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**D E C I S I O N**  
**of 30 July 2004**

**Case Number:** T 0357/02 - 3.3.3

**Application Number:** 95914085.6

**Publication Number:** 0751965

**IPC:** C08F 10/00

**Language of the proceedings:** EN

**Title of invention:**

Process for transitioning between incompatible polymerization catalysts

**Patentee:**

ExxonMobil Chemical Patents Inc.

**Opponents:**

Basell Polyolefine GmbH  
BP Chemicals Ltd  
Mobil Oil Corporation

**Headword:**

-

**Relevant legal provisions:**

EPC Art. 54, 56

**Keyword:**

"Novelty (yes)"  
"Inventive step - problem and solution"  
"Inventive step - obvious combination of known features"

**Decisions cited:**

-

**Catchword:**

It follows from the minimalist character of a technical problem objectively arising from a closest state of the art, which can only be formulated as a modification of that state of the art, regardless of a success or failure of the measures applied, that almost any modification of the latter might be regarded as a feasible alternative by a person skilled in the relevant art, and therefore obvious, since each corresponding solution would be equally useful (or useless).

(cf. Reasons 6.2)



Case Number: T 0357/02 - 3.3.3

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.3  
of 30 July 2004

**Appellant:** Basell Polyolefine GmbH  
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**Representative:** -

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**Representative:** Smith, Julian Philip Howard  
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**Respondent:** ExxonMobil Chemical Patents Inc.  
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**Representative:** UEXKÜLL & STOLBERG  
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**Decision under appeal:** Interlocutory decision of the Opposition  
Division of the European Patent Office  
announced orally on 19 December 2001 and issued  
in writing on 4 February 2002 concerning  
maintenance of European patent No. 0751965 in  
amended form.

**Composition of the Board:**

**Chairman:** R. Young  
**Members:** A. Däweritz  
E. Dufresne

## Summary of Facts and Submissions

I. The grant of European patent No. 0 751 965 in respect of European patent application No. 95 914 085.6, based on International patent application No. PCT/US95/03348 (which had been published as WO-A-95/26370), filed on 24 March 1995 and claiming priority of 25 March 1994 of an earlier application in the USA (08/218277), was announced on 25 March 1998 (Bulletin 1998/13). The patent contained 14 claims.

Claims 1 to 5, 7, 10 and 11 as granted read, respectively, as follows:

- "1. A process for transitioning from a polymerization reaction catalyzed by a first catalyst to one catalyzed by a second catalyst comprising a metallocene catalyst, wherein said first and second catalysts are incompatible, said process comprising the steps of:
  - a) discontinuing the introduction of the first catalyst into a reactor;
  - b) introducing into the reactor an irreversible catalyst killer in an amount greater than about 1 molar equivalent based on the total gram atom metal of the first catalyst in the reactor; and
  - c) introducing the second catalyst into the reactor.
  
2. A process for transitioning from a polymerization reaction catalyzed by a first catalyst to one catalyzed by a second catalyst wherein said first

and second catalysts are incompatible, said process comprising the steps of:

- a) discontinuing the introduction of the first catalyst into a reactor;
  - b) introducing a reversible catalyst killer;
  - c) introducing an irreversible catalyst killer;  
and
  - d) introducing the second catalyst into the reactor.
3. A process for transitioning from at least two incompatible catalysts in a gas phase polymerization process occurring in at least one reactor having a fluidized bed, the process comprising the steps of:
- a) discontinuing the introduction of first catalyst;
  - b) contacting said fluidized bed with a reversible catalyst killer;
  - c) contacting said fluidized bed with an irreversible catalyst killer;
  - d) titrating said fluidized bed with an organometallic compound;
  - e) introducing a second catalyst into said reactor.
4. The process in accordance with any preceding claim wherein the first catalyst comprises a traditional Ziegler-Natta catalyst.

5. The process in accordance with claims 2, 3, or 4, wherein the second catalyst comprises a metallocene catalyst.
7. The process of claim 1 wherein the process further comprises introducing a reversible catalyst killer.
10. The process in accordance with any preceding claim wherein the process further comprises introducing an organometallic compound prior to step (c).
11. The process of claims 2 and 3 wherein steps b and c are performed outside said reactor or said steps are performed in the order a, b, c and then d."

The remaining dependent Claims 6, 8, 9 and 12 to 14 related to further elaborations of the processes as defined in independent Claims 1 to 3.

- II. On 23, 23 and 24 December 1998, respectively, three Notices of Opposition were filed in which revocation of the patent in its entirety was requested. In the Notices of Opposition of Opponents 1 and 2, the objection of lack of inventive step was raised (Article 100(a) EPC in conjunction with Article 56 EPC). According to the Notice of Opposition of Opponent 3, the claimed subject-matter was not patentable on the grounds set out in Article 100(a), (b) and (c) EPC, since it did not meet the requirements of Articles 54 (lack of novelty), 56 (lack of inventive step), 83 (insufficient disclosure) and 123(2) EPC (extension beyond the content of the application as filed).

The Oppositions relied on 6 documents including

**D1:** EP-A-0 107 105,

**D2:** EP-A-0 136 029,

**D3:** EP-A-0 471 497 and

**D5:** WO-A-92/14766.

The opposition of Opponent 3 was withdrawn by letter dated 9 March 2000.

By letter dated 18 October 2001, four sets of claims were filed by the Patent Proprietor under the common heading "Auxiliary Request" and with the further subtitles "Set A", "Set B", "Set C" and Set D", respectively. Whilst the Sets A and D were identified as concerning the first and fourth Auxiliary Requests, respectively, the rank of Sets B and C was left open.

(a) Set A differed from the set of claims as granted (section I, above) only in the wording of Claim 1 reading as follows:

"1. A process for transitioning from a polymerization reaction catalyzed by a first catalyst to one catalyzed by a second catalyst comprising a titanium, zirconium or hafnium metallocene catalyst, wherein said first and second catalysts are incompatible, said process comprising the steps of:

a) discontinuing the introduction of the first catalyst into a reactor;

- b) introducing into the reactor an irreversible catalyst killer in an amount greater than about 1 molar equivalent based on the total gram atom metal of the first catalyst in the reactor; and
- c) introducing the second catalyst into the reactor."

(b) Set B differed from the claims as granted in that the definition of the second catalyst had not only been amended in Claim 1 to require the presence of a Ti, Zr or Hf metallocene catalyst as the second catalyst as in Set A (see section II(a), above), but also in Claims 2 and 3; and Claim 5 (section I, above) had been deleted. The further dependent Claims 6 to 14 had been renumbered (Claims 5 to 13). The amendments had resulted in Claims 2 and 3 reading as follows:

"2. A process for transitioning from a polymerization reaction catalyzed by a first catalyst to one catalyzed by a second catalyst comprising a titanium, zirconium or hafnium metallocene catalyst, wherein said first and second catalysts are incompatible, said process comprising the steps of:

- a) discontinuing the introduction of the first catalyst into a reactor;
- b) introducing a reversible catalyst killer;
- c) introducing an irreversible catalyst killer; and



- d) introducing the second catalyst into the reactor.
3. A process for transitioning from at least two incompatible catalysts in a gas phase polymerization process occurring in at least one reactor having a fluidized bed, the process comprising the steps of:
- a) discontinuing the introduction of first catalyst;
  - b) contacting said fluidized bed with a reversible catalyst killer;
  - c) contacting said fluidized bed with an irreversible catalyst killer;
  - d) titrating said fluidized bed with an organometallic compound;
  - e) introducing a second catalyst comprising a titanium, zirconium or hafnium metallocene catalyst into said reactor."
- (c) Set C differed from the claims as granted only in that Claims 1 and 7 (cf. section I, above) had been deleted, and Claims 2 to 6 and 8 to 14 had been renumbered (Claims 1 to 12).
- (d) Set D differed from the claims as granted in that Claims 1, 5 and 7 (section I, above) had been deleted, the remaining Claims 2 to 4, 6 and 8 to 14 had been renumbered (Claims 1 to 11) and Claims 1 and 2 had been amended to read the same as Claims 2 and 3 of Set B (section II(b), above).

In oral proceedings before the Opposition Division held on 19 December 2001, the previous main request directed to the rejection of the oppositions was withdrawn, and the above Set A was made the Main Request.

- III. In an interlocutory decision orally announced at the end of the oral proceedings and issued in writing on 4 February 2002, the patent in suit as amended according to the Main Request (Set A; section II(a), above) was held to meet the requirements of the EPC.

In particular, the decision under appeal acknowledged novelty of the claimed subject-matter and then focused on the objection of lack of inventive step with regard to documents D1, which was acknowledged to represent the closest state of the art, and D2.

According to the decision, document D1 related to a direct conversion of a polymerisation reaction catalysed by a Ziegler-type catalyst into one catalysed by a chromium-based catalyst. Neither the condition that the catalysts had to be incompatible nor that an irreversible catalyst killer in a defined amount had to be used was suggested in D1. Further, D1 was silent about metallocene catalysts.

It was found that, in Example 3, the beneficial effect of allowing the transition to proceed with the use of a reversible and an irreversible catalyst killer had been shown, namely the effect of avoiding the formation of polymer with low melt index or high fines content, as addressed on page 3 of the description. This result was neither obvious in view of D1 nor in view of a combination of D1 and D2. The above effect was

considered to be surprising and, therefore, to support an inventive step, because "that effect could not be derived from the prior art and ... the skilled person had no incentive to consider the combination of defined catalysts and steps of the process claims as amended".

- IV. Against this interlocutory decision, Notices of Appeal were filed by Appellant 2/Opponent 2 on 3 April 2002 and by Appellant 1/Opponent 1 on 5 April 2002. The prescribed fees were paid on the same dates, respectively. The Statement of Grounds of Appeal of Appellant 2 was received on 30 May 2002, that of Appellant 1 on 10 June 2002.

In their respective Statements of Grounds of Appeal, the Appellants further pursued the requests for revocation of the patent in suit in its entirety for the ground of lack of inventive step on the basis of the four documents listed in section II, above. Appellant 1 additionally raised an objection of lack of novelty against Claim 2 of Set A (having the same wording as Claim 2 as granted; section I, above) on the basis of D1.

- V. By letter dated 18 December 2002, the Respondent disputed the arguments of the Appellants and refiled its previous requests (Sets A to D) as identified in sections II(a) to II(d), above.
- VI. Oral proceedings were held on 30 July 2004 in the absence of Appellant 2 who, by fax, received on 27 July 2004, had informed the Board that it would not attend these oral proceedings.

The arguments presented by the Appellants in writing and during the oral proceedings, respectively, and the course of the oral proceedings may be summarised as follows:

- (a) In order to clarify the way it read the claims under consideration, Appellant 1 pointed out that the reference to steps a) to d) preferably being carried out in alphabetic order (Claim 11 as granted and as contained in Set A; sections I and II(a), above) indicated that the individual steps could, according to Claims 2 and 3 of Sets A and B or Claims 1 and 2 of Sets C and D, respectively, be carried out in any order.
  
- (b) Whilst, in Set A, Claim 1 was amended by specifying the second catalyst as comprising a "titanium, zirconium or hafnium metallocene catalyst", Claims 2 and 3 remained unchanged and did not contain any definition as to the chemical composition of the catalysts involved.

On the basis of this situation, the novelty of the subject-matter of Claim 2 over D1 was disputed by Appellant 1 with regard to the use of a hydroxyl-containing compound or a mixture of such compounds in D1 for an interaction by physical or chemical means with a Ziegler-type catalyst before a chromium-based catalyst was introduced into the reactor (D1: page 2, lines 20 to 31; page 7, lines 3/4). In particular, it was argued that a clear distinction between reversible and irreversible catalyst killers (or poisons) could at most be made in the context of a specific

catalyst, but not as regards the generality of catalysts encompassed by Claim 2. Moreover, the effect of such a poison depended on its concentration and the length of contact and, moreover, poisons could be inactivated by post-treatment (cf. Claim 3, step d) of the patent in suit). Both types of poisons could, therefore, be considered as being reversible. Consequently, the distinction between these components in Claims 2 and 3 should be regarded as being irrelevant.

The hydroxyl-containing compounds such as poly(vinyl alcohol) and hydroxyethyl cellulose fulfilled, in the Appellant's view, the definitions of the irreversible catalyst killers listed in the patent in suit (page 7, line 31: alcohols, ethers).

These arguments were disputed by the Respondent with regard to the explanations of the poisons in the patent in suit (page 7). A catalyst, deactivated by an irreversible poison, could not be reactivated, since it was "dead", whilst the effect of a reversible poison could be done away with merely by re-establishing normal polymerisation conditions. Therefore, D1, which did not refer to the use of both reversible and irreversible catalyst poisons, could not anticipate Claim 2.

Moreover, D1 disclosed only a process specifically related to the transition from a Ziegler-type catalyst to a chromium-based catalyst.

- (c) The question of inventive step was then discussed initially on the basis of the wording of Claim 1 of Set A with regard to D1, accepted by all the parties as representing the closest state of the art.

Whilst the Respondent argued that a "universal process for the transition from one catalyst to another one" with minimal reactor down-time had been found which avoided the formation of fines and sheeting during the transition and thereafter, the Appellants did not see any technical effect of the claimed process which would have convincingly been shown. Claim 1 did not even exclude the emptying of the reactor. Therefore the patent in suit only aimed at a further process for transitioning from one catalyst to another one.

Whilst conceding that there was no evidence available for a further shortening of the transition process in comparison with D1, the Respondent pointed to the difference in the second (Ti, Zr or Hf metallocene) catalyst and the required minimum concentration of the irreversible catalyst killer of at least 1 mol of irreversible poison per mol of catalyst in Claim 1 (step b)), neither disclosed nor suggested by D1. Thus, it was necessary that the first catalyst could not restart the polymerisation again and that its remains left in the reactor would not affect the reaction of the second catalyst. Therefore, the second catalyst would also have some influence of the transition as a whole, due to this possible

interference between the second catalyst and the remains of the first one.

In the Appellants' view (maintained by Appellant 1 in the oral proceedings), however, the chemical composition of the second catalyst had no bearing on the solution of the technical problem underlying the patent in suit, because the success of the transition depended only on the complete killing of the first catalyst before the introduction of the second catalyst (the Statement of Grounds of Appeal of Appellant 2, page 3, last paragraph, and that of Appellant 1, respectively, page 4, second complete paragraph).

Moreover, bis(cyclopentadienyl)chromium (chromocene) was mentioned in D1 as a possible second catalyst (page 12, line 11). Hence, the skilled person knew that metallocenes could be used as the second catalyst. This statement was, however, disputed by the Respondent, because the use of chromocene belonged to a different technology, since it was used without an activator contrary to the metallocenes defined in Claim 1.

The attention of the parties was then drawn to the results in Example 4 of the patent in suit which appeared to provide evidence (normally required from the opponents/appellants) relevant for the above issues in dispute between the parties. Thus, this example referred to the criticality of a complete killing of the first catalyst *before* any addition of the second catalyst.

Therefore, the questions arose of whether the second catalyst played any significant role in the process, in particular in view of the facts that (i) the total amount of the irreversible killer, the water, used in Example 4 of the patent in suit amounted to a molar ratio of more than 1:1, and that (ii) the example did not provide the desired results despite the use of a Zr metallocene as defined in Claim 1.

In view of the results in Example 4 of the patent in suit having been discussed in detail, it was no longer disputed by the Respondent that the second catalyst apparently did not play a significant role in the success or failure of the transition.

- (d) Consequently, the situation did not appear to be different with regard to Set B, and consent was given by the Respondent to continue the discussion with Set C and then Set D.
  
- (e) As regards Claim 1 of Set C, which directly corresponded to Claim 2 as granted (section I, above) and to Claim 2 of Set A, Appellant 1 pointed out that the scope of this claim was even broader as regards the catalysts and the amount of the irreversible catalyst killer than Claim 1 (of Set A) hitherto discussed. Since Example 4 of the patent in suit fulfilled also the definitions in steps b) and c) of Claim 1 of Set C, the technical problem to be solved could only be seen in the provision of an alternative method to that of D1, irrespective of the results achieved by the alternative method.



According to Appellant 1, the measures of step b) in Claim 1 of Set C (additional to those steps discussed in relation to Set A, above) were rendered obvious by D2 which referred to the addition of a reversible catalyst poison.

Then, D5 was discussed, which referred to various combinations of catalyst poisons (page 11). Moreover, the document referred to the previously known combination of carbon dioxide and alcohol which could be used as catalyst poisons for Ziegler-Natta catalysts (page 1).

With regard to the latter two documents, the Respondent argued that they did not refer to transition processes but only to methods for stopping a catalysed polymerisation.

- (f) As regards Set D, the Respondent additionally argued that none of the cited documents gave any suggestion to use the specific second catalyst.
- (g) After the closure of the debate, the final requests of the Parties were established. Set B was withdrawn by the Respondent.

VII. The Appellants requested that the decision under appeal be set aside and the patent be revoked.

The Respondent requested that the appeals be dismissed and that the patent be maintained or, in the alternative, that the decision under appeal be set aside and that the patent be maintained on the basis of

auxiliary requests sets C or D, filed with letter dated 18 December 2002.

### **Reasons for the Decision**

1. The appeals are admissible.
2. Since all parties had been duly summoned, the oral proceedings were held in the absence of Appellant 2 in accordance with Rule 71(2) EPC.

#### *Main Request*

3. *Article 123(2) and (3) EPC*

Claim 1 differs from Claim 1 as granted and Claim 1 as published in WO-A-95/26370 by the further definition of the second catalyst, which finds its basis in Claim 1 in conjunction with Claim 5 and page 7, lines 20/21 of the WO-A-publication as filed. This amendment, furthermore, does not extend beyond the scope of the original version of this definition.

The remaining claims correspond to their initial versions.

Consequently, the requirements of Article 123(2) and (3) EPC are met by the claims of Set A.

4. *Novelty*
  - 4.1 Novelty of the subject-matter of Claim 2 over D1 was disputed by Appellant 1 on the basis of the argument

that a clear distinction between the reversible and irreversible catalyst killers was not possible. Therefore, the differentiation between these catalyst poisons in this claim should be ignored. Moreover, the claim was not limited to specific catalyst systems, thus, including chromium-based catalysts such as chromocene (sections VI(b) and VI(c), paragraph 5, above).

- 4.2 The starting point of document D1 has been the fact that Ziegler-type catalysts act as catalyst poisons for chromium-based catalysts and, therefore, have to be removed completely from the reaction zone before polymerisation can be started using a chromium-type catalyst (page 1, lines 18 to 24). For the purpose of allowing a direct transition without any need for emptying and re-charging the polymerisation reactor in shorter periods of time than hitherto necessary (page 2, lines 20 to 31), D1 discloses a process for converting a continuous olefin polymerisation reaction catalysed by a Ziegler-type catalyst into one catalysed by a chromium-based catalyst. This known process of D1 comprises the following steps (a) to (d).

In step (a), the introduction of the components of the Ziegler catalyst system into the polymerisation reactor is discontinued.

In step (b), polymerisation conditions are maintained therein and polymerisation is permitted to continue for a time, in order to allow the components of the Ziegler catalyst system present in the reactor to consume themselves in the production of additional polymer.

In step (c), a hydroxyl-containing compound or a mixture of such compounds, which are capable of reacting with and/or adsorbing the components of the Ziegler catalyst system, is introduced into the reactor. This compound or mixture is then circulated in the reactor for a time and in an amount sufficient for the compound(s) to react with and/or adsorb *all* the catalyst components. In order to ensure removal of all unconsumed catalyst components, an excess of the stoichiometric amount required for this purpose should be employed (eg 150 to 600%). Moreover, during this time, the polymerisation conditions may be adjusted to those employed with the chromium-based catalyst.

In step (d), the chromium-based catalyst is fed to the reactor at a rate of from 25 to 100% of the rate normally used during polymerisation, under reaction conditions suitable for this catalyst, and these reaction conditions are maintained until polymerisation with this catalyst has initiated (D1: Claim 1; page 7, lines 3/4; page 8, lines 5 to 23; page 8, line 29 to page 9, line 7; page 10, lines 7 to 19 and 30 to 33).

Whilst mentioning a number of hydroxyl-containing compounds which can be used individually or in admixture with one another, such as poly(vinyl alcohol), hydroxyethyl cellulose, silica, alumina, thoria and zirconia, D1 is silent with respect to a reversible or irreversible character of these compounds when used in its process.

- 4.3 In Examples 3 to 5 of the patent in suit (page 13, below Table 4; page 15, below Table 6; page 16, below Table 7), steps (a) and (b) of D1 relating to the

reduction of the activity of the first catalyst were also carried out. Then, however, otherwise than D1, the patent in suit refers to the addition of both "reversible" and "irreversible" catalyst killers. Reversible catalyst killers "initially inhibit catalyst activity and polymerisation for a period of time, but do not irreversibly deactivate the catalyst so that after a period of time under normal polymerisation conditions the catalysts reactivate and polymerisation will continue". On the other hand, irreversible catalyst killers "irreversibly inactivate a catalyst's ability to polymerize olefins" (patent in suit: page 7, lines 24 to 27 and 29/30).

Although the term "period of time", as used twice in the above explanation of the reversible killer, is not explicitly defined in the patent in suit (in terms of minutes, hours or days, as argued by Appellant 1 in the oral proceedings), the explanations of the two types of catalyst poisons, quoted above, clearly state that a reversible poison does not "irreversibly de- (or in)-activate the catalyst", whereas the irreversible poison does, irrespective of any time limits.

- 4.4 Moreover, these explanations show that it is not the specific compound which plays the decisive role, but that a certain functionality (as a reversible or irreversible catalyst poison) has to be fulfilled by the compound chosen. The person skilled in the art is, however, familiar with such a situation, wherein a certain functionality is required or has to be avoided at a certain time, as eg shown in D1 (page 1, lines 18 to 27): In the presence of ethylene, the components of a Ziegler-type catalyst promote, in polymerisation

conditions, the polymerisation of the monomer. However, when a chromium-based catalyst is introduced directly in the polymerisation reactor in their presence, they act as catalyst poisons preventing the chromium-based catalyst from promoting polymerisation even if the latter is used in large excess.

Other documents demonstrating the familiarity of the skilled person with such situations are D2 (in particular on page 6, last paragraph and page 7, first paragraph, also referring to "reversible" and "irreversible" catalyst poisons) and D3 (indicating in column 1, line 52 to column 2, line 3, that CO<sub>2</sub> is not an effective poison for chromium oxide-based catalysts).

It follows that the functional definition of a compound as being a reversible or irreversible catalyst killer defines the required interaction between the compound chosen and the catalyst system present rather than the compound *per se*.

Therefore, the Board is of the opinion that, for a given catalytic system which, according to the claims, can, however, be chosen freely, the characterisation of a catalyst poison as a "reversible catalyst killer" or "irreversible catalyst killer" will in each concretely chosen relevant case be unambiguously and, hence, clearly defined. It can, therefore, serve to distinguish between the different catalyst killer components to be used in steps b) and c) of Claim 2.

Since D1 is silent with respect to a characterisation of the hydroxy-containing compounds as "reversible" or "irreversible" catalyst killers (section 4.3, above),

Claim 2 of the patent in suit, however, unambiguously requires the introduction of both a reversible and an irreversible catalyst killer in steps b) and c), respectively, of Claim 2, the Board has come to the conclusion that the subject-matter of Claim 2 is novel with regard to D1.

4.5 No further novelty objections have been raised. Nor does the Board see any basis for such an objection. Claim 1 of the patent in suit requires a minimum amount of the irreversible killer and requires a Ti, Zr or Hf metallocene to be used as a second catalyst; Claim 3 also requires the introduction of both a reversible and an irreversible killer.

4.6 Consequently, the subject-matter according to the Main Request is novel.

5. *Problem and solution*

5.1 The patent in suit relates to a process for transitioning between incompatible polymerisation catalysts by a) discontinuing the introduction of the first catalyst into the polymerisation reactor and b) introducing a compound to completely and irreversibly deactivate those parts of this catalyst not yet used up in the polymerisation reaction before c) the feeding of the second catalyst to the reactor is started.

5.2 Such a process is known from D1 which, in the decision under appeal, was regarded as the closest state of the art, as agreed to by all the parties. The Board has no reason to take a different position.

As mentioned above (section 4.2, *supra*), D1 discloses the direct transition from a polymerisation using a first, Ziegler-type, catalyst to one using a second, chromium-based, catalyst (incompatible with the Ziegler-type catalyst), without the need for emptying and re-charging the polymerisation reactor. The solution found in D1 is based on the introduction of a hydroxyl-containing compound or a mixture of such compounds which are allowed to react with and/or to adsorb all the remaining unconsumed components of the first catalyst before the introduction of the second catalyst into the reaction mixture.

- 5.3 According to the introduction of its description, the patent in suit also aims at a minimal reactor down-time. Further objects of the patent in suit are to avoid the production of very high molecular weight product and to avoid the occurring of fines which can induce operability problems in the reactor such as fouling and sheeting (page 3, lines 19/20 and 40 to 42).

Hence, on this basis, the technical problem underlying the patent in suit might be seen in the definition of a transition process between any polymerisation catalysts, incompatible with one another, thereby avoiding the formation of very high molecular weight polymer and preventing operability problems due to the occurrence of sheeting and fouling, whilst keeping the reactor shut-down period at a minimum.

- 5.4 According to the patent in suit, this problem is solved by means of three different combinations of process features as defined in independent Claims 1, 2 or 3 (section II(a) in conjunction with section I, above).



- 5.4.1 Each of these independent claims relates to a process for transitioning from a polymerisation reaction using a first catalyst to a polymerisation reaction by means of a second catalyst, whereby the two catalysts (or catalyst systems, both terms are used interchangeably; patent in suit: page 3, line 23) are incompatible with each other (page 3, line 55 to page 4, line 2).
- 5.4.2 In all three independent claims, the transition comprises several steps: The first and last steps, common to all of these claims, relate to the discontinuation of the introduction of the first catalyst and to the introduction of the second catalyst into the polymerisation reactor, respectively. Whilst, in Claim 1, the addition of an irreversible catalyst killer (or poison) is the only intermediate measure taken, in each of Claims 2 and 3, both a reversible and an irreversible catalyst killer, respectively, are added.

Moreover, Claim 1 requires the (co-)use of a specific type of second catalyst (ie Ti, Zr or Hf metallocene catalysts) and a minimum amount of irreversible catalyst killer, whereas neither of Claims 2 and 3 contains any such requirement.

- 5.4.3 In view of the wording "the process comprising the steps ..." and "said process comprising the steps ...", respectively, in each of the independent Claims 1 to 3, the Board concurs with the opinion expressed by Appellant 1 (section VI(a), above) that none of these claims excludes any further steps in addition to those explicitly defined therein, such as the introduction of

further reactants. Thus, reference can be made to the further addition of a reversible killer and/or of an organometallic compound in Claim 1 (as specified in Claims 7 and 10, respectively, both being appendant to Claim 1).

Nor is the way, in which each of the process steps is to be carried out, limited in the independent claims. Thus, the wordings of these claims equally include the incremental and/or repeated addition of the catalyst killer(s) and/or different orders of the individual process steps (otherwise the second embodiment of Claim 11 concerning Claims 2 and 3 would be redundant).

Furthermore, whilst Claims 2 and 3 are completely silent with respect to the chemical nature of the second catalyst, Claim 1 only requires that the second catalyst *comprises* a Ti, Zr or Hf metallocene. Moreover, Claims 2 and 3 are also silent with respect to minimum amounts of the catalyst killers.

5.5 Since Appellant 1 disputed that the above technical problem (section 5.3, above) was indeed solved by the patent in suit, it is necessary to consider the examples in the patent in suit for the answer to this issue.

5.5.1 The patent in suit contains six examples, whereby Example 2 is to show the detrimental effect caused by the omission of any deactivation of the first catalyst before the second catalyst is added.

5.5.2 Whilst Example 3, to which reference was made in the decision under appeal, shows the success of one

embodiment claimed, Example 4 demonstrates that - undisputed between the parties and as confirmed on page 14, lines 50/51 of the patent in suit - the complete killing of the first catalyst is crucial to the success of the transition from the first to the second catalyst.

In both these examples, gas phase co-polymerisations of ethylene in fluid bed reactors were carried out using the same catalysts; ie, at first, a conventional Ziegler-Natta catalyst and, thereafter, a bis-(methyl-n-butyl-cyclopentadienyl) zirconium dichloride catalyst.

- 5.5.3 After having copolymerised ethylene and hexene at steady state using the Ziegler-Natta catalyst, the transition was started in Example 4 by reducing the concentration of triethyl aluminium. After nine hours, the catalyst feed was stopped and carbon monoxide (CO) was injected as a reversible catalyst poison. Once the reaction was killed, 20 g of water as the irreversible killer were injected into the cycle gas below the distributor plate and allowed to circle for 30 min. This resulted in 100 ppm by weight of water in the reactor, a concentration corresponding to a molar ratio of water to the Ziegler-Natta active catalyst components including aluminium alkyl of 0.9:1.

A series of blow-downs were taken to adjust the reaction conditions to those required in the second polymerisation reaction. Immediately upon the introduction of ethylene to the reactor a slight reaction was noticed which increased upon the further addition of hydrogen and of hexene. This increase was noted in *the absence of any metallocene catalyst*

addition, ie it could only be caused by residual active Ziegler-Natta catalyst. At the same time, the melt index had dropped, indicating a rise of the molecular weight. Then, the reaction was killed with further CO and a second water injection of 21 g to complete the inactivation of the first catalyst.

Thereafter, the concentrations of the reactants were re-established and the addition of the metallocene catalyst was started. However, shortly thereafter, the reactor had to be shut down due to the formation of reactor sheets (patent in suit: page 15, line 33 to 56).

5.5.4 As conceded in the patent in suit (page 14, lines 50/51 and page 15, lines 52 to 54), this example demonstrates the failure of the transition due to too low an amount of irreversible killer having been added, allowing the Ziegler-Natta catalyst to reactivate and resulting in high fines and ultra high molecular weight polymer.

5.5.5 Whilst it is evident that the reversible killer (CO) had stopped the first polymerisation reaction, the first amount of water added (resulting in a water to catalyst molar ratio of 0.9:1, ie lower than the molar ratio of 1 as required in Claim 1) was insufficient.

However, even the second injection of another 21 g of water, which raised the water/catalyst ratio to a total of more than 1.8:1 (ie exceeding the above minimum amount required in Claim 1) and killed the first catalyst completely, did not avert the failure of the experiment. Nor was this failure prevented by the use of the preferred metallocene catalyst (page 5, lines 16/17) as required in Claim 1. Nevertheless,

ultra high molecular weight polymer and high fines were produced and sheeting occurred (page 14, line 51 and page 15, line 51).

It follows from Example 4 that despite the use of the second catalyst and of an amount of irreversible catalyst killer, both as required in Claim 1 (but not in Claims 2 and 3), the above technical problem was not solved and that, therefore, the objection of Appellant 1 referred to in section 5.5, above, was well founded.

5.5.6 In other words, although Example 4 clearly fulfilled all the requirements of Claim 1 (steps a) to c)), those of Claim 2 (steps a) to d)) and those of Claim 7 appendant to Claim 1, respectively (cf. section 5.4.3, above, referring to the formulations used in the independent claims), it nevertheless resulted in a total failure to accomplish a transition from a polymerisation using a preferred first (Ziegler-Natta) catalyst to a polymerisation by means of a preferred second (Zr metallocene) catalyst (cf. Claims 1, 4 and 5; section I, above) and equally in a failure to avoid ultra high molecular weight polymer, fines and sheeting, as mentioned in section 5.3, above.

Thus, this example, far from demonstrating any superiority in the claimed process, rather demonstrates that each of the above independent claims of the Main Request includes embodiments which do not solve the technical problem as defined in section 5.3, above. In other words, the measures applied according to these claims do not solve the problem over the whole range claimed.

Apart from these facts and findings, the Respondent conceded in the oral proceedings that no evidence was available which would indicate any improvement in respect of the speed of the claimed processes over those according to the closest state of the art.

- 5.6 Therefore, the technical problem objectively arising from the closest state of the art must be worded in a less ambitious manner, and can only be seen in the provision of an alternative method for the transition between two processes using two different catalysts, incompatible with each other, or, more particularly, in the modification of the transition process of D1, regardless of whether it avoids ultra high molecular weight polymer, fines and sheeting, ie irrespective of a success or failure of the measures carried out.

The latter formulation of the technical problem was not disputed by the Respondent.

6. *Inventive step*

It remains to be decided whether the solution found was obvious to a person skilled in the art having regard to the state of the art relied upon by the Appellants.

- 6.1 In view of the overlap of the hydroxyl-containing compound used in D1 and the definition of the irreversible catalyst killer in the patent in suit (page 7, lines 29 to 34, in particular alcohols and ethers) and in view of the fact that D1 recommends that "an excess of the stoichiometric amount (of the hydroxyl-containing compound) required for this purpose

should be employed in order to ensure complete removal of all unconsumed components of such (ie the first) catalyst" (D1: page 8, line 29 to page 9, line 3), the solution found and claimed in Claim 1 differs from D1 only in that a Ti, Zr or Hf metallocene is used as the second catalyst.

6.2 It follows from the minimalist character of the technical problem objectively arising from D1 as established by the Board (section 5.6, above), namely, a modification of the transition process of D1, regardless of whether it avoids ultra high molecular weight product, fines or sheeting, ie irrespective of a success or failure of the measures applied, that almost any modification of the latter process might be regarded as a feasible alternative by the person skilled in the relevant art, and therefore obvious, since each corresponding solution would be equally useful (or useless).

6.3 In the Board's view, merely using a Ti, Zr or Hf metallocene as the second catalyst, amounts to no more than such a modification, which must therefore be regarded as an obvious measure to solve the stated problem. This view is confirmed, in particular, by the acknowledgement of prior art in the patent in suit itself, page 2, lines 34 to 36 (the application as filed: WO-A-95/26370, page 2, lines 8 to 11) and by D5 (the paragraph bridging pages 2/3), according to which it had been known that metallocenes could successfully be used as catalysts in the production of polymers.

Consequently, the subject-matter of Claim 1 is obvious in view of D1.

- 6.4 Since a decision can only be made on a request as a whole, the Main Request cannot be successful due to the obviousness of the subject-matter of Claim 1.

*First Auxiliary Request (Set C)*

7. Since, apart from the renumbering, the wording of claims 1 to 12 of this Auxiliary Request corresponds to that of Claims 2 to 6 and 8 to 14 as granted (section II(c), above), ie the amendment resides in the deletion of one originally claimed process, the requirements of Article 123(2) and (3) EPC are met.

8. *Novelty*

Independent Claim 1 of this set also corresponds to Claim 2 of Set A (cf. section II(a), above). Since the difference in comparison to D1 lies in the use of both a reversible and an irreversible catalyst killer, the findings concerning the novelty of Claim 2 of Set A are also valid for Claim 1 of this request (cf. sections 4 to 4.6, above). Consequently, the subject-matter of Claim 1 is novel.

9. *Problem and solution*

As already shown in section 5.5.6, above, Claim 2 of Set A encompasses embodiments which do not solve the technical problem derived from the description of the patent in suit (section 5.3, above), therefore, requiring the formulation of a less ambitious technical problem (section 5.6, above). Due to the identity of the process features in that Claim 2 and in Claim 1 of



Set C, these findings are also valid for the latter request.

Consequently, the relevant technical problem is the same as defined in section 5.6, above.

10. *Inventive step*

It remains to be decided whether the solution found was obvious to a skilled person vis-à-vis the cited documents.

10.1 Claim 1 of this auxiliary request requires the introduction of both a reversible and an irreversible catalyst killer.

10.1.1 Whilst disclosing the possibility of introducing a mixture of hydroxyl-containing compounds as catalyst killers, document D1 is silent about any combination of reversible and irreversible catalyst killers. Nor does it provide an incentive to use a mixture of such killers.

The question to be decided is, therefore, whether the use of such a combination amounts to a modification which might be regarded as a feasible alternative by the skilled person in the sense of section 6.2, above.

10.1.2 Document D5 discloses a continuous process for preparing olefinic polymers by means of a metallocene catalyst system wherein a volatile catalyst killer component and a non-volatile catalyst system killer component are "added downstream of the polymerisation zone to act respectively, to suppress polymerisation in

the recycled monomer and the separated molten polymer" recovered from the reactor effluent (Claim 1).

Water, carbon monoxide, carbon dioxide and alcohols are examples for volatile catalyst killer components to be used in its own process (D5: page 11).

Apart from the particulars of its own process, the document refers, however, also to a number of patent documents from which low molecular weight gases and liquids were previously known to kill Ziegler-Natta catalyst systems and, thus, to stop the polymerisation catalysed by those catalysts.

In this context, mention is made of "EP 116917" which "for example describes a killer of CO<sub>2</sub> and alcohols. These products are said to react with catalyst to form non-volatile compounds, not active in polymerization" (D5: page 1, paragraph 2 of the Background of the invention). This document is also referred to in the patent in suit (page 2, lines 26/27: "For example, EP-A-116,917 describes using carbon dioxide and alcohol as Ziegler-Natta catalyst killers") and in the application as filed (WO-A-95/26370: page 1, lines 32 to 34).

Hence, the person skilled in this art had been aware of the teaching of the document referred to in D5 that a catalyst, when it was a Ziegler-Natta catalyst (as preferred in the patent in suit: Claim 4; page 2, lines 5 to 8; page 4, lines 3/4) could efficiently be killed by using combinations of CO<sub>2</sub> and alcohols.

Carbon dioxide is, however, one example for a reversible catalyst killer, and alcohols are referred

to as irreversible catalyst killers in the patent in suit (page 7, lines 22/23 and 30/31).

- 10.1.3 Consequently, the use of a combination of CO<sub>2</sub> and alcohol, known from the document mentioned in D5, in the process of D1 in order to completely kill the first catalyst, before the second catalyst is added, had been a known technical option for a person skilled in this art.

Therefore, the subject-matter of Claim 1 derives in an obvious way from the state of the art.

- 10.2 For the same reason as addressed in section 6.4, above, the first Auxiliary Request cannot, therefore, be successful due to the obviousness of the subject-matter of Claim 1.

*Second Auxiliary request (Set D)*

11. The claims according to the second Auxiliary Request (Set D) differ from those of as granted by the deletion of Claims 1, 5 and 7 as granted and by a reference to the presence of Ti, Zr or Hf metallocene catalysts in the definition of the second catalyst in renumbered Claims 1 and 2 of this request. The deletions concern one of the alternatives of the claimed process and the removal of redundant features from the dependent claims. As regards the modification of the definition of the second catalyst, the findings concerning Article 123(2) and (3) EPC in the above section 3 are also valid for the claims according to this Auxiliary Request.

Likewise, the findings in the above sections 4 to 4.6 concerning novelty of Claim 2 of Set A are also valid for Claim 1 of this Auxiliary Request due to the mandatory use of reversible and irreversible catalyst killers. Additionally, Claim 1 differs from D1 in that D1 does not refer to the presence of Ti, Zr or Hf metallocene catalysts in the second catalyst.

Consequently, the subject-matter of this Auxiliary Request (Set D) fulfils the requirements of Articles 54, 123(2) and 123(3) EPC.

12. *Problem and solution*

This second Auxiliary Request differs from the first Auxiliary Request (Set C) only in that both independent claims contain a reference to the presence of a Ti, Zr or Hf metallocene in the second catalyst. As, however, demonstrated by Example 4 of the patent in suit (sections 5.5.2 to 5.5.6, above), the presence of a Zr metallocene did not prevent the failure of the transition from the first to the second catalyst, and the technical problem as defined in section 5.3, above, was not solved despite the use of such a second (preferred) metallocene catalyst. Consequently, the relevant technical problem to be solved has again to be formulated in a less ambitious manner in accordance with section 5.6, above.

13. *Inventive step*

It remains to be decided whether the solution of this problem, as claimed, derives in an obvious way from cited documents.

On the one hand, having regard to the choice of the second catalyst, the findings in sections 6.2 and 6.3, above, are also applicable and valid with regard to Claim 1 of this request, ie, merely using a Ti, Zr or Hf metallocene as the second catalyst, amounts to no more than a modification which may be regarded as a feasible alternative by the person skilled in the relevant art and must, therefore, be regarded as an obvious measure to solve the stated problem.

The other relevant difference of the process of present Claim 1 (Set D) from that of D1 resides in the introduction of both a reversible and an irreversible catalyst killer, before the feed of the second catalyst is commenced. Therefore the findings concerning the assessment of whether Claim 1 (Set C) of the first Auxiliary Request involves an inventive step (sections 10 to 10.2, above) are also valid for Claim 1 of this second Auxiliary Request.

Consequently, the subject-matter of Claim 1 of the second Auxiliary Request (Set D) derives in an obvious way from the state of the art.

14. Since a decision can only be made on a request as a whole, the second Auxiliary Request cannot be successful due to the obviousness of the subject-matter of Claim 1.
  
15. Since all the valid requests on file fail due to the obviousness of the subject-matter claimed, the patent in suit must be revoked.

**Order**

**For these reasons it is decided that:**

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar:

The Chairman:

E. Görgmaier

R. Young