Case Number: T 0299/03 - 3.3.3
Application Number: 93109168.0
Publication Number: 0575810
IPC: C08G 64/30
Language of the proceedings: EN
Title of invention: Process for producing polycarbonate
Patentee: IDEMITSU KOSAN CO., LTD.
Opponent: Teijin Limited
Bayer MaterialScience AG
Headword: -
Relevant legal provisions: EPC Art. 54, 56, 83, 84, 114(2), 123(2), 123(3) EPC R. 71(2)
Keyword: "Main request - added subject-matter (yes)"
"Auxiliary request - novelty (yes)"
"Auxiliary request - inventive step (yes)"
Decisions cited: -
Catchword: T 0201/83, T 0301/87, T 0355/99
Case Number: T 0299/03 - 3.3.3

DECISION
of the Technical Board of Appeal 3.3.3
of 17 March 2005

Appellant: Bayer MaterialScience AG
(Opponent)
Patents and Licensing
D-51368 Leverkusen (DE)

Representative: -

Respondent: IDEMITSU KOSAN CO., LTD.
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Representative: -


Composition of the Board:
Chairman: R. Young
Members: C. Idez
H. Preglau
Summary of Facts and Submissions

I. The grant of the European patent No. 0 575 810 in the name of Idemitsu Petrochemical Co., Ltd (later Idemitsu Kosan Co., Ltd) in respect of European patent application No. 93 109 168.0 filed on 8 June 1993 and claiming priority of the Japanese patent application JP 162748/92 filed on 22 June 1992 was announced on 19 August 1998 (Bulletin 1998/34) on the basis of 8 claims.

Claims 1 to 8 read as follows:

"1. A process for producing a polycarbonate which comprises transesterifying (A) dihydroxy compound and (B) carbonic diester in a reactor made of metallic material containing at least one metal selected from the group consisting of Fe, Cr, Mo, Ni and Cu, characterized in that the quantity of water in the reactor is controlled to be 500 ppm or less, and the amount of oxygen in the reactor is 10 ppm or less.

2. The process according to Claim 1 wherein the amount of residual metals in the polycarbonate obtained is (i) 10 ppm or less in total of Fe, Cr and Mo, and (ii) 50 ppm or less in total of Ni and Cu.

3. The process according to Claim 1 wherein (A) dihydroxy compound is at least one selected from the group consisting of aromatic dihydroxy compounds, aliphatic dihydroxy compounds, bisesters of aromatic dihydroxy compounds,
bisesters of aliphatic dihydroxy compounds, carbonates of aromatic dihydroxy compounds and carbonates of aliphatic dihydroxy compounds.

4. The process according to Claim 1 wherein (B) carbonic diester is at least one compound selected from the group consisting of diaryl carbonates, dialkyl carbonates and alkylaryl carbonates.

5. The process according to Claim 1 wherein the water content in the reactor is 300 ppm or less.

6. The process according to Claim 1 wherein the water content in the reactor is 100 ppm or less.

7. The process according to Claim 1 wherein an amount of oxygen in the reactor is 5 ppm or less.

8. The process according to Claim 1 wherein the transesterification is conducted under the condition that the value of X in the equation (i) is $1 \times 10^{-7}$ or less and the value of Y in the equation (ii) is $1 \times 10^{-9}$ or less:

$$X = [\text{OH}] \times t \times \exp (-6.1 \times 10^3/T)$$

wherein [OH] is the concentration of the hydroxyl group (mol/g), t is reaction time (hr), and T is reaction temperature (K),

$$Y = S \times t \times \exp (-1.5 \times 10^4/T)$$

wherein S is the contacting area (m$^{-1}$) of the metallic container per unit volume of the sample, and t and T are as defined above."
II. Notices of Opposition were filed against the patent

(i) by Teijin Ltd (Opponent I), on 17 May 1999, on the grounds of lack of inventive step (Article 100(a) EPC);

and

(ii) by Bayer AG (later Bayer MaterialScience AG) (Opponent II) on 19 May 1999, on the grounds of lack of novelty and lack of inventive step (Article 100(a) EPC), and on the ground of insufficient disclosure (Article 100(b)).

The oppositions were supported *inter alia* by the following documents:


D2: US-A-3 442 854;


D4: H. Schnell, "Chemistry and Physics of Polycarbonates"; Polymer Reviews; Vol.9, 1964, pages 44-51;


D11: US-A-3 153 008; and

D12: "High Pressure Gas Security Technology Text"; textbook in training courses of the personnel responsible for safety in the manufacture of class A chemical equipment"; First version issued 24 April 1987; revised version issued 14 April 1989.

III. By a decision announced orally on 19 November 2002 and issued in writing on 9 January 2003 the Opposition Division held that the grounds of opposition did not prejudice the maintenance of the patent in amended form.

IV. The decision of the Opposition Division was based on the following requests of the Patent Proprietor:

Claims 1 to 7 submitted with letter dated 7 January 2000 as main request;
Claims 1 to 5 submitted at the oral proceedings of 19 November 2002 as first auxiliary request; and
Claims 1 to 4 submitted as second auxiliary request at the oral proceedings of 19 November 2002.

Claim 1 of the main request was a combination of Claims 1 and 2 as granted. Dependent Claims 2 to 7 corresponded to granted Claims 3 to 8.
Claim 1 of the first auxiliary request was a combination of Claims 1, 2 and 5 as granted. Dependent Claims 2, 3, 4, and 5 corresponded to granted Claims 3, 4, 6 and 7, respectively.

Claim 1 of the second auxiliary request read as follows:

"A process for producing a polycarbonate which comprises transesterifying (A) dihydroxy compound and (B) a carbonic diester in a reactor made of metallic material containing at least one metal selected from the group consisting of Fe, Cr, Mo, Ni and Cu, the quantity of water in the reactor being controlled 102 ppm or less, the amount of oxygen in the reactor being 10 ppm or less, the amount of residual metals in the polycarbonate obtained being (i) 10 ppm or less in total of Fe, Cr and Mo, and (ii) 50 ppm or less in total of Ni and Cu."

Dependent Claims 2, 3, and 4 corresponded to granted Claims 3, 4 and 7 respectively.

According to the decision, the Opposition Division did not accept the introduction into the proceedings of several documents filed by Opponent I after the nine-month opposition period (i.e. with its letters dated 10 May 2000 and 17 August 2001), in particular that of document D14 (DE-A-2 439 552), since these documents were not more relevant than those then on file.

Concerning the main request, the Opposition Division held that Claim 7 thereof did not fulfil the requirements of Article 83 EPC.
Concerning the first auxiliary request, the Opposition Division held that it met the requirements of Article 123(2) and 123(3) EPC as well as those of Article 83 EPC. The subject-matter of the first auxiliary request was considered as novel over documents D1, D2, D3, D8, and D11 but as lacking inventive step, since no specific effect had been shown in relation to the value of the upper limit of the water content, i.e. 300 ppm.

Concerning the second auxiliary request, the decision held that Claims 1 to 4 met the requirements of Article 123(2) and 123(3) EPC. The decision stated that the subject-matter of this auxiliary request was novel, since Claim 1 thereof was more restricted than Claim 1 of the first auxiliary request.

Document D5 was considered as the closest state of the art, since it pointed out the problem of the metal of which the reactor was made and which was responsible for the colouration of the polycarbonate.

Starting from D5 the technical problem was seen in the preparation of clear and colourless polycarbonates.

According to the decision, the patent in suit had shown that there was a relationship between the yellowness of the polycarbonates and the specific amount of residual metals, and that threshold values of residual metals could be obtained by carrying out the process in the presence of 102 ppm or less water and of 10 ppm or less oxygen.
Although documents D1, D2, D6 and D7 taught to avoid the presence of water and oxygen during the preparation of polycarbonate by transesterification and although D12 disclosed that oxygen and water were responsible for rust in iron corrosion, they did not contain concrete values in order to obtain a clear and colourless polycarbonate.

Thus, the opposition division came to the conclusion that the subject-matter of the second auxiliary request was based on an inventive step.

V. A Notice of Appeal was filed on 7 March 2003 by the Appellant (Opponent II). In the Statement of Grounds of Appeal filed on 28 April 2003, the Appellant argued essentially as follows:

(i) Concerning novelty:

(i.1) The limits 102 ppm for water and 10 ppm for oxygen were arbitrary values.

(i.2) They could not justify the novelty of the claimed subject-matter. In that respect, reference was made to the Guidelines C IV 7.5.

(ii) Concerning inventive step:

(ii.1) Document D4 related to the transesterification process for the manufacture of polycarbonate. It taught to exclude oxygen in order to avoid discolouration of the polycarbonates.
(ii.2) D2 also related to the transesterification process and taught to work under the exclusion of humidity.

(ii.3) D2 or D4 could be regarded as equally qualified to be used as closest state of the art.

(ii.4) The claimed process differed from D2 or D4 in that the amount of water has been limited to 102 ppm and the amount of oxygen has been limited to 10 ppm.

(ii.5) The technical problem underlying the patent in suit was the production of polycarbonate with low yellowness i.e. with a low metal content.

(ii.6) It had not been shown that this problem was solved by the combination 102 ppm water and 10 ppm oxygen.

(ii.7) It was further evident that other parameters (catalyst and amount thereof) might influence the colouration of the polycarbonate; i.e. the technical problem was not necessarily solved exclusively by these two parameters (amount of water and amount of oxygen).

(ii.8) There was no unexpected relationship between yellowness or metal content and water and oxygen amounts.

(ii.9) In view of Comparative Example 1 and of Example 1, the reduction of metal content was proportional to the reduction of water content. Thus, no unexpected effect had been shown.
(ii.10) The Opposition Division had refused the first auxiliary request, because no effect had been shown in view of the value 300 ppm for water.

(ii.11) There was no experimental data between 590 ppm and 102 ppm. It was, hence, unclear what occurred in that range.

(ii.12) It was further evident that, beyond a specific value, the further reduction of the amount of oxygen and of water would not allow a corresponding reduction of the metal content.

(ii.13) In view of document D12 it was expected that the metal content would show an asymptotic behaviour in relation with the amount of water and oxygen.

(ii.14) With routine experiments, the skilled person would have determined the values of oxygen and water from which a further reduction of these amounts was either economically or technically meaningless.

(ii.15) No inventive step could, however, be acknowledged for such routine experiments.

VI. The arguments presented by the Respondent (Patentee) in its letter dated 16 September 2003 may be summarized as follows:

(i) Concerning novelty:

(i.1) Paragraph C IV 7.5 of the Guidelines referred to by the Appellant dealt with the problem of definition of products by parameters.
(i.2) It was therefore of no relevance in the present case.

(ii) Concerning inventive step:

(ii.1) Document D2 dealt with the problem of avoiding discolouration of polycarbonates by selecting a specific catalyst. It related to the provision of catalyst which did not lead to colouring of the polycarbonates.

(ii.2) Document D4 mentioned that exclusion of oxygen was not effective to avoid discolouration.

(ii.3) According to D4 the discolouration was related to the instability of the 4,4'-dihydroxy-diphenyl alkanes.

(ii.4) Thus, there was no reason not to accept D5 as closest state of the art.

(ii.5) The Appellant had submitted that the invention would not be working under all circumstances (e.g. type of catalyst and amounts thereof). The invention had found a different solution for improving the colouration of polycarbonates obtained by transesterification. The Examples proved that the invention was working.

(ii.6) There was no need of experimental data for the interval between 590 ppm and 102 ppm. The patent in suit contained enough examples and comparative examples.
VII. With letter dated 21 January 2005, Opponent I informed the Board that it would not attend the oral proceedings scheduled to take place on 17 March 2005.

VIII. Oral proceedings before the Board were held on 17 March 2005 in the absence of Opponent I.

Following preliminary observations by the Board concerning the allowability of Claim 1 of the main request (i.e. the set of Claims on which the Opposition Division decided that the patent could be maintained), the Respondent submitted an auxiliary request. Claim 1 of the auxiliary request reads as follows:

"A process for producing a polycarbonate which comprises transesterifying (A) dihydroxy compound and (B) a carbonic diester in a reactor made of metallic material containing at least one metal selected from the group consisting of Fe, Cr, Mo, Ni and Cu, the quantity of water in the reactor being controlled 100 ppm or less, the amount of oxygen in the reactor being 10 ppm or less, the amount of residual metals in the polycarbonate obtained being (i) 10 ppm or less in total of Fe, Cr and Mo, and (ii) 50 ppm or less in total of Ni and Cu."

Claims 2 to 4 thereof correspond to Claims 2 to 4 of the main request.

The Appellant objected to the introduction of this request at such a late stage of the proceedings.
After a short interruption of the oral proceedings, the Board informed the Parties that the auxiliary request was admitted into the proceedings.

The Appellant having indicated that it had no objection under Article 123 EPC in view of the auxiliary request and that it no longer had objections under Article 100(b) EPC, the discussion focussed on (i) the allowability of the claims of the auxiliary request under Article 84 EPC, (ii) the assessment of novelty of the subject-matter of the auxiliary request in view of documents D2, D5, and D8, and (iii) the assessment of inventive step.

(i) Concerning Article 84 EPC:

(i.1) The Appellant submitted that the indication of the oxygen content in Claim 1 was unclear, since it was not stated whether it was based on the gas phase in the reactor or on the total of the starting components.

(i.2) It further argued that the expression "100 ppm or less" in relation with the water content was unclear, since it encompassed the value 0 which could not be assessed.

(i.3) The Respondent argued that the value 0 for the water content corresponded to the amount which could not detected by analytical methods at the priority date.

(ii) Concerning novelty:

(ii.a) By the Appellant:
(ii.a.1) Example 14 of document D5 disclosed the manufacture of a polycarbonate by transesterification in a nickel lined reaction vessel. The yellowness index of the polycarbonate was between 3 and 4 according to the Gardner scale.

(ii.a.2) This value would be in the range of the yellowness indexes disclosed in the Examples of the patent in suit using a nickel lined reactor (Examples 1 to 6, 8-9).

(ii.a.3) Thus, the amount of water and oxygen in Example 14 would have inevitably been in the range claimed in Claim 1 of the auxiliary request.

(ii.a.4) The same conclusion could be drawn from Example 2 of D2 which disclosed the manufacture of a polycarbonate being almost colourless in a stainless steel reaction vessel.

(ii.a.5) Example 1 of D8 which disclosed the manufacture of a polycarbonate by transesterification under dried nitrogen would also be novelty destroying, since the starting components (bisphenol A, diphenyl carbonate) might also be present as pure substances.

(ii.b) By the Respondent:

(ii.b.1) There was no evidence that the method for determining the yellowness index in D5 (Gardner scale) was the same as the one used in the patent in suit, i.e. according to the standard JIS K7103-77. Thus, the values of yellowness indexes were not comparable.
(ii.b.2) Example 15 of D5, which was a repetition of Example 14, except that a stainless steel reaction vessel instead of a nickel lined reaction vessel was used, showed that the yellowness index was very high. Thus, it could not concluded that the amount of water and oxygen in Examples 14 and 15 would inevitably have been in the claimed range according to the patent in suit.

(ii.b.3) The indication in Example 2 of D2 that the polycarbonate obtained was almost colourless was vague. It could not, hence, be deduced that the polycarbonate had a yellowness index as those of the polycarbonate exemplified in the patent in suit.

(ii.b.4) Example 1 of D8 could not be novelty destroying, since the manufacture of the polycarbonate had taken place in a glass reactor, i.e. the reactor was not made of a metallic material as required by the patent in suit.

(iii) Concerning inventive step:

(iii.a) While essentially relying on the arguments presented in the written phase of the appeal proceedings, the Appellant made further submissions which may be summarized as follows:

(iii.a.1) D2 should be taken as the closest state of the art. It related to the manufacture of polycarbonate having a good colour. D2 taught to work under the exclusion of oxygen and water. Starting from D2, the technical problem might be seen in the provision of polycarbonate having an improved colour.
(iii.a.2) It was generally known from document D6 that the presence of water and oxygen should be avoided when producing polycarbonate by transesterification.

(iii.a.3) The graph, which the Appellant submitted at the oral proceedings, showed that there was a quite linear dependency of the Ni/Cu content of the polycarbonate according to the examples of the patent in suit on the water content. Thus, the skilled person aware of the teaching of D6 would have by routine experiments determined the level of water in order to obtain an improved colouration.

(iii.a.4) The subject-matter of the patent in suit was also obvious in view of the combination of D2 with D14. Document D14 should be introduced in the proceedings.

(iii.a.5) D5 taught to work in metallic vessel comprising metals such as nickel. The polycarbonates obtained had a low yellowness index. It would also have been obvious to carry out the process disclosed in D5 while limiting the amount of oxygen and water as taught in D2, D4, and D6.

(iii.a.6) In view of D12 it would have been obvious to limit the amount of water and oxygen in the reactor in order to reduce the corrosion thereof.

(iii.b) The Respondent, while relying on its submissions made in the written phase of the appeal proceedings, further argued essentially as follows:
(iii.b.1) Document D2 only mentioned the influence of water in relation with the thermoplastic processing of and the moisture sensitivity of the polycarbonate resin once prepared.

(iii.b.2) D4 and D5 were totally silent on the influence of water on the colouration of the polycarbonate.

IX. The Appellant requested that the decision under appeal be set aside and that the European patent No. 575 810 be revoked.

The Respondent requested that the appeal be dismissed (main request), or in the alternative that the decision under appeal be set aside and that the European patent No. 575 810 be maintained on the basis of the auxiliary request filed during the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

Procedural matters

2. As appears from the Summary of Facts and Submissions, the Board was faced with procedural issues concerning (i) the absence of Opponent I at the oral proceedings, (ii) the admissibility of the auxiliary request filed by the Respondent at the oral proceedings, and (iii) the question as to whether document D14 should be introduced into the proceedings.
2.1 As mentioned above in paragraph VII, Opponent I informed the Board with its letter dated 20 January 2005 that it would not be represented at the oral proceedings. In accordance with Rule 71(2) EPC, the proceedings were continued without it.

2.2 As indicated in paragraph VIII above, the Respondent filed an auxiliary request during the oral proceedings. As is evident from this paragraph, the filing of this set of claims has been made in response to the objections raised by the Board at the oral proceedings in connection with the question of allowability of Claim 1 of the main request under Article 123(2) EPC, so that the filing of this auxiliary request cannot amount to an abuse of procedural rights.

2.3 The Board further notes that the amendment made in Claim 1 (restriction of the upper limit of the amount of water from 102 ppm to 100 ppm) does not substantially change the factual framework of the contested decision, so that no disadvantage would be caused to the Appellant by the late filing of this auxiliary request if it was admitted into the proceedings.

2.4 Under these circumstances the Board decided to admit the auxiliary request into the proceedings.

2.5 During the oral proceedings, the Appellant wished to refer on document D14 when presenting its line of argument concerning the question of inventive step.
2.6 In this connection, the Board notes firstly (i) that document D14 had been filed after the nine-month opposition period, and secondly that the Opposition Division did not admit this late filed document into the opposition proceedings on the grounds that it was not particularly relevant either for novelty or inventive step.

2.7 The Board further observes that, in the written appeal proceedings, the Appellant neither argued that the Opposition Division had wrongly exercised its discretion when not admitting document D14 into the proceedings, nor requested that this document should be reintroduced into the proceedings, and that the Appellant never referred to this document in its written submissions concerning the question of inventive step.

2.8 Furthermore, independently of the fact that there is, in the Board's view, no justification for the very late introduction (i.e. nearly two years after the filing of the Statement of Grounds of Appeal) of this document, the Board, having considered the document D14 and the Opposition Division's decision that such document was not admissible, is satisfied that the Opposition Division did not misuse its discretion under Article 114(2) EPC when it elected to disregard document D14.

2.9 Consequently, the Board decided not to introduce document D14 into the proceedings (Art.114(2) EPC).
Main request

3. Wording of the claims

3.1 Claim 1 of the set of Claims on the basis of which the Opposition Division decided that the patent could be maintained differs from independent Claim 2 as originally filed in that it has been indicated:

(i) that the amount of residual metals in the polycarbonate obtained is 10 ppm or less in total of Fe, Cr and Mo, and 50 ppm or less in total of Ni and Cu;

(ii) that the amount of oxygen in the reactor is 10 ppm or less; and

(iii) that the water content in the reactor is 102 ppm or less instead of 500 ppm or less in original Claim 2.

3.2 Amendments (i) and (ii) are supported by original Claims 3 and 8, respectively.

3.3 Concerning amendment (iii), while the value 102 ppm for the water content is disclosed in original Examples 1, 7, and 8, this is made in association with an oxygen content of 5 ppm (Examples 1 and 7) or of 3 ppm (Example 8).

3.4 Following the principles set out in decision T 201/83 (OJ EPO 1984, 481) the amendment of the water content in Claim 1 on the basis of this specific value (i.e. 102 ppm) disclosed in the examples would be allowable under Article 123(2) EPC, provided the skilled person could have readily recognised this value as not so
closely associated with the other features of the examples as to determine the effect of that embodiment of the invention as a whole in a unique manner and to a significant degree.

3.5 In this connection the Board notes, however, that in view of the application as originally filed (page 7, lines 2 to 8), there is an interdependency between the water content and the oxygen content in order to reduce the elution of metals and the colouring of the resultant polycarbonate, so that the value 102 ppm indicated in these examples cannot be dissociated from the oxygen contents indicated therein.

3.6 It thus follows that amendment (iii) is not allowable under Article 123(2) EPC.

3.7 Consequently, since Claim 1 contravenes Article 123(2) EPC, the main request as a whole must be refused.

Auxiliary request

4. Articles 123(2) and 123(3) EPC.

4.1 Claim 1 of the main request differs from Claim 1 of the main request only in that the amount of water has been restricted to 100 ppm or less instead of 102 ppm or less.

4.2 Since the range 100 ppm or less is, in contrast to the range 102 ppm or less, supported by the application documents as originally filed (cf. original Claim 7), the Board comes to the conclusion that Claim 1 meets the requirements of Article 123(2) EPC.
4.3 The same conclusion is valid for Claims 2, 3 and 4 which find their support in original Claims 4, 5 and 9, respectively.

4.4 Since Claim 1 also corresponds to the combination of Claims 1, 2, and 6 as granted, it is evident that the requirements of Article 123(3) EPC are met by the auxiliary request.

5. Clarity

5.1 Objections of lack of clarity have been raised by the Appellant in view of the indications of the amount of oxygen (i.e. 10 ppm or less) and of the amount of water (i.e. 100 ppm or less) in Claim 1.

5.2 When amendments are made to a patent during an opposition, Article 102(3) EPC requires consideration as to whether the amendments introduce any contravention of any requirement of the Convention, including Article 84 EPC. Article 102(3) EPC, however, does not allow objections to be based upon Article 84 EPC, if such objections do not arise out of the amendments made (cf. also decision T 301/87; OJ EPO, 1990, 335; Headnote 1).

5.3 In the present case, the Board notes that the features of Claim 1 to which objections have been raised under Article 84 EPC by the Appellant (cf. points VIII.(i.1) to (i.2), above) were already present in granted Claims 1 and 6.
5.4 It thus follows that the raising of these objections under Article 84 EPC by the Appellant against Claim 1 cannot be allowed.

5.5 Since Claim 1 results from a combination of granted Claims 1, 2 and 6, and since Claims 2, 3 and 4 correspond to granted Claims 3, 4 and 7, respectively, the Board comes to the conclusion that the requirements of Article 84 EPC read in connection with Article 102(3) EPC are met by all the claims.

6. **Novelty**

Lack of novelty has been alleged by the Appellant against Claim 1 of the auxiliary request in view of Example 2 of D2, of Example 14 of D5 and of Example 1 of D8.

6.1 Document D2 relates to the preparation of high molecular weight thermoplastic polycarbonates by transesterification. According to D2, a dihydroxy compound is reacted with an organic carbonate in the presence of salts of quaternary ammonium, phosphonium or arsonium bases as transesterification catalysts. The polycarbonates obtained are said to be substantially free of discolouration and of thermal decomposition at elevated temperatures (column 2, lines 49-61).

In general, the polycarbonates can be prepared by the interaction between the reactants at a temperature of from 150°C to about 350°C or higher for times varying from 1 to 20 hours, preferably from 1 to 10 hours at atmospheric pressures, subatmospheric or superatmospheric pressures and it is preferred that
the reaction be conducted in the presence of a non-oxidizing or inert atmosphere such as, hydrogen, nitrogen, krypton, neon to prevent undesirable oxidative effects, especially where extremely high reaction temperatures are employed (column 4, lines 32-42).

6.2 More precisely, D2 discloses in its Example 2 the preparation of polycarbonate by transesterification in a stainless steel reactor. According to Example 2, 7000 parts of bisphenol A, 6700 parts of diphenyl carbonate and 0.3 part of tetraphenyl phosphonium tetraphenyl borohydride are melted under nitrogen, in a stainless steel autoclave. The resultant phenol is distilled off with stirring at pressure of 100 mm Hg at a temperature of 166°C. 5300 parts of phenol are distilled off after about 125 minutes. After 15 minutes the vacuum is increased to 0.5 mm Hg and the reaction completed at a temperature of between about 290 and 305°C. During this time, an additional 570 parts of phenol are distilled off. As indicated in Example 2, after 270 minutes, an almost colourless (emphasis by the Board) melt of polycarbonate is obtained.

6.3 Independently of the fact that the reaction is carried out under nitrogen does not preclude that some oxygen might nevertheless be present in the reactor, it is in any case evident that Example 2 does not explicitly disclose the amount of water present in the reactor during the preparation of the polycarbonate.

6.4 Nor could it be implicitly be deduced from the indication of the colouration of the polycarbonate obtained in this example that the water content was
inevitably in the range required by Claim 1 of the auxiliary request for the following reasons:

(i) The rather indefinite and unquantified indication that the obtained polycarbonate is almost colourless does not allow a comparison with the yellowness index of the polycarbonates obtained in the examples of the patent in suit determined according to the Japanese standard JIS K7103-77, so that it cannot, hence, be concluded that the polycarbonate obtained in Example 2 has a comparable yellowness and, thus, the same metal content as those obtained according to the claimed process of the patent in suit; and

(ii) even if it could, the similarity of the obtained products in terms of yellowness and metal content would not inevitably imply that the polycarbonate of Example 2 of D2 had been prepared under conditions falling under the scope of Claim 1 in terms of water content. While, on the one hand, it is normally expected that similar processes lead to similar products, it cannot, on the other hand, be reciprocally deduced that similar products have been inevitably prepared by similar processes.

6.5 Since, as shown above, there is no clear and unmistakable disclosure in Example 2 that the water content in the reactor was in the range 100 ppm or less, the subject-matter of Claim 1 and by the same token that of dependent Claims 2 to 4 must be considered as novel over Example 2 of D2 (cf. also T 355/99 of 30 July 2002; not published in OJ EPO, Reasons point 2.2.4).
Document D5 relates to the production of polycarbonates by transesterification. According to D5, undesired colour formation, that is the discolouration of the polymers produced in the transesterification process, can be reduced or inhibited when the reaction is carried out in the presence of glass or of metals selected from tantalum, nickel or chromium, so that the reaction mixture contacts only surfaces of such metals or of glass (column 1, lines 40-47).

In Example 14 of D5 aromatic polycarbonates are prepared by a continuous process as follows: Into a glass liquid reaction vessel there is introduced 1 part of bisphenol A and 1 to 1.1 parts of diphenylcarbonate and 0.000026 to 0.000037 part of alkaline metal salts as a catalyst. The mixture is allowed to react at a temperature of 160°C to 200°C at atmospheric pressure for 2-4 hours. The prepolymer reaction is completed in two subsequent nickel clad reactors with increasing temperature and vacuum. The temperature is increased to a final 240°C and vacuum is increased to 7-20 mm Hg. The total residence time in the reactors is from 2-5 hours. The final polymerization is carried out in two stainless steel wiped film reactors with a final temperature of 260 to 280°C and a vacuum of 0.2-0.5 mm Hg. A product having an intrinsic viscosity of 0.5 to 0.7 and a Gardner Yellow Index of 3 to 4 is obtained.

From the disclosure of Example 14 of D5, it is immediately evident that this example indicates neither the amount of oxygen present in the nickel clad reactors and in the stainless steel reactors nor the amount of water present in these reactors.
Furthermore, although the polycarbonates obtained in this example exhibit a Gardner Yellow index of 3 to 4, there is no evidence that the Gardner Yellow index exactly corresponds to the yellowness index determined according to Japanese standard JIS K7103-77 specified in the patent in suit, so that it cannot hence be concluded that the polycarbonates obtained in Example 14 have a comparable yellowness or, therefore, the same metal content as those obtained by the claimed process of the patent in suit.

Even if the Gardner Yellow index would exactly correspond to the one determined according to this Japanese standard, the Board further notes that the process of Example 15 of D5, which is carried out as in Example 14 except that the reactors are stainless steel reaction vessels and that the product line is constructed out of stainless steel pipe, results in a product having a similar intrinsic viscosity but a very high Gardner yellow index of 22, i.e. in the range of the comparative examples of the patent in suit. Hence, this would imply that the amount of water and/or of oxygen in Examples 14 and 15 cannot be inside the ranges defined in Claim 1 of the patent in suit.

In any case, even if one would disregard Example 15 and further consider that the polycarbonates obtained in Example 14 have a similar yellow index and hence a similar metal content as those obtained according to the claimed process of the patent in suit, it could not be reciprocally deduced, for the same reasons indicated in paragraph 6.4 above, that the process of Example 14 has inevitably been carried out in presence of an
amount of oxygen of 10 ppm or less and of an amount of water of 100 ppm or less.

6.12 Consequently, Example 14 of D5 cannot destroy the novelty of the subject-matter of Claims 1 to 4.

6.13 The reference made by the Appellant to Example 1 of D8 is even less relevant for the question of novelty. It is immediately evident that this example cannot be novelty destroying for the claimed subject-matter because the transesterification process disclosed therein is carried out in a glass reactor instead of a metallic reactor as required by Claim 1 of the patent in suit.

6.14 It thus follows from the above that the subject-matter of Claims 1 to 4 must be considered as novel over the prior art referred to by the Appellant (Article 54 EPC).

The patent in suit, the technical problem

7. The patent in suit is concerned with a process for making polycarbonate having good colour tone or transparency by transesterification in a metallic reaction vessel.

7.1 Such a process is known from document D5 which the Board, like the Opposition Division, regards as the closest state of the art.

7.2 As indicated above in paragraph 6.6, document D5 relates to the manufacture of polycarbonate by transesterification in reaction vessels made of nickel, chromium or tantalum or clad or lined with such metals.
According to D5, a disadvantage of known transesterification processes is the tendency of undesirable colour or discolouration to form in the resulting polymers but, when the reaction is carried out in reactors or systems substantially totally made of such metals to provide the contact surface or in reactors or systems clad or lined with a layer of such metals to provide the contact surface, a clear product is obtained. As further indicated in D5, when titanium, vanadium, iron, tin, lead, zirconium, stainless steel and Group IIIA metals marked discolouration of the prepolymer occurs (column 1, line 64 to column 2, line 3).

7.3 Starting from D5 the technical problem might be seen in the provision of a process for the production of polycarbonate in order to overcome the problem of discolouration linked to the elution of metals from the reaction vessels (cf. also patent in suit page 2, lines 33 to 40).

7.4 The solution proposed according to Claim 1 of the patent in suit is to carry out the transesterification in a metallic reactor comprising at least one metal selected from Fe, Cr, Mo, Ni and Cu with a water content in the reactor of 100 ppm or less and an oxygen content in the reactor of 10 ppm or less.

7.5 In view of the comparison between Examples 2 to 6 and 9 and comparative Examples 1, 5, and 7 (all using a Ni lined autoclave) which shows that a polycarbonate having a low yellowness index is indeed obtained only when the oxygen and the water contents are in the claimed ranges, the Board is satisfied that the claimed
measures provide an effective solution of the stated problem.

Inventive step

8. It remains to be decided whether the claimed subject-matter was obvious to a person skilled in the art in view of the prior art relied upon by the Appellant.

8.1 As stated in D5, the discolouration of polycarbonates made by transesterification was essentially related to the thermal instability of either partial esters of dihydric phenol still containing free hydroxyl group or to that of bisphenol A at high temperature (column 1, lines 19-37). According to D5 this discolouration is inhibited or reduced when carrying out the transesterification in presence of selective metals or glass, so that the reaction contacts only surface of such metals or glass. It is thus clear that, in D5, the problem of discolouration of the polycarbonate is not correlated with the elution of metal from the reaction vessel and it has been shown (cf. paragraph 7.5 above) that the limitation of the oxygen and water amounts in a Ni lined reactor indeed leads to a reduction of the yellow index of the obtained polycarbonate. Since document D5 is totally silent on the amounts of water and oxygen which should be present in the reactor during the transesterification, it is thus evident that D5 alone cannot suggest the solution proposed in the patent in suit.

8.2 While it is true that document D2 is concerned with a process for manufacturing polycarbonate having a good colour, it is essentially focussed on the choice of
transesterification catalysts which do not promote
discolouration of the polycarbonates even at elevated
temperatures (column 2, lines 44 to 61). While it is
further true that D2 discloses that the
transesterification should be preferably carried out
under a non-oxidizing atmosphere (column 4, lines 32 to
42) and that the water content of the polycarbonate
should be below 0.01% (column 2, lines 1 to 12), this
first point refers to the aim to avoid undesirable
oxidative effects, and the second point is made in the
context of the thermoplastic processing of the
polycarbonate. It is therefore evident that there is no
suggestion in D2 that by using a specific oxygen
content (i.e. 10 ppm or less) and a specific water
content (i.e. 100 ppm or less) in the reactor, this
would lead to a reduction of eluted metals and hence to
a reduced colouration of the obtained polycarbonates.
Thus, D2 would not provide any hint to the solution of
the technical problem.

8.3 Document D4 which relates to the preparation of
aromatic polycarbonates by transesterification merely
teaches that polycarbonates prepared from 4,4'-
dihydroxy-diphenyl alkanes might be discoloured and
insoluble if large quantities of catalyst are used, due
to the thermal instability of the 4,4'-dihydroxy-
diphenyl alkanes, even if care is taken to exclude
atmospheric oxygen (page 45, line three from the bottom
to page 46, line 5). Document D4 furthermore being
totally silent on the influence of water content in the
reaction vessel on the colouration of the prepared
polycarbonates, the Board can only come to the
conclusion that D4 is of no help for solving the
technical problem.
8.4 Document D6 is directed to a process for making aromatic polycarbonate having a desired hydroxyl-terminal ratio and number-average molecular weight in high efficiency by prepolymerizing (A) a dihydroxydiaryl alkane alone or in combination with a dihydroxydiaryl compound and (B) a diaryl carbonate until the number-average molecular weight of the prepolymer reaches 1,000-10,000, determining the number-average molecular weight, the ratio of hydroxyl terminals and that of aryl carbonate terminals to total terminal groups, adding diaryl carbonate to the system when the hydroxyl terminal ratio is higher than the desired level or adding dihydroxydiaryl compound when the ratio is lower than the desired level and continuing the polymerization reaction as to obtain the polycarbonate having the desired OH content and molecular weight. Although D6 indicates that the contamination of oxygen and water should be minimized, it neither discloses threshold values for oxygen and water nor does it make a correlation between these amounts and the colouration of the polycarbonate obtained. Consequently, D6 cannot offer to the skilled person a hint to the solution of the technical problem.

8.5 Document D12 relates to the metal corrosion of chemical equipment and presents only the general principles of wet corrosion of iron in presence of water and oxygen. It is hence clear that D12 cannot suggest the threshold values of oxygen and water content in a reaction vessel for the manufacture of polycarbonate by transesterification in order to obtain a polycarbonate having a low metal content and hence a low yellowness index.
In this connection, the further argument of the Appellant, supported, in the Appellant's view, by a graph submitted at the oral proceedings showing a linear relationship between the metal content (Ni, Cu) of the obtained polycarbonates and the water content in the reaction vessel in the examples of the patent in suit, that the skilled person would have by routine experiments determined these threshold values, cannot alter the conclusions drawn in paragraphs 8.4 and 8.5 above,

(i) firstly, since this argument would be based on knowledge which has been derived from the patent in suit and which is not derivable either from D6 or from D12,

(ii) secondly, since none of the cited documents makes a link between the amount of metal eluted and the yellowness of the obtained polycarbonates,

and (iii) thirdly, since D12 is absolutely not concerned with the wet corrosion of Ni or Cu.

Thus, in view of the above, the Board comes to the conclusion that the subject-matter of Claim 1, and by the same token that of dependent Claims 2 to 4 does not arise in an obvious manner from the prior art relied upon by the Appellant (Article 56 EPC).

In the Board's view one would also have come to the same conclusion, if, for sake of argument, one would, as successively done by the Appellant, have considered either document D2 or document D4 as the closest state
of the art and as requiring both to work under exclusion of oxygen in the transesterification reaction for obtaining almost colourless polycarbonates, since D2 and D4 are totally silent on the influence of the water amount in the reactor on the elution of metal from the reaction vessel, and since this missing element could not have been suggested, for the reasons indicated in paragraphs 8.1, 8.4, 8.5 and 8.6 above, by the documents D5, D6, and D12 used in combination with either D2 or D4.

9. Consequently the auxiliary request of the Respondent is allowable.
Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The main request of the Respondent is refused.

3. The case is remitted back to the first instance with the order to maintain the patent on the basis of Claims 1 to 4 of the auxiliary request filed during the oral proceedings and after any necessary consequential amendment of the description.

The Registrar:    The Chairman:

E. Görgmaier   R. Young