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DECISION
of 6 March 1996

Case Number: T 0845/92 - 3.3.1

Application Number: 88200018.5

Publication Number: 0276030

IPC: C07C 43/23

Language of the proceedings: EN

Title of invention:
Process for the preparation of o-isopropoxyphenol

Patentee:
ENICHEM SYNTHESIS S.p.A.

Opponent:
Bayer AG, Leverkusen Konzernverwaltung RP Patente Konzern

Headword:
Isopropoxyphenol/ENICHEM

Relevant legal provisions:
EPC Art. 56

Keyword:
"Closest state of the art"
"Technical problem"
"Inventive step (no)"

Decisions cited:
-

Catchword:

Europäisches Patentamt
Beschwerdekammern

European Patent Office
Boards of Appeal

Office européen des brevets
Chambres de recours

Case Number: T 0845/92 - 3.3.1

DECISION
of the Technical Board of Appeal 3.3.1
of 6 March 1996

Appellant: (Proprietor of the patent)	ENICHEM SYNTHESIS S.p.A. Via Ruggero Settimo 55 I-90139 Palermo (IT)
Representative:	Fusina, Gerolamo Ing. Barzanò & Zanardo Milano S.p.A. Via Borgonuovo 10 I-20121 Milano (IT)
Respondent: (Opponent)	Bayer AG, Leverkusen Konzernverwaltung RP Patente Konzern Bayerwerke D-51368 Leverkusen (DE)
Representative:	-
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted 24 July 1992 revoking European patent No. 0 276 030 pursuant to Article 102(1) EPC.**

Composition of the Board:

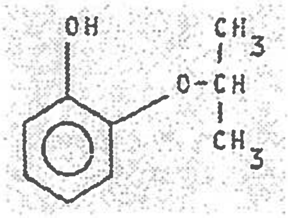
Chairman: A. J. Nuss
Members: R. K. Spangenberg
R. E. Teschemacher

Summary of Facts and Submissions

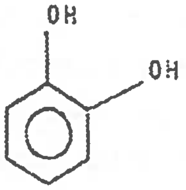
I. This appeal was filed by the patent proprietor (hereinafter the Appellant) against the decision of the Opposition Division of the EPO, dated 24 July 1992, to revoke European patent No. 0 276 030, granted in response to European patent application No. 88 200 018.5.

II. The decision under appeal was based on an amended Claim 1, submitted with letter dated 13 February 1992, reading as follows :

"1. Process for the synthesis of o-isopropoxy-phenol having formula (I):



by means of the reaction of pyrocatechol, of formula (II)



with an isopropyl halide having formula (III)

$CH_3-CHX-CH_3$

wherein X represents a halogen atom, in the presence of a solid inorganic base and of a solid-liquid phase-transfer catalyst, inside a reaction medium consisting of a mixture of two inert organic solvents,

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characterized in that the phase-transfer catalyst is a polyether selected from the polyethyleneglycols of formula HO-$(CH_2-CH_2-O)_n$-H, wherein n is an integer from 2 to 200, the crown-ethers, and the cryptands, and the two-solvent mixture consists of a high-boiling, low-polarity solvent and a lower-boiling, high-polarity solvent."

In the decision under appeal five documents were cited, including the following:

(1) ZW-46/84
(2) EP-A-151 392

The stated ground for revocation was lack of inventive step. The Opposition Division considered that it clearly appeared from the comparison between the process of the amended Claim 1 and the process known from the closest state of the art, represented by the document (1), that, insofar as the amended Claim 1 concerned a process carried out in the presence of a polyethyleneglycol as a phase-transfer catalyst, the only difference resided in the fact that the process according to the patent in suit was carried out in a "two-solvent mixture consisting of a high-boiling, low-polarity solvent and a lower-boiling, high-polarity solvent instead of either a high-boiling, low-polarity solvent or a low-boiling, high-polarity solvent". Accordingly, the problem to be solved by that process, with respect to the process disclosed in (1), was seen in providing an improved process enabling an easy recovery of the products. The solution of this problem by using a "two-solvent mixture consisting of a high-boiling, low-polarity solvent and a lower-boiling high-polarity solvent" instead of either a high-boiling, low-polarity solvent or a low-boiling high-polarity solvent was held not to involve an inventive step, inter alia having regard to the process known from document (2). In addition, the Opposition Division stated that the use of crown ethers or cryptands as phase-transfer catalysts was also obvious, since these compounds were well known phase-transfer catalysts, as was confirmed by two documents submitted by the Opponent after expiration of the opposition period and communicated to the Appellant without an invitation to present his comments.

III. In his statement of grounds of appeal, the Appellant submitted that he had not had an opportunity to comment on documents (4) and (5) cited in the decision under appeal, which was thus taken in violation of Article 113(1) EPC, and should be set aside for that reason alone.

As to the technical matter, he submitted that document (1) consistently disclosed polyethyleneglycol (PEG) as a diluent or solvent, but not as a catalyst. In particular, document (1) did not teach that the concentration of PEG had an influence on the yield or the selectivity of the process for obtaining o-isopropoxy-phenol. The claimed process, however, which aimed at high conversion and concurrently high selectivity in the production of o-isopropoxy-phenol, was, so he argued, characterised by the use of certain phase-transfer catalysts, including PEG, and a particular solvent system. In his submission, the process according to the amended Claim 1 resulted in an unexpected increase in selectivity, which showed that the claimed process was inventive. In support, he submitted a further example, wherein Example 10 of the patent in suit, which he regarded as representing the known process, was modified by adding n-decane as a co-solvent.

IV. Oral proceedings took place on 6 March 1996.

During the oral proceedings the Appellant submitted a further amended Claim 1, which differed from that underlying the decision under appeal by the limitation of the meaning of "X" to chlorine, so that the only isopropyl halide to be used as starting material was isopropyl chloride, and by deleting the crown ethers and the cryptands from the list of suitable phase-transfer catalysts, so that the only remaining phase-transfer catalysts were the polyethyleneglycols having formula $HO-(CH_2-CH_2-O)_n-H$, wherein n is an integer comprised within the range of from 2 to 200.

The Appellant requested that the decision under appeal be set aside and the patent maintained with Claim 1 submitted during the oral proceedings, and Claims 2 to 16 as granted.

The Opponent (hereinafter the Respondent) requested that the appeal be dismissed.

V. At the oral proceedings, the Appellant did not maintain his submission, relating to alternatives no longer claimed, that the decision under appeal was taken in violation of Article 113(1) EPC. Contrary to his written submissions, he argued that document (2) should be regarded as the closest state of the art, and that the basic problem arising therefrom was the need to replace the starting material isopropyl bromide by the more convenient isopropyl chloride, as set out in the patent in suit. Since the phase-transfer catalysts used according to the state of the art turned out to perform unsatisfactorily when isopropyl chloride was used as a starting material, he further argued that the real problem was to find a more suitable **catalyst**, and, since document (1) did not disclose polyethyleneglycols as catalysts, the skilled person had no reason to consider this document when looking for a solution of the said problem.

VI. The Respondent substantially argued that document (1) represented the closest state of the art, and that it implicitly taught that polyethyleneglycols were not only used as solvents, but also as catalysts. In addition, he submitted that it did not matter whether document (1) disclosed the exact function of the polyethyleneglycols, since Claim 1 of the patent in suit related to a process for obtaining o-isopropoxy-phenol and not to a use of polyethyleneglycols as phase-transfer catalysts, so that it was only relevant that this document taught the person skilled in the art that the desired product could be obtained in high yields and excellent selectivity if isopropyl chloride was used as a starting material and the reaction was performed in the presence of polyethyleneglycols and a solvent, the relative amounts of pyrocatechol and polyethyleneglycol according to the patent in suit and according to the examples of document (1) being the same. In addition, the Respondent submitted for the first time during the oral proceedings that an amendment introduced in Claim 1 as granted during the opposition proceedings contravened Article 123(2) EPC.

VII. At the end of the oral proceedings the decision to dismiss the appeal was announced.

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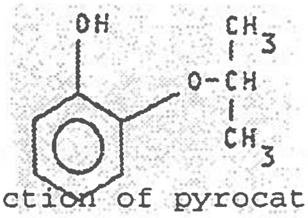
Reasons for the Decision

1. The appeal is admissible.

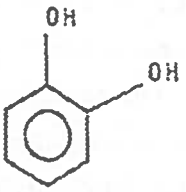
2. The Board is satisfied that the limitations of Claim 1 submitted during the oral proceedings before the Board of Appeal meet the requirements of Article 123 EPC. This was not disputed by the Respondent. It is true, however, that the patent specification states that, "the separation of unconverted pyrocatechol, and of the phase-transfer catalyst results particularly simple, if a solvent mixture is used, which is constituted by a low-polarity, high-boiling solvent, **wherein these compounds are poorly soluble at room temperature**, and by a high-polarity, lower-boiling solvent, **which makes it possible them to be dissolved during the course of the reaction**" (emphasis added by the Board), and that the emphasised functional definitions do not appear in the text of Claim 1 as amended before the Opposition Division, which only defined the two-solvent mixture by the relative polarities and boiling points of the two solvents. For this reason, the admissibility of the amendment introduced into Claim 1 during the opposition proceedings may indeed be questionable in respect of Article 123(2) EPC. However, the objection under Article 123(2) was put forward by the Respondent for the first time at the oral proceedings before the Board of Appeal, hence at a very late stage of the proceedings. Since the appeal had to be dismissed for another reason, the question of the admissibility of that amendment was irrelevant for the outcome of the present appeal proceedings. Therefore, there is neither a need to decide whether such an objection may be raised at this stage of the proceedings nor to decide on the objection in substance.

3. The **novelty** of the process as now claimed has never been disputed. Therefore, the only question that remains to be settled in this appeal is that of **inventive step**.

4. The patent in suit relates to a process for the synthesis of o-isopropoxy-phenol, having formula (I):



by means of the reaction of pyrocatechol, of formula (II),



with an isopropyl halide having formula (III)

$CH_3-CHX-CH_3$

wherein X represents a chlorine atom, in the presence of a solid inorganic base, a phase-transfer catalyst selected from the polyethylene-glycols having formula $HO(CH_2CH_2O)_n-H$, wherein n is an integer comprised within the range of from 2 to 200, and a mixture of two inert organic solvents.

5. Documents (1) and (2) likewise relate to the production of o-isopropoxy-phenol by reacting pyrocatechol with an isopropyl halide in the presence of an inorganic base, a solvent and a phase-transfer catalyst.

5.1 According to all examples contained in document (1), pyrocatechol is reacted with isopropyl **chloride** "in the presence of" a polyethyleneglycol of formula RO-(CH$_2$-CH$_2$-O)$_n$-R' (wherein R and R' represent inter alia hydrogen and n is 2 to 20) and a solvent. In the description it is stated that it is possible to use "co-catalysts", such as amines (see page 4, line 35 to page 5, line 2). However, the polyethyleneglycol is described as a "diluent", and it is stated that, in addition, any inert organic solvent may be used as "a further diluent" (page 5, lines 3 to 5). According to the examples, the reaction is carried out in the presence of a "solvent mixture" consisting of toluene and polyethyleneglycol (Examples 1 and 2), propylene glycol monomethyl ether and polyethyleneglycol (Example 3), isopropanol and polyethyleneglycol (Example 4), n-butanol and polyethyleneglycol (Example 5). The yields of 2-isopropoxyphenol (based on pyrocatechol) were 82.3%(Ex.1), 85.0% (Ex.2), 84.6% (Ex.3), 84.2% (Ex.4), and 77.9% (Ex.5), whereas the conversion of the pyrocatechol was 98.1%(Ex.1), 96.6% (Ex.2), 97.2% (Ex.3), 91.5% (Ex.4), and 97.1% (Ex.5).

5.2 The process according to document (2) is characterised in that the reaction for obtaining the o-isopropoxyphenol is conducted by treating the pyrocatechol with isopropyl halide in the presence of a phase-transfer catalyst and in the presence of one or more organic solvents. The preferred (and only specified) phase-transfer catalysts are quaternary ammonium or quaternary phosphonium salts (see Claims 1 to 3). The only isopropyl halide specifically mentioned is isopropyl bromide, which is used in all examples. The solvent may preferably be a mixture of solvents of very different polarities (page 3, lines 21 to 23 and Claim 7). The advantages of using such a solvent mixture, preferably consisting of a low-boiling alcohol and a high-boiling hydrocarbon, is described in detail in that document (see page 3, line 25 to page 4, line 3). It is, however, not stated there that the use of such solvent mixtures would give rise to a further improvement of the yield and selectivity of the described process. The conversion rates of the pyrocatechol obtained according to the process of document (2) are below 60%, whereas the yields, based on converted pyrocatechol, are about 95% (Examples 1 and 2).

6. In the Board's judgment, the closest state of the art is represented by document (1), since this is the only document describing a process for obtaining o-isopropoxy-phenol from the same starting materials as those used in the process now claimed, namely pyrocatechol and isopropyl **chloride**. By contrast, document (2) relates to a different process, in which isopropyl **bromide** is used as the starting material.

7. In respect of document (1), the Appellant had submitted in the statement of grounds of appeal that by the use of the solvent mixture according to Claim 1 the recovery of the desired product, as well as that of the catalyst and the unreacted pyrocatechol, is improved, and that, in addition, a **higher selectivity** with respect to the use of one single solvent is achieved, so that, accordingly, the technical problem in respect of the process described in document (1) should be seen in achieving **all** these improvements.

7.1 However, the technical problem can only be formulated in that way if it is credible that the above advantageous results are in fact obtained by substantially all embodiments of the process as defined in Claim 1. It is therefore not sufficient if some specific examples, employing specific reaction conditions, catalysts and

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solvents, result in, for example, a better selectivity, if these examples cannot be regarded as being representative for all embodiments covered by Claim 1. In this respect, the Opposition Division has stated in the decision under appeal that it had not been demonstrated that the higher selectivity alleged by the Appellant had been achieved by the embodiment exemplified by Example 6. During the appeal proceedings, the Appellant argued that Example 6 of the patent in suit was not a suitable basis for comparison, since one should only compare yields and selectivities obtained under comparable conditions, i.e. a process according to document (1) in which one single solvent is used with a process according to the patent in suit using the same amount of reactants and the same reaction conditions, with the only exception that the single solvent is replaced by a solvent mixture as defined in Claim 1. The Board agrees with that submission.

7.2 According to the Appellant, such a correct comparison was made in the additional example submitted on 30 June 1993. In this example two processes were compared which differed solely in respect of the solvent employed. The first process used methyl isobutyl ketone as the sole solvent, whilst the second process used instead a mixture of methyl isobutyl ketone and n-decane. An increase of selectivity from 85% to 93% in favour of the solvent mixture was observed. However, this comparison was **not** made with an **exemplified embodiment of the process of document (1), but with an example of the patent in suit** using a single solvent (see Example 10), and which happens to be covered by the generic disclosure of document (1) and is, in view of the amendment introduced into Claim 1 of the patent in suit during the opposition proceedings, no longer covered by the present Claim 1 (see points II and IV above). In these circumstances, particular care must be taken in order to make sure that the effect demonstrated by the chosen example is representative for all embodiments of the process as now claimed.

7.3 In the present case, the Board is not satisfied that this is the case, since document (1) contains examples, e.g. Example 5 (see point 5.1 above), wherein, in the presence of one single solvent, yields and selectivities are obtained which are equal or even better than those obtained with the solvent mixture according to the above comparative example. However, in the Board's judgment an improvement obtained with respect to a single solvent which is not optimal, in comparison with the specifically disclosed solvents, cannot be regarded as evidence which would allow the conclusion, on the balance of probabilities, that the replacement of a more suitable single solvent, such as isobutanol (used in Example 5 of document (1)), by a mixture of that solvent with a high-boiling, low-polarity solvent such as decane, would still further improve the very high yield and selectivity reported in Example 5 of document (1). Therefore, the Appellant has not made credible that the alleged improvement in respect of the process of document (1) has in fact been achieved by all embodiments covered by the present Claim 1.

7.4 The Board therefore eventually concurs with the Opposition Division's finding that the technical problem objectively to be solved, on the basis of which the question of inventive step has to be examined, is merely that of improving the separation of the products at the end of the reaction.

7.5 The patent in suit essentially proposes as a solution to this problem to use, instead of one single "further diluent", the blends of different-polarity solvents identified in Claim 1.

7.6 In the Board's judgment it is credible, in view of the common general knowledge and the disclosure in document (2), that the stated problem is thereby solved. Since this has never been disputed, it is not necessary to give more detailed reasons for this finding.

7.7 As set out in point 5.2 above, document (2) proposes the same solvent mixture as a solution to substantially the same technical problem, arising in a similar process for the synthesis of o-isopropoxyphenol. The generic disclosure of that document includes all usual phase-transfer catalysts and isopropyl halides. The skilled person had therefore no reason to disregard the said solvent mixture when attempting to solve the above-stated technical problem and thereby to improve the process disclosed in document (1). In particular, this person had no reason to expect that by a modification of the solvent the yield or selectivity of the process disclosed in document (1) would be significantly changed, since this document shows that high yields and selectivities were obtained with diluents of quite different polarities, such as toluene (see Example 1) or isobutanol (see Example 5). This finding is not in contradiction with the facts considered in point 7.2 above, since these facts do not form part of the state of the art.

7.8 For these reasons, the Board holds that it was obvious for a person skilled in the art to modify the process disclosed in document (1) in the way set out in the present Claim 1 in order to improve the recovery of the desired product, as well as that of the unreacted pyrocatechol and the polyethyleneglycol.

8. During the oral proceedings the Appellant insisted that the closest state of the art was represented by document (2) and that the main objective of the claimed process was to avoid the use of isopropyl bromide as a starting material, hence to find a catalyst which allowed to obtain o-isopropoxy-phenol by using, instead, the more convenient isopropyl chloride (see the patent in suit, page 2, line 58 to page 3, line 3). The Board has thus examined whether this hypothetical approach would give rise to a different conclusion in respect of the inventive step of the process according to the patent in suit.

8.1 If the Board therefore takes, for the sake of argument, document (2) as starting point for the assessment of the inventive step, then the technical problem may be seen, as stated in the patent specification (see page 3, lines 1 to 8), but excluding any element belonging to the solution, in providing a process for the manufacturing of o-isopropoxy-phenol in high yields and good selectivity, which allows the use of a more convenient while maintaining the possibility of an easy recovery of the desired product.

8.2 The patent in suit proposes to solve the above-defined problem by using isopropyl chloride as starting material and the polyethyleneglycols defined in Claim 1 as phase-transfer catalysts.

8.3 Having regard to the examples contained in the patent specification, this problem can be regarded as being thereby effectively solved.

8.4 The Appellant did not dispute that it did not require inventive skill to recognise that isopropyl chloride would in principle be an advantageous starting material, since it was more readily available and would reduce the possibility of environmental pollution. Moreover, this starting material was already employed in the process of document (1) (see point 5.1 above).

8.5 During the oral proceedings the Appellant submitted that a skilled person, looking for a solution of that technical problem, would only consider documents wherein suitable phase-transfer catalysts for the reaction of pyrocatechol and isopropyl chloride in the presence of an inorganic base are described, and would thus not consider document (1). However, the Board is unable to accept this submission. On the contrary, in the Board's judgment, and in accordance with the statement in the patent in suit, page 3, lines 4 to 7, the skilled person would have looked, with a view to solving the above-defined technical problem, for documents disclosing **reaction conditions** suitable for achieving the desired yields and selectivities, using isopropyl chloride as a starting material. Therefore, a skilled person would certainly have considered the disclosure of document (1), which, in addition to the disclosure in the examples (see point 5.1 above), generally suggests that the replacement of phase-transfer catalysts such as quarternary ammonium or phosphonium salts by the polyethyleneglycols disclosed therein would result in improved yields and good selectivities (see page 2, last paragraph in combination with page 3, first paragraph and page 4, lines 6 to 15). Thus, even if the skilled person had not known that polyethyleneglycols were in fact phase-transfer catalysts, he would nevertheless have considered the reaction conditions disclosed in document (1) as a solution of the above-defined problem. Apart from the solvent to be used as a "further diluent", these reaction conditions, including the ratio of pyrocatechol and polyethyleneglycol, are identical with those employed in the process according to the patent in suit. Since document (1) states that any organic solvent, be it high-boiling or low-boiling could be used as "further diluent", regardless of its polarity (see points 5.1 and 7.7 above), there was no need to abandon the advantageous solvent mixture taught by document (2) when replacing the quarternary ammonium salt by the polyethyleneglycol.

8.6 Consequently, also starting from document (2) the Board would not have reached the conclusion that the process of Claim 1 involved an inventive step.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

E. Görgmaier

The Chairman:

A. Nuss

BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS ★ BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE ★ CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS ★

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