufficiency of disclosure and novelty in view of the amendments made to the claims. The closest prior art

document (1) disclosed all the features of claim 1 of the patent in suit, except the feature that "the elapsed time from the start of nitrobenzene addition to the completion of the first step (i) does not exceed 3.5 hours". The feature of the claimed process concerning the ratio of water to TMAH of not less than 0.6:1 at the end of the coupling reaction was not clear since the end of the coupling reaction was not defined. This feature was satisfied in the process of document (1) since the coupling reaction ended well before the hold period ended assuming that the skilled man would operate the azeotropic distillation in the coupling reaction of example 1 of document (1) similarly as in example 6 of the patent-in-suit. The burden of proof to demonstrate that this requirement was not fulfilled in the coupling reaction of document (1) was on the Appellant. The problem of providing an improved process was not solved since there were only 0.3% TMAH decomposition in the process of example 1 of document (1), while more TMAH decomposition occurred in the claimed process as shown in Appellant's annex I. Moreover, the comparison made in this annex was not fair because the process disclosed therein was operated with a much stronger azeotropic distillation than that used in the process of example 1 of document (1). The claimed threshold of 3.5 hours was arbitrary, since no technical effect was shown for this value. The proposed solution was obvious from document (1) itself which taught that too small proportions of water hindered a good recovery of the base (TMAH) and that the process was preferably to be performed in the presence of water. It was not relevant for the stability of TMAH whether it was a solid or present in solution, the crucial point was the presence or absence of water whose dipole

stabilized TMAH. The skilled person wishing to minimize TMAH decomposition and being furthermore aware from documents (2) and (6) that TMAH was thermally unstable, would shorten the reaction time of the coupling reaction while keeping water in the reaction medium, thus arriving at the proposed solution.

VII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request, or, subsidiarily, on the basis of the auxiliary request, both requests as filed during the oral proceedings before the Board.

The Respondent requested that the appeal be dismissed.

VIII. At the end of the oral proceedings the decision of the Board was given orally.

Reasons for the Decision

1. The appeal is admissible.

Main request

2. *Amendments*

Claim 1 is the combination of claims 1 and 4 as granted and is based on the combination of claims 1, 4 and 5 as originally filed. Additionally the process is restricted to the preparation of the 4-aminodiphenylamine starting from aniline and nitrobenzene according to page 8, lines 6 and 7 of the application as filed. The base used in the process of claim 1 is furthermore

restricted to tetramethylammonium hydroxide or carbonate salt thereof, according to page 8, lines 13 to 17 of the application as filed, the sentence following on lines 17 and 18 indicating furthermore the preference for that particular base. These sections on page 8 of the application as filed belong to the general description of the process of the invention and, thus , to any embodiment of that process with the consequence that the amendment made on the basis of the disclosure of that original page 8 does not add subject-matter. Claims 2 to 19 are backed up by original claims 2, 3, 6 to 15 and 17 to 22, respectively.

The requirements of Article 123(2) and (3) EC are thus satisfied, which finding was not contested by the Respondent.

3. *Sufficiency of Disclosure*

The appealed decision found the invention to be sufficiently disclosed (see point III above). Sufficiency of disclosure was no longer contested in the appeal proceedings, nor does the Board see any reason to take a different view. Hence, it is unnecessary to go into more detail in this respect.

4. *Novelty*

The Respondent did not raise any objection with regard to the novelty of the subject matter of fresh claim 1. The Board on its own does not see any reason to take a different view. Hence, it is unnecessary to go into more details in this respect.

5. *Inventive step*

According to the established jurisprudence of the Boards of Appeal it is necessary, in order to assess inventive step, to establish the closest state of the art, to determine in the light thereof the technical problem which the invention addresses and successfully solves, and to examine the obviousness of the claimed solution to this problem in view of the state of the art. This "problem-solution approach" ensures that inventive step is assessed on an objective basis and avoids an ex post facto analysis.

5.1 *Closest prior art*

5.1.1 The Board considers, in agreement with the Opposition Division and the Parties, that document (1) represents the closest state of the art, and, hence, takes it as the starting point in the assessment of inventive step.

5.1.2 Document (1) discloses a process for the production of 4-aminodiphenylamine wherein aniline is reacted with nitrobenzene in the presence of water and/or alcohols and a base, followed by the catalytic hydrogenation of the resulting nitro- and/or nitrosodiphenylamine in the presence of water. The catalytic hydrogenation of the reaction mixture is performed in the presence of 25 to 80 wt. % of water, relative to the weight of the reaction mixture from the condensation reaction. The hydrogenation catalyst is then removed from the hydrogenation mixture and the resulting organic phase is separated in order to isolate the

4-aminodiphenylamine. Finally, the aqueous phase is returned to the initial reaction mixture (see claim 1). In the process of example 1, the base is TMAH with a molar ratio of water:TMAH of approximately 4:1 at the start of the coupling reaction (see column 4, lines 11 and 12). The nitrobenzene is fed during 3 hours and there is further a hold time period of 4 hours. Thereafter the hydrogenation is carried out with a large amount of added water, i.e. the molar ratio total water to base is far above 4:1 at the end of the hydrogenation step.

5.1.3 The Parties had divergent views of the issue whether or not document (1) discloses the feature that the ratio of water to TMAH should be not less than 0.6:1 at the end of the coupling reaction.

5.1.4 According to the Respondent document (1) implicitly discloses that feature.

It is a matter of fact that this molar ratio is not explicitly disclosed in document (1). The process exemplified in example 1 of document (1) does not indicate the specific molar ratio H₂O:TMAH at the end of the coupling reaction and the missing parameters of the azeotropic distillation occurring during the coupling reaction prevents the calculation of that ratio. Hence, the Respondent's allegation that this ratio is disclosed in document (1) is based on a sequence of assumptions to fill in a gap in the disclosure of document (1), in particular that the azeotropic distillation occurs in document (1) with the same rate of water removal as in the process described in example 6 of the patent-in-suit or as in the process of

annex I also that the coupling reaction ends well before the end of the hold period of 4 hours.

Thus, assumptions must be made in order to be capable of calculating this ratio. Therefore, the Respondent when reading example 1 of document (1) made speculations with the consequence that the particular molar ratio is not necessarily satisfied in the process described in that example.

According to established jurisprudence of the Boards of Appeal a document does not disclose a specific technical feature if it does not emerge clearly and unambiguously therefrom.

In claim 1 of the patent-in-suit, the indication of the lower limit for the molar ratio water/TMAH at the end of the coupling reaction, which is lacking in document (1), amounts to the addition of fresh information not provided for the skilled person by that document (see e.g. decision T 99/85, OJ EPO 1987, page 413, point 2.2 of the reasons). Applying this principle in the present case results in the conclusion that example 1 of document (1) does not disclose clearly and unambiguously a molar ratio of water to TMAH at the end of the coupling reaction within the claimed range, with the consequence that the Board concurs with the finding of the decision under appeal in relation to the absence of any implicit disclosure of a particular molar ratio water to TMAH at the end of the coupling reaction in example 1 of document (1).

5.1.5 The Respondent further argued that in view of its submissions the burden of proof to demonstrate that

this requirement of claim 1 is not satisfied in the process of example 1 of document (1) was reversed and, hence, was on the Appellant.

According to the established jurisprudence of the Boards of Appeal, each of the parties to the proceedings carries the burden of proof for the facts it alleges. In the present case, the Respondent has not discharged its duty, merely speculating without substantiating and filing corroborating evidence for its allegation that the molar ratio water/TMAH would necessarily be not less than 0.6:1 at the end of the coupling reaction in the process of example 1 of document (1). As there is no apparent and compelling technical reason why this should be the case, and in the absence of any corroborating evidence, the Respondent has not discharged its burden of proof, with the consequence that this unsubstantiated allegation cannot to be taken up by the Board. This burden of proof applies especially when it comes to determining whether a technical feature is implicitly disclosed in a prior art document (see T 525/90, point 3.2 and 4 of the reasons; T 954/93, point 7.6 of the reasons; T 506/99, point 3.5.1 of the reasons; T 762/04, point 4.8.1 of the reasons, none of them published in OJ EPO).

- 5.1.6 The Board therefore holds that document (1) does not disclose directly and unambiguously a molar ratio water/TMAH of not less than 0.6:1 at the end of the coupling reaction.

5.2 *Technical problem underlying the patent in suit*

In view of document (1), the Appellant submitted during the oral proceedings that the technical problem underlying the patent in suit consisted in providing an improved process of preparing 4-aminodiphenylamine, i.e. reducing TMAH decomposition and improving the hydrogenation step.

5.3 *Solution*

The proposed solution to this problem is the process according to claim 1 characterized by a ratio of water to TMAH of not less than 0.6: 1 at the end of the coupling reaction and an elapsed time from the start of nitrobenzene addition to the completion of the reaction in step (i) which does not exceed 3.5 hours.

5.3.1 Although claim 1 stipulates that a ratio of water to TMAH of not less than 0.6:1 must be satisfied at the end the coupling reaction, the Parties were in dispute with respect to the moment in time during the claimed process corresponding to the said end of the coupling reaction.

5.3.2 According to a technically sensible approach for determining this moment in time, the end of the coupling reaction cannot be earlier than the moment when all reactants directly involved in the coupling reaction have been fed to the reaction vessel and cannot be later than the moment when the coupling reaction has been completed, i.e. when all of the limiting reactant has been consumed, or in other terms when 100% conversion of that reactant has been achieved.

However, the end of the coupling reaction may occur at any moment within this interval of time, since the operator of the process may well choose at whim any moment at which he wishes to end the coupling reaction.

This technical approach is supported by the patent specification on page 4, line 48 to 51 indicating that the coupling reaction may be terminated when at most 2% of the limiting agent, i.e. nitrobenzene, remains corresponding to at least 98% conversion, and that a hold period could be required after the end of nitrobenzene feed to complete the reaction and additionally to complete water removal. The fact that water removal may occur after the completion of the coupling reaction indicates that the hold time is technically to be divided into two sections, the first section corresponding to the time to complete the reaction and the second section corresponding to the time to complete water removal. Therefore the hold time may continue after 100% conversion, i.e. after the end of the coupling reaction, which finding is in line with the embodiment disclosed on page 8, lines 26 and 27 of the patent specification, which is labelled "typical procedures according to the invention for coupling and hydrogenation reactions" (page 8, line 16). Therein a distinction is made between the end of the "**reaction**" and the end of the **coupling reaction**. This typical procedure reveals that the reaction time was considered to include the feed time, a first hold time to complete the coupling reaction and a second hold time to reach a water/TMAH molar ratio of 1.0-2.5, the whole of it being the "reaction".

Accordingly, the requirement of claim 1 concerning the elapsed time from the start of the nitrobenzene addition to the completion of the reaction in step (i) has the technical meaning that the reaction time, consisting of the feed time, the hold time to complete the coupling reaction up to 100% conversion, and any additional hold time, e.g. to remove water, should not exceed 3,5 hours.

5.4 *Success*

5.4.1 According to the Appellant's uncontested submissions, the impurities resulting from the TMAH decomposition hamper a smooth running of the hydrogenation step. It follows that the partial problem of improving the hydrogenation step is directly and causally linked to the problem of reducing the decomposition of TMAH. In other words, if there is less TMAH decomposition in step (i), this would necessarily result in an improvement of the subsequent hydrogenation step, with the consequence that it is in fact the same sole problem.

5.4.2 The Appellant referred *inter alia* to Annex I in order to show that the problem underlying the patent-in-suit has been solved.

This annex shows the effect of the hold period on TMAH decomposition in the coupling reaction of aniline with nitrobenzene.

In this experiment, nitrobenzene was continuously fed during 1 hour 40 minutes to a mixture of aniline and aqueous TMAH with an initial H₂O/TMAH molar ratio of

about 4.6 to 4.7:1 obtained by water-aniline azeotropic distillation and the reaction mixture was held under stirring. The water-aniline azeotropic distillation was continued during the feed and the hold time.

Following step (i) of the claimed process, the reaction mixture was hold during 30 minutes, i.e. the elapsed time from the start of the nitrobenzene addition to the completion of the reaction was 2 hours and 10 minutes, 100% conversion of nitrobenzene was achieved, the final H₂O/TMAH molar ratio was 1.04:1 and the result was 0.52% TMAH decomposition.

To reflect the process of the closest prior art document (1), the hold time was prolonged to 4 hours, i.e. the elapsed time from the start of nitrobenzene addition to the end of the reaction was 5 hours and 40 minutes. The final H₂O/TMAH molar ratio was 0.21:1 and the result was up to 11.8% TMAH decomposition.

Hence, these results reveal that the combination of a higher molar ratio of water to TMAH at the end of the reaction with a shorter reaction time in step (i) of the claimed process leads to less decomposition of TMAH making thus credible that the combination of a content of water in relation to the TMAH of not less than 0.6:1 at the end of the coupling reaction and an elapsed time from the start of nitrobenzene addition to the completion of the reaction in step (i) not exceeding 3.5 hours reduces the TMAH decomposition, thereby improving the subsequent hydrogenation step compared with the prior art process.

Since the results of Annex I convincingly show that the technical problem is credibly solved by the claimed process, it is superfluous to address the results of example 6 of the patent specification, which moreover were contested by the Respondent.

- 5.4.3 According to the Respondent, no problem was solved with respect to document (1), since the process of example 1 of that document already achieved a decomposition of TMAH as low as 0.3%, which was much better than that obtained by the processes according to the patent-in-suit as described in annex I.

The Respondent is comparing in absolute terms the values of TMAH decomposition ever achieved in document (1) and in the patent-in-suit, i.e. a comparison to be made in order to assess whether the invention achieves any technical progress over the state of the art. However, technical progress is not a requirement for the recognition of inventive step under the EPC. In the case where comparative tests are chosen to demonstrate an inventive step with an improved effect over a claimed area, the nature of the comparison with the closest state of the art must be such that the effect is convincingly shown to have its origin in the characterizing features of the invention. In the present case the Appellant convincingly demonstrated that an improvement in relative terms is caused by the characterizing features, i.e. the combination of a higher molar ratio water to TMAH at the end of the coupling reaction with a shorter reaction time in step (i), which demonstration is sufficient to show that the problem underlying the patent-in-suit is successfully solved. Accordingly, the Respondent's argument which is

based on a comparison where more parameters than only the characterizing features have been varied, is not relevant and, hence, must be rejected.

- 5.4.4 The Respondent further alleged that the comparative experiment was not pertinent since the process reflecting the closest prior art was carried out with a stronger distillation than that used in the process of document (1).

However, this allegation is not supported by the facts, since there is no indication in document (1) how the azeotropic distillation is in fact carried out. Furthermore, the distillation step was operated in the same way in the comparative example and the example according to the invention. Notwithstanding these facts, it is sufficient to show that the reduced decomposition of the TMAH is caused by the characterising features. Any reaction conditions can be used, provided the comparative test, as in the present case, truly reflects the impact on the TMAH degradation of the technical features characterising the claimed process, namely the ratio water/TMAH of not less than 0.6:1 at the end of the coupling reaction combined with a shorter elapsed time from the start of nitrobenzene addition to the completion of the reaction.

- 5.4.5 Lastly, the Respondent argued that the claimed threshold of 3 hours 30 minutes to the completion of the reaction in step (i) was purely arbitrary, since it was in the middle of the values indicated for the processes compared in Annex I, those processes being operated with step (i) durations of 2 hours 10 minutes and 5 hours 40 minutes, respectively.

However, the comparative test in Annex I demonstrates that maintaining a certain amount of water at the end of the coupling reaction combined with a shorter time to complete the reaction in step (i) lowers the degradation of TMAH occurring in that step. On account of the nature of the invention, it is reasonable to expect that said effect progressively decreases when increasing the hold time. Hence, the Board sees no technical reasons why this effect relating to the TMAH degradation would not also be supposed to occur for any periods of time shorter than the claimed threshold of 3 hours 30, which is far below of the 7 hours for the completion of step (i) of example 1 of document (1).

According to the established jurisprudence of the Boards of Appeal, each of the parties to the proceedings carries the burden of proof for the facts it alleges. If a party, whose arguments rest on these alleged facts, does not discharge its burden of proof, this goes to the detriment of that party and such a party may not shift the onus of proof onto the other party (see T 270/90, OJ EPO 1993, 725, point 2.1 of the reasons; T 355/97, point 2.5.1 of the reasons; T 836/02, point 4.5 of the reasons; T 176/04, point 5.6.3 of the reasons; all but T 270/90 not published in OJ EPO).

The Respondent neither substantiated its allegation of non-achievement of the effect for a process having an elapsed time from the start of nitrobenzene addition to the completion of the coupling reaction below 3 hours 30 minutes as indicated in claim 1, nor filed corroborating evidence for its allegation that an increase of the duration of step (i) from the 2 hours

10 minutes used in the comparative test to 3 hours 30 minutes corresponding to the claimed threshold would necessarily destroy the effect on the TMAH degradation highlighted in the comparative test. As there is no apparent and compelling technical reason why this should be the case, and in the absence of any supporting evidence, the Respondent by merely expressing doubts has not discharged its burden of proof, with the consequence that these unsubstantiated doubts are not to be taken into account by the Board.

Hence, the Board is satisfied that the technical problem underlying the patent in suit of decreasing TMAH decomposition, and therefore also that of improving the hydrogenation step, has been successfully solved by the proposed solution, i.e. by the process according to claim 1 characterized by an amount of water in relation to TMAH of not less than 0.6: 1 at the end of the coupling reaction and an elapsed time from the start of nitrobenzene addition to the completion of the reaction in step (i) not exceeding 3.5 hours.

5.5 *Obviousness*

Finally, it remains to be decided whether or not the proposed solution to this objective technical problem is obvious in view of the cited state of the art.

- 5.5.1 During the oral proceedings before the Board, the Respondent mainly addressed document (1) in order to object to obviousness. However, although this document addresses the importance of the recovery of the base in the process, it does not tackle the problem of its

degradation let alone gives any hint on how to minimize this degradation. Hence, for this simple reason that document cannot point to the claimed solution for solving the technical problem underlying the patent in suit.

- 5.5.2 The Respondent argued that document (1) on column 1, lines 23 to 35 suggested the solution by teaching that too small proportions of water hamper a good recovery of the base.

However this passage is concerned with problems occurring during the work up of the product obtained from the coupling reaction. Thus it deals with the recovery of the base from the reaction product, but does not address the problem of reducing the decomposition of the base during the coupling reaction. There is no incentive in this section to preserve the base by maintaining a certain water content during the reaction as proposed by the patent-in-suit.

- 5.5.3 The Respondent further pointed to the passages at column 2, lines 29 to 37 and 54 to 58 of document (1) indicating that the process was preferably performed in the presence of water.

However, these sections address the presence of water at the **start** of the reaction, what is already reflected in example 1 of document (1) where the initial molar ratio water to base is about 4 to 1. Accordingly these passages do not address the proposed solution which is characterized by a ratio water to base at the **end** of the coupling reaction. Furthermore these passages also do not address the other feature characterizing the

proposed solution which indicates a maximum duration of step (i).

- 5.5.4 The Respondent also referred to column 3, lines 55 to 59 of document (1) where it is disclosed that a higher content of water provide considerably shorter reaction time and substantial yield increase.

However, this argument must be rejected since this passage exclusively addresses the hydrogenation step, i.e. step (ii), which is also already reflected in example 1 of document (1) where 12 litres of water are added before the hydrogenation, while in the present case the issue of inventive step is based on the preservation of the base during the preceding coupling reaction of step(i).

- 5.5.5 Furthermore, the Respondent referred to document (2) relating to the cleavage of quaternary ammonium hydroxides under heating and disclosing that methanol is formed on heating TMAH in water, indicating, thus, the thermal instability of TMAH, to conclude that the skilled person would have been advised against using a long reaction time in the presence of TMAH.

Document (2) is a textbook in the field of organic chemistry, i.e. is a secondary source literature, which is based on the available chemical literature. The bibliographic references of the primary sources used for the review are indicated for further reading. Those relevant primary literature sources referred to are Annexes II, III and IV.

It is disclosed in those primary sources that the decomposition of TMHA in water occurs at temperatures above 250°C, which temperatures are far higher than those at which the coupling reaction of the claimed process is carried out, namely preferably in the range of 50 to 100°C and typically 75 to 80°C (see Annex II, page 3432, right-hand column, first sentence; Annex IV, page 200, left-hand column, lines 9 to 11 and compare with the patent-in-suit, page 5, lines 3 and 4 and example 6; Annex I).

As these documents indicate that the degradation in the aqueous medium occurs at temperatures above 250° C, the skilled person would thus understand that TMAH is stable in that medium at lower temperatures, thus at the temperatures conventionally used for the coupling reaction. Accordingly the skilled person would have no reason upon reading these documents on the thermal instability of TMAH in aqueous media to shorten the reaction from 7 hours, as disclosed in example 1 of document (1), to less than 3,5 hours, as required in claim 1 of the patent-in-suit, in order to preserve TMAH from degradation.

- 5.5.6 The Respondent finally relied on page 3432, left-hand column, last sentence of Annex II and page 960, right-hand column, last paragraph of Annex III, where it is indicated that thermal decomposition of TMAH in the dry state occurs at 135 to 140°C and on document (6) disclosing that solid TMAH monohydrate (ratio water: TMAH of 1:1) decomposed at 130 to 135°C.

Those sections only address the decomposition of TMAH in solid forms. However, the stability of a compound in

its solid state is different to that in solution, since different states of matter are concerned involving different physical properties, as already apparent from the cited documents themselves which indicate different temperatures of degradation depending on whether TMAH is in the solid form or is in water, i.e. 130 to 140 °C in the solid state compared to 250°C in water (see point 5.5.5 above). Accordingly, the skilled man starting from the process of example 1 of document (1) where TMAH is present in solution in excess aniline would not have taken into consideration those sections addressing the decomposition of solid TMAH. As to the Respondent's attempt to explain the observed difference of the decomposition temperature of TMAH by alleging that it is caused by the physical property of water being a dipole, the Board considers this allegation as a mere speculation, which cannot, in the absence of any substantiating facts and corroborating evidence, be convincing.

5.5.7 It remains that document (1) alone, or in combination with any of the documents (2) or (6), lacks any hint on how to solve the problem underlying the invention, i.e. to reduce the degradation of TMAH in the coupling reaction and thereby improving the following hydrogenation step.

5.6 In respect of obviousness, the Respondent did not rely on any further documents and the Board is not aware of further documents relevant in this respect. Thus, the Board is satisfied that none of the other documents in the proceedings renders the proposed solution obvious.

Therefore, the subject-matter of claim 1, and for the same reason, that according to dependent claims 2 to 19 involve an inventive step within the meaning of Article 56 EPC.

Auxiliary request

6. Since the main request is considered to be allowable, it is not necessary to decide on the lower-ranking auxiliary request.

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 with the order to maintain the patent on the basis of the main request as filed during the oral proceedings before the Board and a description yet to be adapted.

The Registrar

The Chairman

C. Rodríguez Rodríguez

R. Freimuth