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DECISION
of 8 March 2006

Case Number: T 1236/03 - 3.3.03

Application Number: 95907421.2

Publication Number: 0739365

IPC: C08F 10/00

Language of the proceedings: EN

Title of invention:
Alumoxanes and catalysts comprising alumoxanes

Patentee:
ExxonMobil Chemical Patents Inc

Opponents:
BP Chemicals Ltd (OI)
Basell Polyolefine GmbH (OIII)

Former Opponents:
Basell Polypropylen GmbH (OII)
Borealis Technology Oy (OIV)

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56, 83, 100
RPBA Art. 10a, 10b

Keyword:
"Novelty: yes"
"Inventive step - problem invention: yes"
"Sufficiency - no undue burden"

Decisions cited:
T 0793/93, T 0002/83

Catchword:
-

Case Number: T 1236/03 - 3.3.03

DECISION
of the Technical Board of Appeal 3.3.03
of 8 March 2006

Appellants:
(Opponent I)

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(Opponent III)

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Respondent:
(Patent Proprietor)

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Representative:

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Decision under appeal:

Interlocutory decision of the Opposition Division of the European Patent Office posted 8 October 2003 concerning maintenance of European patent No. 0739365 in amended form.

Composition of the Board:

Chairman: R. Young
Members: M. Gordon
E. Dufrasne

Summary of Facts and Submissions

I. Mention of the grant of European Patent No. 0 739 365 in the name of Exxon Chemical Patents Inc., later ExxonMobil Chemical Patents Inc. in respect of European patent application No. 95 907 421.2, filed on 10 January 1995 as international application No. PCT/US95/00472 and claiming priority of US patent application no. 180 171 dated 11 January 1994, published as WO95/18836 on 13 July 1995, was announced on 8 December 1999 (Bulletin 1999/49) on the basis of 10 claims which read as follows:

"1. A composition comprising alumoxane and porous support wherein the ratio of (1) the ratio of aluminum to the support element outside the support to (2) the ratio of aluminum to support element inside the support is 2.0 or less.

2. A composition comprising porous support material and alumoxane-transition metal complex, wherein the ratio of (1) the ratio of aluminum to the support element outside the support to (2) the ratio of aluminum to support element inside the support is 2.0 or less.

3. A composition prepared by combining porous support material and the reaction product of alumoxane and a transition metal compound wherein the ratio of (1) the ratio of aluminum to the support element outside the support to (2) the ratio of aluminum to support element inside the support is 2.0 or less and wherein more than 90% by weight of the alumoxane used to make the reaction product is

composed of particles having a diameter of 5 nanometers or less.

4. A composition prepared by combining porous support material and alumoxane wherein greater than 90% of the alumoxane is composed of particles having an average diameter of 5 nanometers or less, and wherein after combination the ratio of (1) the ratio of aluminum to the support element outside the support to (2) the ratio of aluminum to support element inside the support is 2.0 or less.

5. The composition of claim 1 or 2 wherein the composition is prepared by combining porous support with alumoxane wherein at least 75% by weight of the alumoxane is composed of particles having a diameter of 5 nanometers or less.

6. The composition of claim 1 or 2 wherein the composition is prepared by combining porous support with alumoxane wherein more than 90% by weight of the alumoxane is composed of particles having a diameter of 5 nanometers or less.

7. The composition of any of the preceding claims wherein the ratio is 1.5 or less.

8. The composition of any of the preceding claims wherein the support material is silica.

9. The composition of any of the preceding claims wherein the alumoxane is methylalumoxane and/or the transition metal compound is a bis cyclopentadienyl transition metal compound.

10. A method for producing polyolefin polymer comprising contacting a catalyst system comprising the compositions of any of the preceding claims with olefin monomer(s) under polymerization conditions."

II. Four notices of opposition, were filed as follows:
7 September 2000 by BP Chemicals Limited (OI),
8 September 2000 by Targor GmbH (OII),
8 September 2000 by Elenac GmbH (OIII)
8 September 2000 by Borealis Technology Oy (OIV)

All opponents requested revocation of the patent in its entirety. OI and OII invoked the grounds of lack of novelty and inventive step (Art. 100(a) EPC) and of lack of sufficiency of disclosure (Art. 100(b) EPC). OIII in the notice of opposition invoked the grounds of lack of novelty and lack of inventive step pursuant to Article 100(a) EPC and in the course of the opposition proceedings (with letter dated 25 April 2003) further introduced objections of lack of sufficiency of disclosure pursuant to Article 100(b) EPC and extension beyond the content of the application as filed, pursuant to Article 100(c) EPC. OIV invoked the grounds of lack of novelty and inventive step, lack of sufficiency of disclosure and extension beyond the content of the application as filed, pursuant to Article 100(a), (b) and (c) EPC.

III. With letter of 20 March 2001, the proprietor informed the European Patent Office of a change of name to ExxonMobil Chemical Patents Inc. Supporting documentation was provided.

IV. With letter of 21 May 2001, OIV withdrew its opposition.

V. With the letter dated 25 April 2003 OIII informed the office that the company was now trading as Basell Polyolefine GmbH, supporting documentation in the form of an excerpt from the Trade Register of the Amtsgericht Kehl, Germany, being submitted.

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

D1: US-A-5 057 475
D3: EP-A-0 206 794
D3a: US-5-191 052 (US equivalent of D3)
D5: P. Galli *et al*, Angew. Makromol. Chem. 120, 1984, 73-90
D7: US-A-5 240 894.

Together with its notice of opposition, OI submitted an experimental report relating to a replication of example 8 of D1.

In its letter dated 25 April 2003, OIII submitted an experimental report relating to a replication of the example "Catalyst X" of D3a.

VII. In a decision announced orally on 25 June 2003 and issued in writing on 8 October 2003, the opposition division found that the patent could be maintained in amended form on the basis of the fourth auxiliary request.

Claims 1, 2, 4 and 7 to 10 of this request were identical to the corresponding granted claims as reproduced above. Claim 3 read as follows, the differences compared to the corresponding granted claim being indicated in **bold**.

"3. A composition prepared by combining porous support material and **an alumoxane-transition metal complex** wherein the ratio of (1) the ratio of aluminum to the support element outside the support to (2) the ratio of aluminum to support element inside the support is 2.0 or less and wherein more than 90% by weight of the alumoxane used to make the reaction product is composed of particles having a**n average** diameter of 5 nanometers or less."

Claims 5 and 6 were also amended, as compared to the claims as granted by specifying that the particle size was an average. Claim 5 further differed from claim 5 as granted in specifying that the lower limit of alumoxane particles having the specified particle size was 70% by weight (as opposed to 75% by weight in the granted claim).

The decision held with regard to sufficiency of disclosure:

(a) that it was possible to determine the fraction of small particle size alumoxane using high resolution electron microscopy and if necessary continue settling the alumoxane solution until the required fraction was obtained;

(b) that the measurement of the ratio of Al/Si outside the support to Al/Si inside the support was amply disclosed and that the preferred method - XPS - had been shown as being suitable whereas the method proposed by the Opponents (EDXS) was not mentioned in the patent and was not suitable;

(c) that with regard to treatment of the samples submitted to XPS analysis, it was possible to ascertain when a sample had been sufficiently ground by carrying out repeated grindings and measurement until a constant ratio was achieved.

Regarding novelty, it was held that none of the documents cited disclosed either explicitly or implicitly the required Al/Si outside to Al/Si inside ratio. It was also held that the experimental data furnished by the opponents failed to establish that example 8 of D1 and Catalyst X of D3a anticipated the subject matter claimed. Specifically, it was objected that example 8 of D1 had not been exactly repeated as there were differences regarding the scale and the order of addition of components. With regard to D3a it was held that the Al/Si outside to Al/Si inside ratio of catalyst X was outside the scope of the claims.

With regard to inventive step, it was held that D1 represented the closest prior art, the distinguishing feature being the defined Al/Si outside to Al/Si inside ratio. The technical problem was to provide alumoxane supported silica which did not result in reactor fouling when used for polymerisation of olefins. D1 did not teach that alumoxane on the outside of porous silica would lead to reactor fouling, or that increasing the amount on the inside surfaces of the particles would lead to a reduction in reactor fouling. Further, no prior art document taught the role played by settling and decantation of alumoxane solutions to allow a certain particle size content and to increase the amount of alumoxane inside the porous surface. Hence an inventive step was recognised.

VIII. On 22 December 2003 OIII (Basell Polyolefine GmbH) lodged an appeal against the decision of the opposition division, the requisite fee being paid on the same date. It was requested to set aside the decision of the opposition division and to revoke the patent in its entirety, an auxiliary request being made for oral proceedings.

On 15 December 2003 a letter was received on the letterhead of BP International Limited, stating "We hereby appeal against the decision of the Opposition Division dated 17 October 2003." The letter referred to the application and publication numbers of the patent in suit and was signed by "J P H Smith" and referred to General Authorisation No. 67. The letter further bore, in the line beneath the date, a reference terminating in "BPO 252" and in the line immediately following the data relating to the patent in suit, "Ref: BPO 252". The date given for the decision against which appeal was being filed was that of the despatch of the minutes of the oral proceedings before the opposition division. A fee corresponding to the amount of the appeal fee was paid on the same day.

IX. A statement of grounds of appeal was received on 27 February 2004 from Basell Polyolefine GmbH (OIII) together with an additional document:
D25: EP-A-294 942,
and an experimental report reflecting example 1 thereof.

(a) The conclusions of the decision under appeal in respect of Article 100(b) EPC were disputed.

  (i) Specifically it was objected that the particle size was not reported in the examples of the patent in suit; that no method of measurement of particle size was exemplified in the patent and that the method referred to in the patent could not be guaranteed to yield results reliably reflecting the proportion of particles of a given size. Further the meaning of the requirement that 90 weight % of the particles have a certain average particle size was queried.

  (ii) It was disputed that X-ray photoelectron spectroscopy (XPS) was suitable since this was a surface technique and could provide no information from within the particles.

  (iii) The information in the patent in suit relating to the grinding step used to prepare the samples subjected to XPS analysis was insufficient since it was not explained in the patent what was to be understood by a "fine powder" in view of the fact that the starting material itself had an <u>average</u> particle size of 10-100 μm.

  (iv) It was observed that according to experimental data filed by OIII and the proprietor during the opposition proceedings the time of grinding required to arrive at a constant value for the ratio differed significantly. The examples furnished by OIII reported a time of the order of 10 minutes while those furnished by the proprietor reported a time of the order of 20 seconds. The skilled person thus faced a burden of extensive experimentation to repeat the teachings of the patent.

(b) The objections of lack of novelty raised in the opposition proceedings in respect of D3a were maintained. Objections of lack of novelty based on the newly filed D25 and the experimental report based thereon were raised.

With regard to Catalyst X of D3a, it was argued that since the results of the repetition furnished during the opposition proceedings showed that a ratio within the required range was obtained after 4 minutes grinding it had been established that Catalyst X of D3a fulfilled this ratio and so anticipated the subject matter of claims 1 and 2 as maintained by the opposition division.

With respect to D25 it was argued (on the strength of the experimental report) that the composition of example 1 thereof after grinding for 10 minutes yielded an Al/Si outside to Al/Si inside ratio which anticipated the subject matter of claims 1 and 2 as upheld by the opposition division.

(c) Regarding inventive step, it was submitted that since catalyst compositions which would have solved the problem set out in the patent in suit of providing polymers of controlled bulk density, low ash content and good morphology with low reactor fouling were already known in the art from D7, the technical problem was to provide further catalysts with the same advantages. It was known from D5 that homogeneous distribution of active sites throughout catalysts was a requirement for high performance of catalysts, so leading to the subject matter claimed.

X. A submission bearing the heading "Grounds of Appeal" was received on 16 February 2004 on the letterhead of BP International Limited, the covering letter bearing the aforementioned reference "BPO 252" and stating "Please find enclosed the Grounds of Appeal...". This submission was accompanied by a further citation:

D24: WO 92/05203

(a) With regard to sufficiency of disclosure, it was submitted that the skilled person faced an undue burden to establish by trial and error the level of alumoxane particle size required to obtain the specified Al/Si outside to Al/Si inside ratio and the specified degree of removal of larger particles. The particle size was not specified either in the claims or the examples, and the absence of any method for determining this was objected to.

(b) With regard to novelty, objections based on example 8 of D1 were maintained. With regard to the dismissal of the objection of lack of novelty based on the experimental report repeating example 8 of D1 on 1/10th scale, it was argued that no evidence had been provided to support the position of the proprietor that carrying out the reaction at this scale would make any difference and it should be assumed, in the absence of evidence to the contrary, that scale did not affect the outcome of the reaction.

Regarding D24, it was argued that the skilled person repeating example 1 of D24 would inevitably have obtained a composition within the scope of claim 1 as maintained by the opposition division.

(c) Regarding inventive step it was submitted that the required ratio of aluminium to support element would inevitably be obtained when following the teachings of the prior art including D1, D3 and D24. It was common practice in the art to allow alumoxane solutions to settle and to decant the supernatant, as taught *inter alia* by D3. The ratio of 2.0 or less was likely to have been achieved inadvertently. The proprietor had merely discovered a benefit of a conventionally obtained composition.

XI. In its response, dated 27 July 2004, the respondent (proprietor) submitted that BP International Limited had not been a party to the opposition proceedings and so was not a party adversely affected by the decision. Hence the admissibility of the appeal filed with the letter of 15 December 2003 on the letterhead of BP International Limited was challenged. Nevertheless the response addressed the issues raised in the submissions of both BP International Limited and Basell Polyolefine GmbH.

(a) It was objected to the submission of D24 and D25 at this stage of the procedure.

(b) With regard to sufficiency of disclosure, it was submitted that the patent provided extensive teaching regarding the settling and decanting steps and how the fraction of small particles could be determined. The XPS test was appropriate since it determined the surface concentration. The evidence submitted before the opposition division relating to grinding showed that at low grinding times the samples were insufficiently ground, whereas at longer grinding times there were no unground particles. The skilled person was able to ascertain when the sample had been ground sufficiently as the ratio measured tended to a limiting value. All that was necessary was to grind further and repeat the XPS measurement to see if an increased ratio was obtained.

(c) Regarding novelty:

(i) The validity of the repetition of example 8 of D1 was challenged, in particular it was argued that it could not be assumed that scale would exert no influence on the outcome of the reaction. This was particularly the case with heterogeneous and highly exothermic reactions. Further the second stage of the example of D1, i.e. addition of the transition metal compound and heptane followed by reaction for a further hour had been omitted which, it was submitted, would alter the course of the reaction. The catalyst of D1 had a productivity of 49g polymer/g catalyst/hour compared with a productivity of 960g polymer/g catalyst/hour in example 1 of the patent in suit, suggesting that the catalyst of D1 was different from that of the patent in suit.

(ii) The examples based on D3a showed that on sufficient grinding a ratio outside the scope of claim 1 of the patent in suit was obtained. It was disputed that the decantation step disclosed in D3a, which had a different purpose from that in the patent in suit, would have had the same effect as the decantation taught in the patent in suit.

(iii) The literal disclosure of D24 did not disclose the required ratio.

(iv) Concerning D25 a number of discrepancies between the procedure disclosed in the citation and that carried out in the experimental report were identified rendering the conclusions drawn invalid. *Inter alia* it appeared that the ratio of the components employed, in particular alumoxane to metallocene had been modified, a different alumoxane and a different silica had been used ("Sylopol 952" instead of "F 952").

(d) Regarding inventive step, the respondent submitted that the invention was based on the discovery that alumoxane solutions were made up of a mixture of large and small particles. The proportion of small particles could be enhanced, for example by allowing the larger particles to settle, leading to compositions in which the proportion of alumoxane inside a porous support was increased as compared to previously known supported alumoxane compositions. There was no recognition of the nature of the particle size distribution of alumoxanes in the prior art, that it would be possible to enhance the proportion of alumoxane on the internal surfaces of a porous support, or of the reduction in reactor fouling resulting therefrom. It was emphasised that settling over a "period of time" was necessary, and that the fact that a solution might appear free of cloudiness did not mean that the solution was free of particles of greater than 20nm diameter. Rather, cloudiness would only result from the presence of particles having a diameter at least as great as the wavelength of optical light (300-700nm). It was in any case previously believed that alumoxane should be employed "fresh" and in the case that old alumoxane was used, the bottles were commonly shaken before use to ensure that the alumoxane was of the original concentration.

Regarding the objection based on a combination of D7 and D5, it was submitted that D5 related to Ziegler-Natta catalysts and therefore would not have been considered by the skilled person seeking assistance with improving alumoxane metallocene systems. It did not explain what was meant by "homogeneous" distribution of active centres or how this was to be achieved. D7 did not contain any teaching to employ a prolonged settling step followed by decantation.

XII. On 28 July 2004 a submission from BP International Limited dated 29 July 2004, was received. This submission consisted of an experimental report relating to a repetition of example 1 of D24.

XIII. In a letter dated 2 February 2005 from BP International Limited, it was argued that the appeal was admissible. The submissions of the respondent to inventive step in respect of the increased content of small particles were challenged since the claims did not define this feature.

XIV. With a letter dated 22 June 2005 headed "Change of Representative" it was stated that Innovene Europe Limited had "taken over responsibility for handling the opposition". The letter was jointly signed by "S.E. Stevens, Head of Formalities" and "Michael Preece". The latter signatory referred to General Authorisation No. 67 (see above). In an annexed table the number of the patent in suit was listed against the "Case Number" BPO252.

XV. The Board issued on 8 December 2005 a summons to attend oral proceedings. In the accompanying communication, the issue of admissibility of the appeal apparently filed in the name of BP International Limited was discussed. Further some objections pursuant to Article 100(c) EPC were indicated.

XVI. With a letter dated 20 December 2005 it was stated that BP Chemicals Limited had assigned their rights in the opposition against European Patent 739 365 to O & D Trading Limited on 1 April 2005, which entity had on 1 June 2005 changed its name to Innovene Europe Limited. The letter was signed by "S.E. Stevens, By Power of Attorney". This letter contained an annex entitled "Assignment", bearing the reference "Opposition No. BPO 252" assigning "all right and title in the said opposition" to Innovene Europe Ltd. The "Assignment" bore two signatures, both dated 20 December 2005, one of "Michael Preece by Power of Attorney" signing on behalf of BP Chemicals Ltd, and a second signature on behalf of Innovene Europe Limited by "Susan Elizabeth Stevens, By Power of Attorney". Further, a certificate of incorporation recording the change of name from O & D Trading Limited to Innovene Europe Limited was submitted. It was requested that the "Assignment" be recorded and the transfer be registered.

XVII. In a letter dated 21 December 2005 on the letterhead of Innovene, and signed by "J P H Smith" it was requested that prior to the oral proceedings a decision be made on the admissibility of "our appeal". It was noted that the General Authorisation reference given on the notice of appeal was that of BP Chemicals Limited and that it should be assumed that this entity was the appellant.

XVIII. In a communication dated 13 January 2006 the Board stated that the question of admissibility of the appeal would be decided during the oral proceedings. The Board further raised a number of objections concerning the admissibility of the requested transfer of the opposition.

XIX. With a letter dated 7 February 2006, the respondent submitted a main and six auxiliary requests, as well as 4 declarations of experts by:
Milham S. Howie dated 17 January 2006
Jeffrey H. Butler dated 26 January 2006, the final page bearing the signature having the date 30 January 2006.
Gary M. Brown dated 30 January 2006
David M. Glowczwski dated 30 January 2006
and two additional citations:

D26: A paper given at Polyethylene '93 by M. S. Howie;

D27: Barber, J. "On a single site", Asia-Pacific Chemicals 1999 pages 30-31.

It was requested that the experimental report filed with the letter dated 29 July 2004 in respect of example 1 of D24 not be admitted into the proceedings since this had been filed late.

(a) Compared to the main request as upheld by the opposition division, the newly submitted main request differed in that:

  (i) claim 3 had been deleted and the following claims renumbered accordingly.

  (ii) the dependency of claims 4 and 5 (former claims 5 and 6) had been restricted to claim 1.

  (iii) the alternative "and" had been deleted from claim 8 (former claim 9).

  Accordingly amended claim 8 read as follows:

  "8. The composition of any of the preceding claims wherein the alumoxane is methylalumoxane or the transition metal compound is a bis cyclopentadienyl transition metal compound."

(b) With regard to the objections of lack of sufficiency of disclosure relating to the grinding time raised in the letter of 16 February 2004 on the letterhead of BP International Limited - referred to by the respondent as "Opponent I" - in the "statement of grounds of appeal" it was argued that since OI had filed two separate test reports reproducing this test with no indication of difficulty or uncertainty, inconsistent positions were being adopted.

With regard to the measurement of the alumoxane particle size, it was submitted that this was amply described in paragraphs [0014] and [0045] of the patent.

With regard to the analytical method for determining the ratio, it was submitted that XPS was a surface method and hence was suitable. The method proposed by the opponent (EDXS) was not suitable since it measured concentrations through the bulk. The unsuitability of this method was confirmed by the results of OIII showing that measurements by this method on crushed and uncrushed samples gave almost identical Al/Si ratios.

It was further submitted that inert conditions were not required during grinding, identical results being obtained if grinding was carried out after deactivating the catalyst by exposure to air.

(c) Regarding novelty, it was objected that the report based on example 1 of D24 failed to reproduce this example correctly.

It was further submitted on the basis of two sets electron photomicrographs forming part of the Butler declaration, one dating from investigations carried out in 1991 and one based on a contemporary study, that alumoxane solutions commercially available at the priority date of the application contained a significantly higher proportion of large secondary particles than did "modern day" alumoxanes.

(d) Regarding inventive step, the argument that prior to the patent in suit, it was not known that alumoxane exhibited a bimodal particle size distribution was emphasised, reference being made to the Howie declaration and to D26. No document cited by the opponents contained any teaching about this.

Regarding the question of decantation of alumoxane solutions, it was submitted with reference to the Howie declaration that at the priority date of the patent in suit alumoxane was provided in steel cylinders under nitrogen, the material being removed by applying nitrogen pressure to force alumoxane out via a dip tube which reached to the bottom of the cylinder. This rendered decantation impossible. Further Howie stated that due to the risk of gelation of the alumoxane it was common practice to agitate the cylinders.

An alternative practice was to store alumoxane in a continuously stirred tank. It was further submitted that gelled alumoxane was just as active at activating metallocene as gel-free alumoxane with the consequence, in view of the cost and hazardous nature of alumoxane, that the skilled person would have been motivated away from decanting supernatant methyl alumoxane and discarding the gels. According to the respondent this meant that there was a prejudice against storing methyl alumoxane, decanting and using only the supernatant. Thus the procedure of decantation proposed by the opponents was contrary to the recommendations of methyl alumoxane manufacturers at the relevant time and would not even have been possible with the commercially available alumoxanes supplied in steel bottles.

XX. With a letter dated 6 March 2006 on the letterhead of Innovene and addressed to Directorate General 2 of the European Patent Office, it was stated that the representative would provide an authorisation from BP Chemicals Limited so that this party could be represented at the Oral Proceedings in the case that the Board maintained its view that the transfer requested had not been effected.

XXI. Oral proceedings were held on 8 March 2006.

(a) *Parties present*
The representative of Basell Polypropylen GmbH (OII, formerly Targor GmbH) and Basell Polyolefine GmbH (OIII) informed the Board that the former OII and OIII had merged. The opposition raised by OII was maintained. The Board observed that since Basell Polypropylen GmbH was no longer in existence it could not be a party to the present proceedings. The representative indicated his agreement to represent only the appellant Basell Polyolefine GmbH.

(b) *Requested transfer to Innovene Europe Limited of the appeal filed by BP International Limited*
The Representative jointly authorised by Innovene Europe Limited and BP Chemicals Limited indicated that the request for transfer of the opposition was not pursued and it was no longer part of the proceedings. Thus the sole party represented was BP Chemicals Limited.

Following deliberation, the Board decided that the appeal filed apparently in the name of BP International Limited had in fact been filed on behalf BP Chemicals Limited, which was a party adversely affected by the decision of the opposition division. Hence this appeal was admissible and BP Chemicals Limited was a party to the appeal proceedings.

(c) *Grounds invoked*

The grounds of opposition pursuant to Article 100(a) (Art. 54 and 56 EPC) and 100(b) EPC were maintained. No objections were raised pursuant to Articles 100(c) or 84 EPC.

(d) *Admissibility of D24, D25 and the experimental reports*

The respondent objected to the filing of the experimental report based on example 1 of D24, which had not been foreshadowed in the statement of grounds of appeal, 5 1/2 months after the statement of grounds, and after the filing of its Rejoinder.

The appellant BP Chemicals Limited argued that the respondent had been aware of this objection from the outset of the Appeal proceedings and had had sufficient time to consider the evidence prior to the oral proceedings before the Board.

(e) *Sufficiency of disclosure*

(i) *Grinding*

The appellants submitted that there was no teaching in the patent of the need to grind until constant values had been achieved, and no disclosure in the patent that would allow the skilled person to understand what "complete grinding" was.

The respondent submitted that the purpose of grinding was to expose the internal structure of the particles. A detailed discussion was unnecessary and could even be misleading since different operators would work differently. It would be evident to the skilled person that the final value would be approached asymptotically. This effect was rendered evident by the photomicrographs presented during the opposition proceedings.

(ii) *Analysis method*

The appellants submitted that since it was necessary to know the content of aluminium within the particle a method which allowed analysis through the cross section such as EDXS would be employed. Since different measurements were available which although equally appropriate gave different results the invention was not sufficiently disclosed.

The respondent argued that the analytical method to be employed was a surface method, and XPS, the method of choice in the patent in suit and employed in the examples was such a method. It had never been accepted that EDXS was an appropriate method for surface analysis. Rather, EDXS made no distinction between the surface and the bulk of a sample.

(iii) *Obtaining a content of particles of defined average size*

The appellant Basell Polyolefine GmbH submitted with respect to claims 3 to 5 of the main request that the requirement that a proportion (90 or 70%) of the particles have an average diameter of 5nm of less was unclear since it was not explained which particles were taken into account when calculating the average. Since it was not known how to calculate this, it would be impossible to know whether a given composition was within the scope of the claims.

The respondent argued that there was much teaching in the patent regarding the bimodal particle size distribution of the alumoxanes and the definition objected to was a consequence of this bimodal distribution. There were two populations of particles and it was necessary to set a limit to define to which group of particles a given particle belonged as explained at page 4 of the patent in suit.

(f) *Novelty*

(i) With regard to the repetition of example 8 of D1, the appellant BP Chemicals Limited relied essentially on the submissions made in the written procedure, arguing that the factors discussed by the respondent would not have affected the outcome.

The respondent referred to its submissions in the written procedure emphasising the issue of scale.

(ii) Regarding D3a the appellant Basell Polyolefine GmbH referred essentially to the written submissions, in particular emphasising the effects that grinding times had on the results obtained.

(iii) The appellant BP Chemicals Limited made no further submissions relating to D24.

(iv) Regarding D25 and the experimental report based thereon, the appellant Basell Polyolefine GmbH acknowledged that there were differences but submitted that these were not relevant to the outcome.
With regard to the differing designations of the silica ("F-952" in D25 and "Sylopol 952" in the experimental report) it was submitted these were different commercial designations for the same product, which submission was not challenged.

(g) *Inventive Step*
The appellants reiterated the objections raised in the written procedure that such compositions would have inevitably been obtained in carrying out prior art teachings.

(i) Further it was submitted that the skilled person would inevitably have rejected gelled or hazy MAO solutions since it was known that these led to reactor fouling.
Decantation would have been routinely carried out leading to the invention of the patent in suit.

(ii) The appellant BP Chemicals Limited referred to the use, in their laboratories, of the "daily bottle" of alumoxane from which a portion was regularly decanted for use in the preparation of catalysts.

(iii) The closest prior art was considered to be D1, the problem to be solved was to avoid reactor fouling and the solution of using clear MAO solutions was obvious. The patent in suit merely provided an explanation of what the skilled person would in any event have done. No relationship between the particle size and the ratio defined in the claims had been shown.

(iv) With regard to D7 and D5 the position set out in the written submissions was essentially reiterated.

The respondent argued that it had not been established that fouling was the result of the use of gelled MAO.

(v) The invention was not the removal of gels, but selection of a certain particle size of less than 20nm. The arguments put forward in the written procedure, that this requirement went beyond an optically clear solution, were reiterated.

(vi) The respondent was not aware of the "daily bottle" of alumoxane practice referred to by the appellant BP Chemicals Limited. On the contrary their practice was to subject steel cylinders to agitation (Section XIX(d) above) to break up the gels, leading away from the suggestion of a settling period.

(vii) The contribution of the patent in suit to the art was twofold: the realisation that fouling and bulk density were related to the distribution of alumoxane within the silica particles and the realisation that alumoxane exhibited a bimodal particle size distribution.

(viii) D7 did not teach to remove gels, but taught that residual solvent posed a problem. The catalyst of example 2 of the patent was prepared according to the method of D7 and did not fall within the scope of the claims.

(ix) D5 was specific to a different branch of catalyst technology, namely Ziegler-Natta catalysts, which did not contain silica, alumoxane or metallocene. Further, the support referred to in D5 was not an inert material as in the case of the patent in suit but played an active role in the reaction.

XXII. The final requests of the parties were:

The appellants (opponents) requested that the decision under appeal be set aside and that the European patent No. 739 365 be revoked.

The respondent (proprietor) requested that the decision under appeal be set aside and the patent maintained on the basis of the main request, or in the alternative, on the basis of one of the 1st to 6th auxiliary requests, in that order, all filed with the letter dated 7 February 2006.

Reasons for the Decision

1. *Admissibility of the appeals*

1.1 The appeal filed by former OIII (Basell Polyolefine GmbH) is admissible. This has not been disputed.

1.2 Regarding the "Notice of Appeal" filed with the letter dated 15 December 2003 on the letterhead of BP International Limited, the following facts are relevant:

- The General Authorisation referred to, No. 67 is assigned to BP Chemicals Limited.
- The letter was signed by the same person who filed all submissions in the opposition phase and attended the oral proceedings before the opposition division in the name of BP Chemicals Ltd.
- The same internal reference, "BPO 252", is present on the letter announcing that an appeal is being filed and on all correspondence from BP Chemicals Limited filed in the opposition phase.

In view of these facts the Board is satisfied, the absence of an explicit reference to BP Chemicals Limited in the "Notice of Appeal" notwithstanding, that the Appeal was filed on behalf of the former OI BP Chemicals Limited and was therefore found admissible.

2. *Admissibility of the documents filed together with the statements of grounds of appeal*

2.1 The appellants each filed a new citation together with the respective statements of grounds of appeal (D24 and D25). Further, the appellant Basell Polyolefine GmbH filed concurrently an experimental report based on

example 1 of D25. The Board is satisfied that there was no reason for the parties to file these documents before the opposition division, since other documents and evidence were being relied upon at that stage, which facts and evidence were found not to be convincing by the opposition division.
Accordingly D24, D25 and the experimental report relating to example 1 of D25 were admitted to the appeal proceedings.

2.2 The experimental report relating to example 1 of D24, was, however, filed on 28 July 2004, some 5 1/2 months after the respective statement of grounds of appeal. It was neither announced in the statement of grounds that any such report was in preparation nor was any other indication given that it was intended to submit such a report.
According to Article 10a(2) of the Rules of Procedure of the Boards of Appeal, in the version valid as from May 2003 which is applicable to the present case, the statement of grounds of appeal shall contain a party's complete case. Further Article 10b(1) of the Rules of Procedure states that any amendment to a party's case may be admitted and considered at the Board's discretion, said discretion being exercised in view of the complexity of the new subject matter, the current state of the proceedings and procedural economy. The case made by the appellant in the statement of grounds of appeal was that the literal disclosure of example 1 of D24 provided an anticipation of the subject matter of the patent in suit. The argument based on an experimental report allegedly repeating this example was not, however, part of the case initially made and

therefore could not be taken into account by the Respondent in preparing its reply to the appeals.
The submission of this report, therefore, resulted in a material change in the case of the Appellant. Further the report, which was not foreshadowed in the statement of grounds of appeal, was submitted after the respondent had prepared its response to the appeals, which response included claims amended to take into account the submissions made in the two statements of grounds of appeal. The submission of the experimental report potentially made it necessary for the respondent to reappraise the submissions made in order to ascertain whether any modifications or additions thereto had become necessary due to the new situation thus created. Such a procedure would not be in the interests of procedural economy.
In view of the foregoing considerations and the request of the respondent to exclude this, the experimental report relating to example 1 of D24, filed by appellant BP Chemicals Limited with letter dated 29 July 2004 was not admitted to the procedure.

2.3 No objections have been raised by the appellants to admission of the two citations (D26 and D27) and the four declarations provided with the submission of the respondent dated 7 February 2006, nor has the Board any objections of its own.

Accordingly D26 and D27 and the four declarations were admitted to the procedure.

2.4 Regarding the submission of a total of seven sets of claims as the main and 1st-6th auxiliary requests by the respondent with its letter dated 7 February 2006, neither appellant has raised any objection to the filing thereof one month prior to the oral proceedings. The board is satisfied in the light of the explanations of the respondent in the written proceedings that these newly filed requests were submitted in response partly to the evidence filed for the first time at the appeal stage and partly to objections raised in the communication of the board accompanying the summons to oral proceedings. Accordingly these requests were admitted to the proceedings.

3. *Article 123(2) and (3) EPC - main request*

Neither of the appellants has raised any objections under these articles against the claims of the main request, nor has the Board any objections of its own. Accordingly it is concluded that claims 1 to 9 of the main request meet the requirements of Articles 123(2) and (3) EPC.

4. *The patent in suit*

According to claim 1 of the main request, the patent in suit relates to a composition comprising alumoxane and porous support, wherein the ratio of:

(1) the ratio of aluminium to the support element outside the support to
(2) the ratio of aluminium to support element inside the support

is 2.0 or less.

4.1 The compositions of the patent in suit are used in combination with metallocene compounds as polymerization catalysts for polyolefins. According to paragraph [0001] of the patent in suit, the catalysts are capable of providing polymers in granular form having narrow molecular weight distribution, low ash content and superior bulk density. It is explained (paragraph [0004] of the patent) that during the polymerization process with supported catalysts the catalyst becomes shattered into fragments which end up scattered throughout the polymer ("ash"). The ash content is reduced if catalyst efficiency is increased, i.e. if more polymer is produced using less catalyst. Polymer production can be adversely influenced by inconsistencies in the behaviour of a particular catalyst if, for example, the catalyst becomes inhomogeneously distributed throughout the reactor, leading to "hot spots" where the temperature becomes so high that the polymer melts and fuses, resulting in fouling of the reactor. Thus homogeneity of the reactive species throughout the reactor is preferred.

4.2 The physical form of the polymer is controlled by fragmentation of the catalyst which in turn is determined by the dispersion of the catalyst on and throughout the support. The consequence is that the catalyst loading on to the support, i.e. location and chemical nature within the support microstructure is an important consideration (paragraph [0005]).

4.3 The preferred catalyst structure is to have the same or a higher loading of alumoxane inside the support than outside (paragraph [0031]). According to the patent in suit, it has been discovered that alumoxane exhibits a bimodal particle size distribution, one group of particles having an average particle diameter of 5nm, the second group having an average size around 20nm or more. The smaller size particles are non-interactive and exist as free flowing particles, while the larger particles have a propensity to agglomerate (paragraphs [0037], [0040], [0043], [0044]). By removing the larger particles, e.g. by standing to allow settling of the particles and decantation prior to combining with the support it is possible to obtain a solution having an increased content of the particles of size 5nm or less (paragraph [0055]).

4.4 The distribution of the particles within the support is determined by techniques such as electron microscopy, secondary ion mass spectroscopy or X-ray Photoelectron spectroscopy (XPS)(paragraph [0033]). XPS is emphasised in paragraphs [0034] and [0035] and is the method employed according to the examples. A measurement is carried out on the supported catalyst to determine the ratio of aluminium to support. The sample is then crushed (ground), crushing being defined as "to a finely ground solid" and a second measurement carried out, the ratio of the ratios measured for the noncrushed to the crushed samples gives the ratio of aluminium to support outside:inside. According to paragraph [0035] it is assumed that the aluminium measured in the crushed samples that is derived from the "external surface" of the support is negligible when included in the total aluminium. In the case of a silica support the ratio measured would be that of Al:Si.

4.5 The examples show the preparation of a catalyst employing a clear decanted methylalumoxane (MAO) supernatant (MAO 30 weight %) which is reacted first with a metallocene and to which product is then added dehydrated silica. This catalyst is employed in the polymerization of propylene yielding a polymer free of chunks and fouling. The productivity of the polymer is reported as being 0.96 kg/g/hour (kilograms of polymer/g catalyst/hour). Further examples show that in the case where no settling step is carried out the measured ratio of Al/Si outside the support to Al/Si inside is 2.79 to 0.78 (3.6) or 200 to 0.97 (206) whereas in the case where settling was effected and the supernatant decanted, a ratio of 0.48 to 0.66 (0.72) was determined.

5. *Article 83 EPC - main request*

5.1 Characterisation of the composition: Grinding

The subject matter of claim 1 of the main request is characterised by the ratio of aluminium to support element outside the support to the ratio of aluminium to support inside the support as explained above. The respondent submitted at the oral proceedings before the Board (paragraph XXI.e.i above) that it would be evident to the skilled person that the purpose of the grinding was to release material from within the particles so that it could be observed and hence that no detailed instructions were necessary.
As established by the evidence provided during the opposition proceedings, specifically the repetitions of example 8 of D1 and Catalyst X of D3a, it was possible for the opponents to carry out the necessary measurements to obtain this ratio. It is also apparent that the opponents were able to establish the necessary conditions and degree of grinding in order to obtain reliable, or "correct" values.

In particular, the opponents observed that the ratio measured changed with the progress of the grinding time, approaching a final value asymptotically. This is shown by the evidence filed by opponent Basell Polyolefine GmbH with the statement of grounds of appeal in the form of a table showing the change in ratio as grinding time increase. Further evidence of this is provided by the data submitted during the procedure before the opposition division, both by the opponent Basell Polyolefine GmbH (letter of 25 April 2003) and the proprietor (letter of 25 April 2003) in the form of electron micrographs and optical micrographs respectively. The opponent also submitted with the aforementioned letter a graphical representation of the change in ratio as a function of grinding time. This evidence shows the progressive reduction in particle size as grinding progresses, that the degree of breaking up and homogeneity of the particles increases as the grinding progresses and that the final value of the relevant ratio is approached asymptotically.

According to paragraph [0035] of the patent in suit, the assumption is that the contribution of the "external" surface is negligible when included in the total aluminium determined for the crushed sample. This condition can be met only if the contribution of the "internal" surface to the ratio measured is dominant and that of the "external" surface is insignificant. This in turn can only be achieved by maximising exposure of the internal surfaces of the support to ensure that the greatest possible contribution to the measured ratio is derived therefrom.

Hence based on the information in the patent in suit, in particular paragraph [0035] the skilled person would have understood the need to grind until a constant value was obtained, corresponding to the situation where the internal surface was completely exposed such that further grinding led to no additional exposure thereof. On the basis of this understanding, the skilled person would have been able to determine when a sample had been sufficiently ground and hence when the "correct" value for the "inside" ratio had been measured.

Therefore, since it is possible on the basis of the teachings of the patent in suit to ascertain when grinding has been sufficient, the absence of any specific instructions as to how to carry out the grinding does not represent a burden to carrying out the measurement, and hence to carrying out the claimed invention. This conclusion is supported by the fact that the opponents were able to identify the necessary boundary conditions, i.e. the need to grind until a constant result was obtained.

It is therefore concluded that the information in the patent relating to grinding is sufficient to have enabled the skilled person to understand the purpose of the grinding step and how to carry this out in order to correctly measure the ratio.

5.2 Characterisation of the composition: Analytical method

The patent in suit teaches in paragraph [0033] a number of methods that can be employed to determine the location of the particles. From paragraphs [0034], [0035] and the examples, it is clear however that XPS is the method of choice. The appellants have not shown that XPS would not be suitable. Rather the appellants have restricted themselves to arguing and demonstrating

that a different method - that is not even mentioned in the patent in suit (EDXS (energy dispersive x-ray spectroscopy)) - led to different results and hence that the invention was not sufficiently disclosed.
In order to prove the allegation of lack of sufficiency of disclosure it would have been necessary for the appellants to establish that the method disclosed in the patent in suit would not lead to the correct results. However this has not been done.
Thus the appellants have failed to establish that the patent in suit does not disclose sufficiently the analytical method for determining the ratio of Al:Si.
It is therefore concluded that the analytical method employed for determining the ratio of aluminium to support element is sufficiently disclosed.

5.3 Achievement of a content of particles of the defined average size

The patent in suit explains in paragraphs [0012], [0043] and [0044] that alumoxane exhibits a bimodal particle size distribution, one fraction being 10-190 Ångstrom primary particles, which remain independent in the colloidal suspension, and the other fraction being secondary particles of size 200-10,000 Ångstrom which agglomerate into networks. In paragraph [0045] it is taught that the size of the particles may be determined by high resolution electron microscopy - which statement has not been challenged by the appellants. Paragraph [0012] teaches that the distribution and content of aluminium particles can be controlled by allowing the larger particles to settle out, so enhancing the content of small particles. A similar teaching is given in the examples, in particular

example 3 which teaches decanting the supernatant from an aged bottle of alumoxane.
Therefore the patent in suit teaches how to increase the content of small alumoxane particles (by settling) and how to establish what the content of particles of different sizes is (electron microscopy).
Accordingly it is possible to obtain, and confirm the existence of, the alumoxane suspensions required for the patent in suit.

5.4 A further objection raised in the context of Article 83 EPC by the Appellants related to the meaning of the definition of the particle sizes in, for example, claim 3 wherein greater than 90% of the alumoxane should be composed of particles having an average diameter of 5nm or less.
This appears to be an objection in relation to clarity. Since this wording was already present in claim 4 as granted and was not introduced into the claims during the opposition proceedings, and since Article 84 EPC is not a ground of opposition, this objection is not admissible.

5.5 It is therefore concluded that the invention is sufficiently disclosed and thus that the requirements of Article 83 EPC are satisfied.

6. *Novelty - main request*

The objections of the appellants with respect to novelty rely on the documents D1, D3a, D24 and D25, supplemented in the case of all documents with the exception of D24, by experimental reports. Although an experimental report was also submitted in respect of an example of D24, for the reasons explained in paragraph 2.2 this has not been admitted to the procedure.

6.1 General considerations

In the case of arguing a lack of novelty based on such a replication of an example of a prior art citation, the case being made is that the subject matter claimed, even if not anticipated by the explicit literal disclosure of the citation is nevertheless implicitly anticipated to the extent that in carrying out the express literal disclosure and instructions of a prior art document (e.g. an example) subject matter falling within the terms of the claims of the patent in suit is the inevitable outcome. This has the consequence that there can be no space for doubt and hence that the "balance of probability" is not the appropriate standard to apply. Rather, a stricter standard of proof, namely "beyond all reasonable doubt" needs to be applied. This means that if there is any reasonable doubt as to what may or may not be the result of carrying out the literal disclosure and instructions of a prior art document, i.e. if there remains a "grey area" then the case of anticipation based on that document must fail (see T 793/93, 27 September 1995, not published in the OJ EPO, Headnote and section 2.1 of the reasons).

6.2 D1

6.2.1 According to claim 1, D1 discloses a catalyst system comprising an inert support, a transition metal compound of defined formula, and an alumoxane. According to claim 21 the supported catalyst system of

D1 is prepared by dissolving an alumoxane in a hydrocarbon solvent, mixing the alumoxane with an inert support material and then adding to the mixture the transition metal component (defined as in claim 1 of D1 which in one embodiment can be a metallocene). Regarding the discussion of preparation of the supported catalyst system in the description of D1, according to column 5, lines 4 to 18 the supported catalyst systems may be prepared by various methods. The transition metal component and the alumoxane component can be mixed together before addition of the support material, for example by mixing in a common solvent which preferably is suitable for use as a polymerization diluent. Alternatively the alumoxane can be placed on the support material followed by addition of the transition metal component, or the transition metal component can be applied to the support material followed by addition of the alumoxane. According to column 5, lines 18 to 22 the alumoxane employed may be either purchased as a commercial solution or produced *in situ* by the addition of a trialkylaluminium to a wet support, e.g. by addition of trimethylaluminium to wet silica. This latter method, which results in preparation in a first step of alumoxane on the support material is discussed as "Preparation Method 3" starting at column 14, line 33 of D1.

6.2.2 This "Preparation Method 3" is exemplified in Example 8 of D1:

A 1.4M solution of trimethyl aluminium in 200 ml heptane was placed in a 1l flask. Untreated silica gel (Davidson 948 Grade, 50g) containing 9.6% water was slowly added to the flask. After completion of the addition of silica, the mixture was stirred for one hour at ambient temperature. A transition metal compound MePhSi(Me_4C_5)(N-t-Bu)$TiCl_2$ (1.35g, 3.1 mmol) slurried in 50 ml heptane was added. The mixture was permitted to react for one hour and was then heated to 65°C while a nitrogen stream was passed through the flask to remove the solvent. The nitrogen stream was stopped when the mixture in the flask turned into a free flowing powder. Polymerisation of ethylene in a gas phase laboratory reactor gave a polymer yield of 49g polymer/g catalyst/hour.

6.2.3 Neither the general discussion nor the examples of D1 disclose explicitly the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support.

6.2.4 The appellant BP Chemicals Limited argued on the strength of the experimental report submitted together with its notice of opposition that the composition of example 8 of D1 would exhibit the required ratio.
In the reported repetition of this example the following was carried out, without however the transition metal catalyst being added.
5.0g of silica (Davidson 948 Grade, determined to contain 9.67% water) was added to 20 ml of a 1.4 molar solution of trimethyl aluminium over 5 minutes. The solution was maintained at 25°C for one hour with stirring. The mixture was then heated to 65°C under a nitrogen stream to remove the solvent, yielding a free flowing solid.

(a) A comparison of the experimental report submitted and the disclosure of example 8 of D1 however reveals the following differences:

(i) Example 8 of D1 employed 50g of silica whereas the repetition employed 5g. The amounts of the other components employed were similarly reduced by a factor of 10.

(ii) In example 8 of D1 subsequent to the step of stirring trimethyl aluminium with the silica a further step of addition of a transition metal compound slurried in heptane, followed by reaction for one hour was carried out.

(iii) Therefore in the repetition two steps reported in example 8 of D1 were omitted. Firstly, addition of the transition metal compound together with the heptane in which it was slurried was not carried out. Secondly, the further reaction period of one hour was omitted, meaning that the total reaction time carried out in the repetition was reduced by this period compared to the disclosure of example 8 of D1.

(iv) As a consequence a product was isolated according to the experimental report which in D1 was not isolated. Rather, according to example 8 of D1 the product at this stage was subject to reaction with additional components (transition metal compound in heptane) and for a further period of time (1 hour).

(b) Therefore there exist a number of discrepancies between the process carried out by the appellant and the disclosure of D1, specifically:

- The repetition was carried out on 1/10th scale;
- An intermediate product was isolated which in the example of D1 was subjected to further reaction without isolation;

- two reaction steps (addition of a metallocene compound in heptane and one hour further reaction) were omitted.

(i) Regarding the question of scale, it has been argued by the respondent and not disputed that the reaction in question is a heterogeneous system. The respondent further argued that it could not be assumed that the difference in scale between example 8 as disclosed in D1 and the repetition would exert no influence on the outcome of the reaction, which argument has been disputed. The board notes in this respect that it has not been demonstrated that in the 1/10th scale repetition of example 8 of D1, all factors, in particular geometry of the reaction apparatus and rate of addition of the reactants were appropriately scaled. These factors are of particular importance due to the heterogeneous nature of the reaction in question. As a consequence, it has not been established that the reaction conditions employed in the repetition of example 8 of D1 were appropriately modified to take account of the different amounts of reactants employed.

(ii) It is further apparent, in particular due to the isolation of an intermediate and omission of reaction steps, that the process as disclosed in D1, example 8 was not in fact carried out. Therefore the product resulting from the repetition is not the product obtained in example 8 of D1.

(c) The Appellant has advanced no evidence that despite the differences noted, the properties of the product, in particular the noted ratio, would have been identical to those obtained had the procedure of example 8 of D1 been followed accurately. In particular, it has not been demonstrated that the properties were not in some way modified by the isolation of a product at an intermediate stage. It has also not been demonstrated that the omission of the final two steps had no effect on the product properties. Accordingly, the Appellant has not demonstrated to the required standard of proof (see 6.1 above) that the product resulting from carrying out the literal disclosure of example 8 of D1 exhibits the ratio of the ratio of aluminium to support element (i.e. silicon) outside the support to the ratio of aluminium to support element inside the support as required by the independent claims of the main request. Accordingly it has not been demonstrated that the implicit disclosure D1, as represented by the product of example 8 thereof anticipates the subject matter according to claim 1 of the main request.

(d) Regardless of the foregoing, other evidence indicates that the product of D1 example 8 is not identical to the compositions according to claim 1 of the main request. According to the results of the polymerisation experiments reported in D1 the productivity of the catalyst is 49g polymer/g catalyst/hour which is a factor of 20 lower than the value of 960g polymer/g catalyst/hour reported for the catalyst demonstrated in example 1 of the patent in suit.

6.2.5 Therefore neither the evidence of the explicit, literal disclosure of D1 nor the evidence relating to the implicit disclosure thereof, submitted in the form of an experimental report allegedly repeating example 8 of D1 supports the position of the appellant that the catalyst system disclosed in D1 example 8 exhibits the ratio of the ratio of aluminium to support element outside the support to the ratio of aluminium to support element inside the support as defined in the independent claims according to the main request.

6.2.6 Accordingly the subject matter claimed according to the main request is novel with respect to the disclosure of D1.

6.3 D3a

6.3.1 According to claim 1 D3a relates to unsaturated copolymers the chains of which comprise polymerised units of ethylene and 1,3-butadiene. The butadiene is incorporated into the backbone chain in various structures, namely as *trans*-1,2-cyclopentane units, 1,2-non-cyclic butadiene comonomer units and 1,4-*cis* and *trans* non-cyclic butadiene comonomer units. According to column 1, lines 23 to 26 the polymers have improved clarity, toughness and lower unsaturation than prior art ethylene-butadiene polymers. According to column 2, lines 49 to 55, D3a also provides a new metallocene/alumoxane catalyst for olefin polymerization. According to column 2, lines 56 to 60 and column 13, lines 44 to 50 the catalyst is prepared by reacting an alumoxane and a metallocene in the presence of a solid support material.

6.3.2 According to column 3, lines 34 to 39, a preferred method for preparing the alumoxanes is to contact an aluminium alkyl (e.g. aluminium trimethyl) with a hydrated salt such as hydrated ferrous sulphate. The method comprises treating a dilute solution of aluminium trimethyl in for example toluene with ferrous sulphate heptahydrate. This is the method employed according to the "Example" reported at column 22, lines 18 to 33 of D3a. In this example, the alumoxane is prepared by addition of ferrous sulphate heptahydrate to a rapidly stirred solution of trimethyl aluminium in toluene. After addition and further reaction for 6 hours the reaction mixture is allowed to cool and settle for an unspecified time. The clear solution containing the alumoxane is separated by decantation from the insoluble solids.

6.3.3 The example "Catalyst X" of D3a reported the preparation of a catalyst indicated generally above. Specifically the catalyst is prepared by the steps of slurrying silica in toluene, adding a solution of alumoxane prepared as indicated in paragraph 6.3.1 above, isolating the resulting solid, redispersing this in toluene and adding thereto a solution of the required metallocene compound, allowing further reaction and then isolating the resulting solid.

6.3.4 Neither the general description nor the examples of D3a disclose the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support.

6.3.5 The aforementioned Catalyst X of D3a was repeated by the appellant Basell Polyolefine GmbH, as evidenced by an experimental report filed initially during the opposition proceedings and enclosed with the statement of grounds of appeal. The respondent has not challenged the accuracy of the replication as set out in the corresponding experimental report, nor has the Board any objections of its own in this respect.
According to the experimental report, the product obtained was subjected to grinding and the resulting change in the structure determined by electron microscopy.
The results of the determination of the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support upon grinding for progressively longer periods of time are as follows:

Grinding Time (*n* minutes)	Ratio Al/Si after *n* minutes' grinding	Ratio of (Ratio Al/Si) at 0 minutes to (Ratio Al/Si) at *n* minutes
0 minutes	0.76	1.00
1 minute	0.62	1.23
4 minutes	0.56	1.36
10 minutes	0.36	2.11

This demonstrates that at short grinding times (i.e. incomplete grinding) the ratio measured is within the scope of the claims. However as grinding time increases and the sample is more completely broken up (as corroborated by the electron micrographical evidence) so that the value determined tends to the "true", i.e. correct value, the ratio determined is outside the scope of the claims. This demonstrates that the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support in these particles lies outside the range defined in the independent claims of the main request.

6.3.6 Therefore neither the evidence of the explicit, literal disclosure of D3a nor the evidence relating to the implicit disclosure thereof, submitted in the form of an experimental report repeating Catalyst X of D3a and analysing the product so obtained supports the position of the appellant that the catalyst system disclosed in D3a Catalyst X exhibits the ratio of the ratio of aluminium to support element outside the support to the ratio of aluminium to support element inside the support as defined in the independent claims according to the main request.

6.3.7 Accordingly, the subject matter claimed according to the main request is novel with respect to D3a.

6.4 D24

6.4.1 This document is derived from the same priority document as D1 and therefore is also concerned with providing a catalyst system comprising an inert support, a transition metal compound (which in one embodiment can be a metallocene) and an alumoxane. It is stated that the supported metallocene/alumoxane catalyst is highly active at low ratios of aluminium to the transition metal, leading to a low level of catalyst metal residue in the polyolefin product, i.e. low ash content (page 1 lines 10 to 13). The teachings concerning the preparation of the catalyst system and provenance of the alumoxane (D24 page 8, line 15 to page 9, line 7) are identical to those noted above for D1, as is the reference to "Preparation Method 3" discussed in paragraphs 6.2.1 and 6.2.2 above.

6.4.2 According to example 1 of D24, dried silica is slurried with a commercial solution of methylalumoxane in toluene and stirred for 0.5 hours. No specific handling or treatment of the methylalumoxane solution is disclosed. The slurry is filtered and washed five times with pentane and dried in vacuo. The transition metal compound ($Me_2Si(Me_4C_5)(N\text{-}t\text{-}Bu)ZrCl_2$) is combined with 1.0 M methylalumoxane in toluene and stirred for 5 minutes. Then the treated silica is added. After stirring for a further 5 minutes, the toluene is removed via vacuum and the catalyst recovered. The catalyst is then used in the polymerization of ethylene.

6.4.3 D24 does not report either in the general description or in the examples the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support. Therefore the explicit teaching of D24 contains no disclosure that said ratio is within the range required by the independent claims of the main request.

6.4.4 Accordingly, the disclosure of D24 does not anticipate the subject matter of the independent claims of the main request.

6.5 D25

6.5.1 D25 relates according to claim 1 to a solid catalyst for polymerizing an olefin prepared from an organometallic compound, a fine particle support ("carrier" in the terminology of D25), an alumoxane, a compound of group IVb transition metal and an olefin polymer produced in a preliminary polymerisation. According to the description, the solid catalyst can polymerize an olefin at a high polymerizing activity and produce a high molecular weight olefin polymer, even when the alumoxane content of the catalyst is reduced. Further, it is taught that the solid catalyst can produce a spherical olefin polymer having a good particle size distribution as well as excellent bulk density in slurry or particularly gas-phase polymerization. It is further taught that the solid catalyst of D25 can polymerize an olefin with high polymerizing activity and produce an olefin polymer having narrow molecular weight distribution and, in the case of copolymerization, also narrow composition distribution (page 2, lines 4 to 13). According to page 5, line 59 to page 6, line 12 the preferred process for producing the alumoxane is by preparing a hydrocarbon medium suspension of an absorbed-water-containing compound such as water-containing silica and adding a trialkylaluminium into the suspension for reaction. The description of D25 contains no information about treatment or handling of the so obtained alumoxane.

The catalyst can be prepared by one of three methods corresponding to different orders of addition of the alumoxane and organometallic components to the fine particle support (page 9, lines 3 to 15), corresponding to the methods in D1 discussed in paragraph 6.2.1 above.

The preferable method is to first treat the fine particle support with the organometallic compound and further treat the support with alumoxane, this method

being stated to produce a catalyst having a particularly superior polymerization activity (page 9, lines 14 to 15).
The contacting treatments are carried out with the fine particle support suspended in an inert solvent into which solutions of the other components are added (page 9, lines 35 to 39). In all three methods disclosed the alumoxane is combined with the support by a process involving precipitation of alumoxane onto the support. Depending on the method adopted to prepare the catalyst (see above), the support may have been previously treated with the organometallic component of the catalyst system. The precipitation is effected by contacting the alumoxane solution with a solvent in which the alumoxane is insoluble or hardly soluble (page 9, line 50 to page 11, line 23). One way in which this can be realised is explained at page 10, lines 3 to 8. Specifically, a suspension containing the solvent used for dissolving the alumoxane and the fine particle support, optionally pretreated with the organometallic compound is contacted with the alumoxane or alumoxane solution to generate a suspension containing both alumoxane and the fine particle support. A solvent in which the alumoxane is insoluble or hardly soluble is added to precipitate the alumoxane onto the support.
A further step required to prepare the solid catalyst of D25 is to carry out a prepolymerisation of the catalyst (cf claim 1). According to page 11, lines 45 and 46 the transition metal compound is supported on the carrier by the preliminary polymerization.

6.5.2 The method in which in the first step alumoxane is placed on the support is demonstrated in example 1. According to this example, alumoxane is prepared by dissolving $Al_2(SO_4)_3.14H_2O$ in toluene and adding to this solution trimethylaluminium diluted with toluene. After reaction (10 hours) the solid is recovered by filtration and toluene removed from the filtrate yielding white solid alumoxane. From this alumoxane is prepared a solution in toluene (1.25 mol/l Al solution). A nitrogen purged 100 ml flask is charged with 2.7g calcined silica (F-952, Fuji Devison K.K. calcined at 700°C for 6 hours) and 15 ml toluene to produce a suspension. To the suspension, 13.5 mol of a toluene solution of triisobutyl aluminium (Al, 1 mol/l) is added and agitated at 23°C for 30 minutes. The aforementioned alumoxane solution (18 ml) is added followed by agitation for a further 10 minutes at 23°C. 30 ml of n-decane is added to this solution and the toluene removed at 50°C and 2mmHg. Removal of toluene requiring 85 minutes. To the so obtained fine particle solid suspended in n-decane, 4.5 ml of a toluene solution of bis(cyclopentadienyl)zirconium dichloride (Zr, 0.04 mol/l) is added, and the toluene again removed (50°C, 2 mmHg), removal of toluene requiring 45 minutes. Following addition to this suspension of additional n-decane (100 ml), ethylene gas at atmospheric pressure is continually introduced to effect a preliminary polymerisation (2 hours, 35°C). N-decane is removed by filtration and the reaction mixture washed three times with n-hexane at 60°C.

6.5.3 Neither the general discussion nor the examples of D25 disclose explicitly the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support.

6.5.4 An experimental report was submitted by appellant Basell Polyolefine GmbH (see section IX, first sentence and section IX, paragraph (b) above) according to which it was alleged, example 1 of D25 had been repeated, and the ratio of the ratios of aluminium to silicon outside the support/inside the support of the resulting solid catalyst determined to fall within the scope of the independent claims of the main request.

(a) As silica Sylopol 952 from Grace Davison was employed, which was submitted to be identical to that employed in D25, which submission was not disputed (see section XXI.f.iv above).

(b) According to the experimental report, the silica (7.2 g) was calcined at 700°C for 6 hours, placed in a 250 ml argon flushed flask and suspended in 40 ml of toluene. Subsequently, 36 ml of a 1 mol/l solution of triisobutyl aluminium in toluene was added and the suspension stirred at 23°C for 30 minutes. Then 45 ml of a 1.24 mol/l solution of methyl alumoxane in toluene (prepared from 13 ml of a 30 wt% commercial solution of methyl alumoxane in toluene and 32 ml toluene) was added and the reaction mixture stirred for a further 10 minutes at 23°C. 50 ml n-decane was added and the toluene removed in vacuum at 50°C, this process taking 85 minutes. To the resulting suspension was added 32 ml of a 0.04 mol/l solution of bis(cyclopentadienyl) zirconium dichloride in toluene. The toluene was removed under vacuum for 45 minutes. The product was filtered and the filtrate washed four times with 50 ml portions of hexane. The catalyst was then dried for 3 hours under vacuum at room temperature.

(c) A comparison of the experimental report and the disclosure of example 1 of D25 however reveals the following discrepancies:
  (i) Compared to D25 the quantities of silica, triisobutyl aluminium and toluene in the repetition were increased by a factor of 2.66.
  (ii) The amount of alumoxane was increased by a factor of 2.48.
  (iii) The amount of n-decane added (50 ml) was increased by a factor of 1.6.
  (iv) The amount of metallocene was increased by a factor of 7.1.
  (v) The washing conditions employed were not identical. According to D25 example 1 the product is washed three times with non-specified quantities of hexane at 60°C whereas according to the submitted experimental report the washing was effected with four 50 ml portions of hexane at an unspecified temperature.
  (vi) According to D25 the product, without isolation from suspension in decane and hence prior to the washing step referred to in the preceding paragraph, is subjected to prepolymerisation with ethylene. This step was however omitted in the repetition and the product isolated without any prepolymerisation step.

(d) The Appellant acknowledged the noted discrepancies, but was unable to offer any justification therefor.

(e) Due to the discrepancies between the disclosure of D25, example 1 and the procedure as set out in the experimental report, it must be concluded that the cited example of D25 has not in fact been reproduced with the consequence that the properties determined for the product of the experimental report do not correspond to those of the product of the process disclosed in example 1 of D25.

6.5.5 Therefore neither the evidence of the explicit, literal disclosure of D25, nor the evidence relating to the implicit disclosure thereof, submitted in the form of an experimental report allegedly repeating example 1 of D25 supports the position of the appellant that the catalyst system disclosed in example 1 of D25 exhibits the ratio of the ratio of aluminium to silicon outside the support to the ratio of aluminium to silicon inside the support as specified in the independent claims of the main request.

6.5.6 Accordingly the subject matter claimed according to the main request is novel with respect to D25.

6.6 Since it has not been shown that the ratio of the ratio of aluminium to the support element outside the support to the ratio of aluminium to support element inside the support of 2.0 or less is disclosed, either explicitly or implicitly in the prior art, it is concluded that the subject matter of claims 1 to 9 of the main request is novel.

7. *Inventive step - main request*

7.1 The technical problem

As discussed in section 4.1 above and set out in paragraphs [0001] and [0005] of the patent in suit, the technical problem which the patent in suit sets out to solve is to provide catalyst systems capable of polymerizing olefinic monomers into granules having a narrow molecular weight distribution, low ash content and superior bulk density and controlled particle size distribution and provide sustained operability under reactor conditions (no fouling of the reactor).
The board is satisfied, based on the evidence of example 1 read in the light of paragraphs [0056] to [0058] of the patent in suit, that this problem has effectively been solved.

7.2 The claimed solution to the technical problem

According to the independent claims of the main request, this technical problem is solved by provision of a composition comprising alumoxane, or an alumoxane-transition metal complex and porous support (claims 1, 2) or a composition prepared by combining porous support material and alumoxane wherein greater than 90% of the alumoxane is composed of particles having an average diameter of 5 nm or less (claim 3) wherein the ratio of the ratio of aluminium to the support element outside the support to the ratio of aluminium to support element inside the support is 2.0 or less.

7.3 The closest prior art

As is apparent from the discussion of novelty, all of the documents cited in the procedure fail to disclose the required ratio of the ratio of aluminium to support element outside the support to the ratio of aluminium to support element inside the support.
D1, which during the opposition procedure was considered to represent the closest prior art relates, like the patent, to the provision of catalyst systems capable of producing polymers with higher molecular weight and narrow molecular weight distributions (D1, column 3, lines 24 to 35). A further aim is that the catalyst should make it possible to reduce metal residues (i.e. ash) in the final product. According to column 2, lines 59 to 66 of D1 a further aim is to provide control of the particle size distribution of the polymeric product so to reduce reactor fouling. Accordingly D1 addresses many of the same problems as the patent in suit.
None of the other documents cited, in particular D3a, D24 or D25 is more relevant than D1 since, although these all refer to provision of supported catalysts based on alumoxane and a metallocene, and have, like D1, been alleged to anticipate the subject matter of the main request, there is no disclosure of the required distribution of alumoxane within the support, as expressed by the ratio in the independent claims of the main request (see discussion of novelty in paragraph 6 above).

7.4 Obviousness of the claimed solution

As set out in the patent in suit, the claimed solution was based on the realisation that alumoxane exists in a bimodal distribution of particle sizes and that by allowing the solution to settle, it was possible selectively to increase the content of the smaller particles. These can enter the pores of the support substance, allowing preferential location of the alumoxane within the internal pore structure of the support.

7.4.1 Firstly, none of the prior art documents cited by the appellants provides any teaching which would lead the skilled person to seek such preferential concentration of alumoxane within the pores of the support for any reason. The document D5 relied upon by the appellants (see section IX.c above) does not relate to supported alumoxane systems on an inert support, but to a different type of catalyst (Ziegler-Natta). Further, the statement relied upon by the appellants ("homogeneous distribution of active centres") does not unambiguously disclose that reactive centres should be distributed evenly over the surface and throughout the bulk of the support particles as required by the ratio specified in the independent claims of the main request. Similarly the document D7 relied upon by the appellants (section IX.c above) does not teach that gels should be removed, but teaches that residual solvent poses a problem, as submitted by the respondent at the oral proceedings (section XXI.g.viii).

7.4.2 Secondly, it is also the case that none of the documents cited by the appellants provide any means that would allow such a preferential concentration of alumoxane within the support, once sought, to be achieved. There is no recognition in the prior art of the existence of a bimodal particle size distribution in alumoxane solutions, or any disclosure of conditions that would, nevertheless, give rise to preferential concentration of the smaller particles, e.g. by settling. Although some documents, e.g. D3a do disclose a step of settling and decantation in the preparation of alumoxane, it is apparent that the purpose of this step is not to preferentially augment the content of particles of a particular size, but to eliminate insoluble by-products of the alumoxane synthesis reaction.

7.4.3 Thirdly, even if such preferential concentration of small alumoxane particles was sought to be achieved, the evidence provided by the respondent in the form of D26, D27 and the statements submitted with the letter of 7 February 2006 further indicates that the necessary steps of selective concentration and decantation of alumoxane solution would in fact not have been possible with commercially available materials at the priority date of the patent in suit due to the manner in which they were delivered (sealed, opaque metal cylinder, the material being removed via a dip tube located close to the bottom thereof, or stored in a stirred tank).

7.4.4 The use of the so-called "daily bottle" practice referred to by the appellants at the oral proceedings (see section XXI.g.ii) has been supported by no documentary evidence, and it has been disputed by the respondent that such practice was in fact commonly employed. Accordingly the reference to this practice is dismissed as an unsupported allegation.

7.5 Thus there is no teaching in the prior art which would have guided the skilled person either to seek to attain a specific distribution of alumoxane throughout the support element, or to seek to control the relative amounts of alumoxane inside and outside the support. There is also no indication in the prior art how such a distribution, once defined, may be obtained. Further the steps necessary to attain such a preferential concentration of small alumoxane particles would not even have been possible with the commercially available alumoxane solutions at the priority date of the patent in suit.

7.6 It is thus apparent that the technical contribution of the patent in suit resides in identifying a problem of which there was hitherto no recognition in the prior art, namely the need, in metallocene catalysis systems, to provide supports having a specific distribution of alumoxane on the surface and within the bulk thereof, as indicated by the ratio defined in the independent claims of the main request. As held in T 2/83, relating to so-called "problem inventions" (OJ EPO 1984, 265) the discovery of a hitherto unrecognised problem may in certain circumstances give rise to patentable subject matter. According to that decision this was the case even in the situation that the solution claimed retrospectively was trivial and obvious.
In the present case, not only has a hitherto unrecognised problem been identified, which according to the cited decision T 2/83 could in itself suffice to substantiate an inventive step, but for the reasons

explained in paragraphs 7.4.3 and 7.4.4 the technical measures necessary to provide the solution claimed thereto would not have been obvious or trivial even in retrospect.

7.7 Accordingly it is concluded that the subject matter claimed according to the main request is not obvious in the light of the prior art and is founded on an inventive step.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent on the basis of claims 1 to 9 of the main request as filed with the letter dated 7 February 2006 and after any necessary consequential amendment of the description.

The Registrar:	The Chairman:
E. Görgmair	R. Young