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Datasheet for the decision of 11 July 2018

Case Number: T 1399/13 - 3.3.03

Application Number: 02796654.8

Publication Number: 1456257

IPC: C08F14/06, C08F4/32, C08F6/00

Language of the proceedings: ΕN

Title of invention:

ADDITION OF ORGANIC INITIATORS DURING THE PRESSURE DROP IN VINYL CHLORIDE MONOMER POLYMERIZATION

Patent Proprietor:

Akzo Nobel N.V.

Opponents:

SOLVAY (SOCIETE ANONYME) ARKEMA FRANCE

Relevant legal provisions:

EPC Art. 87(1), 54, 83, 84, 123(2) RPBA Art. 12(2), 12(4), 13(1)

Keyword:

Priority - Main request (no)

Novelty - Main request (no) - Auxiliary request 1 (yes)

Sufficiency of disclosure - Auxiliary request 1 (yes)

Amendments and clarity of disclaimer - Auxiliary request 1 (yes)

Decisions cited:

T 0665/00, G 0001/03, T 2130/11, G 0001/15



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Case Number: T 1399/13 - 3.3.03

DECISION
of Technical Board of Appeal 3.3.03
of 11 July 2018

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Decision under appeal: Interlocutory decision of the Opposition

Division of the European Patent Office posted on 26 April 2013 concerning maintenance of the European Patent No. 1456257 in amended form.

Composition of the Board:

Chairman D. Semino Members: D. Marquis

R. Cramer

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Summary of Facts and Submissions

I. The appeal lies with the interlocutory decision of the opposition division posted on 26 April 2013 concerning maintenance of the European patent No. 1456257 in amended form.

- II. The European patent was granted on the basis of 5 claims, claim 1 reading as follows:
 - "1. Process wherein one or more initiators are used to polymerize a mixture containing one or more monomers of which one is vinyl chloride monomer, wherein at least part of the initiators is added to the polymerization mixture at the reaction temperature when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride monomer, with the proviso that the polymer formed is not subject to radio frequency dielectric heating in the presence of additional organic initiator."
- III. Two notices of opposition were filed in which revocation of the patent in its entirety was requested.
- IV. During opposition proceedings, the following documents inter alia were cited:

D1: US 342434 (priority document)

D6: WO 00/17245
D8: EP 1495057 B1

D9: "Initiators for High Polymers" from Akzo Nobel D18: Declaration of Mr Bodart dated 23 November 2012

V. The decision of the opposition division concerning maintenance of the patent in suit in amended form was announced at the oral proceedings on 24 January 2013.

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The decision was based on a main request filed with letter of 26 October 2010, on a first auxiliary request filed with letter of 20 November 2012 and on a second auxiliary request filed during the oral proceedings before the opposition division.

Claim 1 of the main request read as follows:

"1. Process wherein one or more initiators are used to polymerize a mixture containing one or more monomers of which one is vinyl chloride monomer, wherein at least part of the initiators is added to the polymerization mixture at the reaction temperature when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride monomer, with the proviso that the polymer formed is not subject to radio frequency dielectric heating in the presence of additional organic initiator and wherein the initiator added when the pressure in the polymerization reactor is dropping is an organic peroxide having a half life of from 0.0001 hour to 1 hour at the polymerization temperature."

Claim 1 of auxiliary request 1 differed from claim 1 of the main request in that the proviso that was already part of claim 1 was moved towards the end of the claim and was followed by an additional proviso disclaiming examples 1 and 2 and the comparison examples of D8.

Claim 1 of auxiliary request 2 differed from claim 1 of the auxiliary request 1 in that the additional proviso in claim 1 was formulated to reflect the wording of examples 1 and 2 and the comparison examples of D8 more precisely.

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The decision under appeal as far as relevant to the present decision can be summarised as follows:

- (a) The main request was not entitled to the priority date of 21 December 2001 because document D1 from which priority was claimed did not disclose the features that (1) "at least part of the initiators is added to the polymerization mixture at the reaction temperature when the pressure in the polymerization is dropping", wherein the said initiator was "an organic peroxide having a half life of from 0.0001 hour to 1 hour at the polymerization temperature", and (2) the proviso that "the polymer formed is not subjected to radio frequency dielectric heating in the presence of additional organic initiator". Consequently, D8 was prior art according to Article 54(3) EPC and example 1 of that document anticipated claim 1 of the main request.
- (b) The first auxiliary request contained an additional independent claim which contravened Rule 80 EPC. Also, while the application as originally filed provided a basis for the amendment in claim 1 regarding the addition of the specific initiator when the pressure in the polymerization reactor dropped, claim 1 disclaimed more than was disclosed in the examples of D8. Thus, the disclaimer of claim 1 did not fulfil the criteria set out in G 1/03 (OJ EPO 2004, 413). Also, the application as originally filed did not provide a basis for the feature relating to the addition of the surfactant in claim 3 of the first auxiliary request. On that basis the requirements of Article 13(2) EPC were not met.

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- (c) As to the second auxiliary request, it met the requirements of Article 123(2) EPC and was sufficiently disclosed as the patent in suit contained sufficient guidance as far as the features "when the pressure in the polymerization mixture at the reaction temperature is dropping" and "during the first part of the polymerization reaction" were concerned.
- (d) Since D6 did not clearly and unambiguously disclose that the additional initiator was added to the polymerization mixture at the reaction temperature when the pressure in the polymerization was dropping, claim 1 was novel over D6. D18, which had been filed late in opposition proceedings, was not admitted as it was not deemed to be relevant.
- (e) The closest prior art was D6. The starting point in D6 was example F, which did not disclose an addition of initiator to the polymerization mixture when the pressure in the polymerization reactor was dropping due to the depletion of the vinyl chloride monomer. The supplementary examples filed by the proprietor with letter of 26 October 2010 showed that that feature led to a reduction of the total polymerization time, which could not be expected from the documents on file. The second auxiliary request was therefore inventive.
- VI. The proprietor (appellant) lodged an appeal against that decision. The statement setting out the grounds of appeal was based on a main request and auxiliary requests 1 to 4. The main request corresponded to the main request filed on 26 October 2010. Auxiliary request 1, which is relevant to the present decision, contained three claims, claim 1 being the sole

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independent claim and reading (deletion in strikethrough and additions in bold compared to the main request):

- "1. Process wherein one or more initiators are used to polymerize a mixture containing one or more monomers of which one is vinyl chloride monomer, wherein at least part of the initiators is added to the polymerization mixture at the reaction temperature when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride monomer, with the proviso that the polymer formed is not subject to radio frequency dielectric heating in the presence of additional organic initiator, and wherein the initiator added when the pressure in the polymerization reactor is dropping is an organic peroxide having a half life of from 0.0001 hour to 1 hour at the polymerization temperature, with the proviso that the polymer formed is not subject to radio frequency dielectric heating in the presence of additional organic initiator, and with the further proviso that a process in which vinyl chloride monomer is polymerized in suspension at 57°C in the presence of 1,000 ppm of polyvinyl alcohol by either (i) metering 330 ppm diisobutanoyl peroxide over 12 minutes and subsequently 670 ppm diisobutanoyl peroxide over 220 or 245 minutes or (ii) metering 480 ppm 1-(2-ethylhexanoylperoxy)-1,3-dimethylbutyl-1peroxypivalate over 15 minutes and subsequently 1,120 ppm 1-(2-ethylhexanoylperoxy)-1,3dimethylbutyl-1-peroxypivalate over 120 minutes, all amounts based on vinyl chloride monomer and all peroxides metered as 0.8% w/w methanol solution, is excluded."
- VII. In their replies to the statement of grounds of appeal, opponents 1 and 2 (respondents 1 and 2) requested that

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the appeal be dismissed. Respondent 1 refiled document D18 and filed the following additional documents:

D8': WO 03/087168 A1

D19: declaration of Mr Bodart dated 3 March 2014

D20: Letter of the appellant dated 11 March 2004 to the examining division

D21: Letter of the appellant in response to the official action of the USPTO dated 1 March 2010

D22: Handbook, Ch.E. Wilkes, J.W. Summers, Ch.A. Daniels, Carl Hanser Verlag, Munich, 2005, pages XIII to XIV, 57 to 80 and 93 to 94

D23: Encyclopedia of PVC, 2d Edition, edited by L.I. Naas, C.A. Heiberger, Volume I: Resin Manufacture and Properties, 1986, pages 77 to 82,

D24: R.N. Zaripov et al., Russian Chemical Bulletin,

Vol. 46, n°5, May, 1997, pages 895 to 897

D25: Letter of the appellant dated 4 August 2011 to the opposition division during the opposition of patent EP 1124866 B1

D26: Brochure Peroxid-Chemie GmbH, 09/83

 ${\tt D27:}$ Polymer Handbook, Third Edition, J. Brandrup and

E.H. Immergut, John Wiley & Sons, 1989, pages II/1, II/
2, II/57-65

- VIII. In a communication sent in preparation of oral proceedings, the Board summarised the points to be dealt with at the oral proceedings and provided a preliminary view on the disputed issues.
- IX. With letter of 8 May 2018, the appellant filed additional arguments in support of novelty and submitted documents D28 (Handbook of Chemistry and Physics, page 6-2) and D29 (datasheet Vinyl Chloride, 1999 (https://cameochemicals.noaa.gov/chris/VCM.pdf)).

 A new request was filed as auxiliary request 3 and the

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former auxiliary requests 3 and 4 were renumbered as auxiliary requests 4 and 5.

- X. By letter of 11 June 2018, respondent 1 filed further simulations of the polymerization process of example 2 of D6. A further declaration of Mr Bodart, the technical expert responsible for these simulations, was filed with letter of 19 June 2018.
- XI. Oral proceedings were held on 11 July 2018 in the absence of respondent 2 as announced with letter of 5 June 2018.
- XII. The arguments provided by the appellant, as far as relevant to the present decision, can be summarised as follows:

Main request

Priority claim and novelty in view of D8

- (a) Example 1 of document D1, from which priority was claimed, and example 1 of document D8 contained similar disclosures wherein vinyl chloride monomer (VCM) was polymerized using an organic peroxide with a half life at the polymerization temperature in the range of claim 1, and the organic peroxide was dosed to the reaction mixture for a period of time exceeding the constant pressure time.
- (b) The situation of the present case was similar to that of case T 665/00 in which a Board concluded that the prior art product did not destroy the novelty of a claim because said specific product was covered by the priority. Although in the present case the reaction conditions applied in the

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examples of D1 and D8 were not exactly the same in all respects, the features that made them fall within the scope of claim 1 were the same: polymerization of VCM using the same general procedure, one organic peroxide, said peroxide having a half life at the polymerization temperature in the range 0.0001-1 hour and being added to the reaction mixture for a period of time exceeding the constant pressure time.

(c) D1 was the first application disclosing an example according to claim 1 of the main request. Hence, the first European application disclosing a VCM polymerization process according to claim 1 was the one leading to the patent in suit, via its priority application D1. D8 disclosed this subject-matter in the same manner, but post-dated D1. Hence, the patent in suit validly claimed priority from D1 and D8 was not prior art.

Auxiliary request 1

Admittance

(d) No arguments were provided.

Sufficiency of disclosure

(e) No arguments were provided.

Admittance of D18, D19, D20, D21 and the simulations provided with the letter of 11 June 2018

(f) D18 had not been admitted into the procedure by the opposition division because it was filed late, it was not filed in response to a claim amendment and

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it was not *prima facie* relevant. Also, the computer simulations of D18 were considered insufficient to demonstrate the alleged implicit disclosure of D6 in a clear and unambiguous manner.

(g) Simulations used a simplification of the real world and generally assumed a homogenous distribution of all ingredients during the entire process which did not take the migration effect happening in the case of the patent in suit into account. Furthermore, the simulations were based on assumptions regarding the peroxide activity which had not been shown to correspond to D6. The simulations had also been performed with a different water to VCM ratio than that used in D6. The simulations provided with D18 and D19 and the letter of 11 June 2018 as well as the supporting documents D20 and D21 should therefore not be admitted into the proceedings.

Disclaimer

- (h) Auxiliary request 1 provided a clear and concise disclaimer that satisfied the criterion set out in T 2130/11, that is the disclaimer did not lead to an arbitrary reshaping of the claim. Claim 1 disclaimed the processes in terms of the polymerization temperature, the presence of polyvinyl alcohol, the initiators used, the initiator amounts and dosing times. The disclaimer in this request thus contained all details related to the essence of the invention, namely the manner of dosing initiator and no arbitrary choices were made.
- (i) All other details disclosed in the experimental section of D8 had no effect on the addition of

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initiator during the pressure drop and were therefore not needed. Claim 1 of auxiliary request 1 provided the appropriate balance between the legitimate interest of the proprietor of obtaining adequate protection on the one hand and the interest of the public in understanding what was and what was not protected on the other hand. The disclaimer therefore met the requirements of G 1/03 and was allowable.

(j) The question of when the heating phase started in the examples of D8 was not relevant to the clarity of the disclaimer.

Novelty in view of D6

- (k) The simulations made available were incapable of evidencing the time at which a pressure drop occurred in the experiments of D6. One important parameter in the polymerization process of the patent in suit and D6 was the time needed for the organic peroxide initiator to migrate from the droplet of solvent to the VCM droplets. That had not been taken into account in the simulations provided by the respondent. Also, the simulations assumed that the activity of the initiator was equal in all phases, which in reality was not the case.
- (1) According to the graphs of the computer simulations of D18, the reaction mixture was heated by the jacket until the reactor temperature reached a temperature of about 38°C. From this point on, the temperature inside the reactor (T Reactor) was higher than that of the jacket (T Jacket in and T Jacket out), which meant that from that moment

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on, the reactor was heated by the exothermal polymerization. The jacket acted in this phase as a cooling medium for the reactor. However, in order to heat the reaction mixture of the examples of D6 from 38°C to the maximum temperature of 57°C by the heat produced by the reaction a VCM conversion of 20% was necessary. This meant that, if the figures of D18 were a true reflection of the examples of D6, a 20% VCM conversion was necessary in the period during which the temperature rose from 38°C to 57°C. The simulations of examples 2 and 3 in D18, however, indicated a significantly lower conversion during this period, corresponding to approximately 6%. A possible explanation was that the simulation did not take account of the correct water to VCM ratio (4:1) actually applied in the examples of D6. Instead, given the simulated conversion during the indicated period, the simulation seemed to have applied a water to VCM ratio of about 1:1.

(m) In systems with a higher water to VCM ratio - i.e. more dilute systems - the reaction was slower and the pressure drop would appear at a later stage, simply because the concentration of VCM droplets was lower and it took more time for a peroxide to migrate to a VCM droplet. This meant that more initiator decomposed before it reacted with VCM. Furthermore, part of the initiator dissolved in water, in which it decomposed quicker than in apolar VCM. Hence, the more water, the larger the loss of initiator, the slower the reaction, and the later the pressure drop happened in the course of the polymerization. Since the temperature T Jacket was not provided in the simulation of D19, it could not be determined whether that simulation used the

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same water to VCM ratio as that of the corresponding example of D6.

- (n) The use of an incorrect water to VCM ratio in the simulations also explained why a 90% conversion of the monomer was obtained in 175 min (2 h and 55 min) in the case of the simulation of example 3, whereas more than 4 h were needed in the polymerization of the corresponding example of D6. Thus the simulations provided were not accurate representations of the polymerization process of D6.
- (o) Regarding the simulation of example 2 in D18, the pressure drop was clearly indicated to start after 120 min. Since D6 disclosed that the metering of the initiator was carried out within 2 h, it was clear that in the case of example 2 of D6, no initiator had been added when the pressure in the reactor was dropping. Therefore the simulations provided in D18 did not show that D6 implicitly disclosed the missing feature.
- (p) The reference to the definition of the pressure drop in paragraph 9 of the patent in suit was not relevant to the interpretation of claim 1 since a different wording was used. Claim 1 clearly referred to the point in time at which the pressure was dropping, which was not the wording of paragraph 9, describing thus a different situation in the polymerization process. Claim 1 of the auxiliary request 1 was therefore novel over D6.
- XIII. The arguments of the respondents, as far as relevant to the present decision, can be summarised as follows:

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Main request

Priority claim and novelty in view of D8

- (a) Example 1 of D1 was not identical to the four examples of D8 and could not form the basis of a priority claim for claim 1 of the main request. Also, D1 differed substantially from the claimed subject matter. In particular, D1 did not disclose the proviso that the polymer formed was not subject to radio frequency dielectric heating nor did it disclose the half life of from 0.0001 hour 1 hour at the polymerization temperature.
- (b) Decision T 665/00 did not apply to the present case and the decision G 2/98 required for a priority claim to be valid that its subject matter be directly and unambiguously disclosed in the priority document. That was not the case for claim 1 of the main request.
- (c) The claim to priority of D1 was thus not valid. As a result, D8 took away the novelty of claim 1 of the main request. The same conclusion applied in view of document D8'.

Auxiliary request 1

Admittance

(d) The opposition division rejected the (then) first auxiliary request on the grounds that its claims 1 and 3 contravened Rule 80 EPC. Furthermore, the opposition division indicated that claim 1 of this request did not satisfy the requirements of Article 123(2) EPC. As this equally applied to

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current auxiliary request 1, this request should not be admitted into the proceedings.

Sufficiency of disclosure

(e) Reference was made to the arguments of lack of sufficiency of disclosure provided in the first instance. The claimed process was characterized by the half life of the organic peroxide added when the pressure in the reactor was dropping in the range of 0.0001 - 1 hour at the polymerization temperature. Neither claim 1 nor the patent in suit disclosed the solvent in which that half life had to be determined, nor the initial concentration of the solution for that determination, nor the precision of the measurement. These three parameters were necessary in order to perform the invention. D23, D24, D25, D26 and D27 showed that the value of the half life of an organic peroxide depended on the solvent used. In addition, D24, D26 and D27 showed that value of the half life of an organic peroxide depended on the initial concentration of the solution used for its determination. D24 and D26 further showed that the precision of the measurement was needed to characterize the half life. As a result, the claimed subject matter was not sufficiently disclosed.

Admittance of D18, D19, D20, D21 and the simulations provided with the letter of $11 \, \mathrm{June} \, 2018$

(f) The simulations provided in documents D19 and with the letter of 11 June 2018 as well as documents D20 and D21 were filed in response to the decision of the opposition division not admitting D18 into the

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proceedings. These documents showed that the simulations provided in D18 were adequate and relevant to the question of novelty of the claimed subject matter over D6. These documents should therefore be admitted into the proceedings.

(g) Simulations of polymerization processes as provided by respondent 1 were common in that field. It was established therewith that the simulations were an adequate representation of the process disclosed in the prior art.

Disclaimer

(h) The disclaimer in claim 1 of auxiliary request 1 removed more than necessary to restore novelty over D8. It did not indicate the volume of the reactor disclosed in D8, nor did it specify the mixing equipment used, the repeated purges with nitrogen, the volume of water used in the polymerization process and specific details regarding the polyvinyl alcohol and vinyl chloride used. Furthermore, the disclaimer did not contain details regarding the point in time at which the initiator was metered in the reactor whereas the metering of the initiator started at the end of the heating phase in example 1 of D8. Finally the temperature control of that reactor was not limited in the disclaimer whereas D8 concerned a cold-start process only. Documents D12 and D22 showed that these parameters were essential to the polymerization process. Since these parameters were not part of the disclaimer, claim 1 of auxiliary request 1 did not satisfy the criteria set out in decision G 1/03 in order to meet the requirements

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of Article 123(2) EPC.

- (i) In addition, claim 1 was defined by five disclaimers corresponding to the absence of radio frequency dielectric heating as well as the four examples of D8. That level of complexity added to claim 1 infringed Article 84 EPC.
- (j) Besides, the absence of a definition of when the metering started in the process of claim 1 further added to the lack of clarity of the claimed subject matter.

Novelty in view of D6

- (k) Examples 2 and 3 of D6 disclosed a process of polymerization of VCM involving the metering of an organic peroxide in the course of the polymerization process. The point in time at which the pressure in the reactor dropped was not disclosed in D6. Examples 2, 3 and B of D6 however formed the basis for simulations of the polymerization process provided in D18. The simulations established that an organic initiator had been added to the reactor at the time the pressure was dropping.
- (1) The simulations contained in D18 were based on the process parameters disclosed in D6 as well as on data concerning the reactants that were available in the art to the skilled person. It was clear from D18 that also after the temperature of the reactor had surpassed the temperature of the jacket, heat was not only provided to the reactants by the reaction medium itself but also by the jacket which continued to be heated at ca 40°C at least until

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the 60 min mark. Since less heat was needed thereafter, the temperature of the jacket was decreased as shown in the simulations of D18. These simulations were therefore an accurate representation of the polymerization process disclosed in the examples of D6.

- (m) The simulated process of the examples of D6 revealed that the pressure dropped after 120 min according to the parameters of example 2 or after 160 min according to the parameters of example 3. The initiator had therefore been added to the reactor also when the pressure was dropping in the examples of D6.
- (n) Another simulation of example F of D6 was carried out and provided in D19. It was shown therein that the pressure in the reactor was constant for a period of 200 min and the pressure dropped after approximately 4 h and 20 min after the start of the reaction. That length of time corresponded to the submission made by the appellant in D20 (205 min). That value was also corroborated by the polymerization profile disclosed on page 13 of D21 which revealed that the pressure in the reactor had dropped after a bit more than 4 h after the start of the reaction. D19 established therefore that the simulation of example F made by the respondent was in line with the values provided by the appellant. This also established that the simulations provided by the respondent, and in particular the simulations contained in D18, could be trusted.
- (o) The simulations provided in the letter of 11 June 2018, which actually concerned example 3 and not example 2 of D6, established that there was

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no significant effect of the water to VCM ratio on the pressure drop in the course of the reaction. The argument of the appellant based on the conversion of monomer of 20% obtained in D6 was thus not relevant.

- (p) Paragraph 9 of the patent in suit also clearly indicated that the relevant time for the pressure drop could be up to 30 min before an actual pressure drop was observed. The values of the pressure drop derivable from the simulations provided by the respondent therefore fell within the range of claim 1. Thus, claim 1 of auxiliary request 1 lacked novelty over D6.
- XIV. The appellant requested that the decision under appeal be set aside and the patent be maintained in amended form on the basis of the claims of the main request filed on 26 October 2010 before the opposition division, or on the basis of the claims of one of auxiliary requests 1 or 2 filed with the statement of grounds of appeal, or on the basis of the claims of auxiliary request 3 filed with letter of 8 May 2018, or on the basis of the claims of one of auxiliary requests 4 or 5, filed as auxiliary requests 3 and 4 with the statement of grounds of appeal.

Respondent 1 requested that the appeal be dismissed. It also requested that auxiliary requests 4 and 5 not be admitted into the proceedings, and if they were admitted, that the case be remitted to the department of first instance.

Respondent 2 requested that the appeal be dismissed and that the auxiliary requests filed with the statement of grounds of appeal not be admitted into the proceedings.

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Reasons for the Decision

Main request

- 1. Priority claim and novelty in view of D8
- 1.1 The opposition division concluded that the priority of D1 was not validly claimed and that D8, which was as a result a document according to Article 54(3) EPC, and in particular its example 1, was novelty destroying for claim 1 of the main request (points 3.1 and 3.2 of the reasons).
- 1.2 Example 1 of D8 discloses the polymerization of VCM under continuous metering of Trigonox 187 as organic peroxide initiator (paragraphs 23 and 25). Trigonox 187 is the organic peroxide initiator used in the examples of the patent in suit. It was not disputed that at 57°C, the polymerization temperature according to example 1 of D8, Trigonox 187 has a half life in the claimed range of 0.0001 h to 1 h. That can also be derived from the data provided on pages 3, 6 and 7 of D9. Furthermore, the description made of example 1 in D8 does not mention the use of a radio frequency dielectric heating. Thus the only question that remains in view of example 1 of D8 is whether it discloses that the initiator was added to the polymerization mixture at the reaction temperature when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride monomer.
- 1.2.1 According to Table 1 of D8, 330 ppm of peroxide are added to the reactor over 12 min and 670 ppm are subsequently added over 220 min (232 min of peroxide

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dosage in total). Also, 200 ppm of PVA are added to the reaction mixture before heating and 800 ppm are subsequently metered in 220 min together with the peroxide. D8 discloses that the constant pressure time is 195 min (3 h and 15 min) before the pressure dropped in the course of the polymerization. Since the peroxide initiator is added for a total period of 232 min, it is deduced that some organic peroxide initiator is added during the pressure drop phase in the reactor.

- 1.3 It is clear from the statement of grounds of appeal (point 3) that it was not contested that example 1 of D8 anticipates claim 1 of the main request. It was also not disputed in appeal that document D1, from which priority is claimed, does not disclose the whole breadth of the subject matter of claim 1 of the main request. Rather, the question that needs to be answered in appeal is whether the first priority date of the patent in suit (21 December 2001, the filing date of US 342434 P or D1) is valid at least for the subject matter of the main request that is also disclosed in example 1 of D8.
- 1.4 D1 (claim 1) relates to a process for polymerizing vinyl chloride monomer and optionally further monomers using one or more organic initiators, with at least part of said initiators being dosed to the polymerization mixture at the reaction temperature, characterized in that essentially all of the organic initiator used in the polymerization process has a half life of from 0.0001 h to 0.05 h at the polymerization temperature.
- 1.4.1 In the whole of D1, a pressure drop can only be derived from the description of the polymerization process of example 1. That example discloses the polymerization of

VCM at 68°C under continuous dosing of Trigonox 187 as organic peroxide initiator. According to Table 1 of D1, 500 ppm of peroxide are added over 30 min and subsequently 300 ppm/h during 2.5 h, meaning that in total, peroxide is added over 3 h in the course of the polymerization process. The constant pressure time during that polymerization is 2.5 h, after which the pressure drops (page 8, line 16). It can thus readily be deduced therefrom that the pressure drops (after 2.5 h) while peroxide is still being added to the reactor (up to 3 h).

1.4.2 Since example 1 belongs to D1 from which priority is claimed, it can form the basis for a priority claim for claim 1 of the main request but only inasmuch as the disclosure in example 1 of D1 is concerned. Indeed, in decision G 1/15 (OJ EPO 2017, A82) the Enlarged Board of Appeal has acknowledged the concept of partial priority and has decided that "entitlement to partial priority may not be refused for a claim encompassing alternative subject-matter by virtue of one or more generic expressions or otherwise (generic "OR"-claim) provided that said alternative subject matter has been disclosed for the first time, directly, or at least implicitly, unambiguously and in an enabling manner in the priority document" (see Order). In point 6.4 of decision G 1/15, the Enlarged Board of Appeal explained that, in assessing whether a subject-matter within a generic claim may enjoy partial priority, the first step is to determine the subject-matter disclosed in the priority document. The next step is to examine whether this subject-matter is encompassed by the claim of the application or patent claiming said priority. If the answer is yes, the claim is de facto conceptually divided into two parts, the first enjoying the priority corresponding to the invention disclosed in the

priority document, the second being the remaining part not enjoying this priority.

The process of example 1 of D1, which forms the basis 1.4.3 of the priority claim, differs however from that of example 1 of D8 in several instances. The first difference is in the temperature of the reactor which corresponds to the polymerization temperature. While that temperature is 68°C in D1 (page 7, line 27), it is set to 57°C in example 1 of D8 (page 6, line 29). That difference in temperature is significant for the polymerization process since it impacts the half life of the peroxide in the reaction medium and has some repercussions on the quantity of initiator ultimately dosed and the amount of heat released in the course of the polymerization process (D1: page 1, line 15 to page 2, line 8 and D8: page 2, lines 9-21). Furthermore, example 1 of D8 discloses a polymerization process in which the polyvinyl alcohol (PVA) added as a protective colloid is dosed with the initiator, which has an impact on the disclosed process since by metering both the initiator and the protective colloid, the efficiency of the initiator is improved, increasing the polymerization rate, shortening the polymerization times and increasing the pressure drop rate (paragraph 6 of D8). By contrast, the protective colloid is added to the reactor prior to the initiator in D1 (page 7, lines 21-23). These differences in the polymerization processes according to example 1 of D1 and example 1 of D8 are therefore significant for the course of the process and the phase of the pressure drop. The priority claim that can be derived from example 1 of D1 does therefore not correspond to the disclosure of example 1 of D8. As a result, example 1 of D8 is novelty destroying for claim 1 of the main

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request.

- 1.4.4 The present situation differs from case T 665/00 cited by the appellant in support of its position. In T 665/00 the subject matter of an alleged prior use which fell within the scope of the claims of the patent was described in the priority document. As the priority document did not necessarily describe all the subject matter claimed, the question was whether different elements of a patent application could have different priority dates. In the present situation, the question to be answered pertains instead to the differences between the document from which priority is claimed (in particular an example therein) and the examples of a document cited against the novelty of the claimed subject matter. The conclusion reached in T 665/00 does therefore not apply to the present case.
- 1.5 The same conclusion on novelty applies to claim 1 of the main request in view of example 2 and the comparison examples of D8 and in view of D8' which content is the same as that of D8. The main request does therefore not satisfy the requirements of Article 54 EPC.

Auxiliary request 1

1.6 Auxiliary request 1, which contains three claims, was filed by the appellant with the statement setting out the grounds of appeal, i.e. pursuant to the requirements of Article 12(1) and (2) RPBA. The claims of that request are based on the first auxiliary request filed with letter dated 20 November 2012 (claims 1 and 2) from which however independent claim 3 was deleted and replaced by a dependent claim 3 which was already part of the granted claims since it

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corresponds to its dependent claim 5. The presence of two independent claims was one of the reasons why the opposition division rejected the first auxiliary request (under Rule 80 EPC, contested decision, point 4.1). Auxiliary request 1 filed in appeal can therefore be seen as having been filed in an attempt to address the decision of the opposition division regarding Rule 80 EPC, while not substantially changing the subject matter of the request. The Board sees on this basis no reason to hold auxiliary request 1 filed with the statement setting out the grounds of appeal inadmissible pursuant to Article 12(4) RPBA.

- 2. Sufficiency of disclosure
- 2.1 In its reply to the statement of grounds of appeal, respondent 1 made an unspecific reference to arguments pertaining to an alleged sufficiency of disclosure submitted during opposition proceedings against unidentified claims.
- 2.1.1 In the communication pursuant to Article 15(1) RPBA, the Board drew the attention of the respondent to the requirements of Article 12(2) RPBA, requiring that the statement of grounds of appeal and the reply thereto had to contain a party's complete case. In particular, it was indicated that these submissions had to set out clearly and concisely the reasons why it was requested that the decision under appeal be reversed, amended or upheld, and had to specify expressly all the facts, arguments and evidence relied upon and that a mere reference to the written arguments, documents and facts that had been filed during first instance proceedings was not considered to constitute a party's complete case (see point 8.1 of the communication).

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- 2.1.2 Respondent 1 did not provide a clarification of its unspecific objection of lack sufficiency of disclosure submitted during opposition proceedings, neither in the later letter dated 11 June 2018, nor at the oral proceedings before the Board during which the respondent merely referred to the arguments provided in writing. Thus, neither the Board nor the other party was in a position to consider the objections of respondent 1 and establish whether they justified setting aside the decision of the opposition division on sufficiency of disclosure (contested decision, point 5.3). Under these circumstances, the Board finds that there is no reason to set aside the decision of the opposition division on the grounds of these unspecified arguments.
- 2.2 Additionally, lack of sufficiency of disclosure was raised against claim 1 by respondent 1 as neither claim 1 nor the description of the patent in suit mentioned the solvent in which the half life of the claimed initiator had to be measured. Also, the patent did not disclose the initial concentration of the solution used to perform the determination of the half life of the initiator and did not define the precision of the method of determination of the half life of the initiator.
- 2.2.1 With regards to the selection of the initiator, the patent in suit discloses a list of suitable compounds (paragraph 11) and additionally teaches that other organic peroxides having a half life ranging from 0.0001 to 1 hour at the polymerization temperature can be used. The description further indicates that the determination of whether a peroxide is suitable and has the required half life can be made by conventional thermal decomposition studies in monochlorobenzene, as

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was well-known in the art. In that respect, the description further refers to a brochure "Initiators for high polymers" available from Akzo Nobel (paragraph 12). The Board thus considers that the patent in suit provides a guidance to the skilled person as to the choice of initiators suitable for the claimed process.

- 2.2.2 Based on documents D23 to D27, which had been filed with the reply to the statement setting out the grounds of appeal, respondent 1 contended that the claimed subject matter was nevertheless not sufficiently disclosed.
- 2.2.3 D23 to D27 are all documents of the prior art showing that the half life of a given peroxide is a parameter whose value will depend on the conditions under which it is determined, notably the temperature, the nature of the solvent and the initial concentration of the peroxide in the solution. These documents show that the variation of the half life value as a function of these conditions is a phenomenon that was well established in the art at the priority date of the patent in suit and that the determination of the half life of a peroxide was also extensively known to a skilled person, including in chlorobenzene as a solvent as suggested in the patent in suit (D25: last line of the table and D27: table B).
- 2.2.4 The Board finds that even if the documents D23 to D27 were found to show that the value of the half life of the initiator indeed depends on factors like the solvent in which the measurement is performed or the initial concentration of the initiator in that solvent, the arguments of respondent 1 based on these documents solely relate to a lack of accuracy of the measurement,

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which is an objection of lack of clarity, and not to an objection of lack of sufficiency of disclosure, since it has not been shown that a skilled person would not be in the position to determine the half life value of a given peroxide under a given set of conditions and to perform the subject matter as claimed.

- 2.2.5 With respect to sufficiency of disclosure, the relevant question is whether the skilled person would have been able to perform the process according to claim 1 of the main request on the basis of the guidance found in the patent in suit. In the present case, the reply to the statement of grounds of appeal of the respondent 1 does not show how the possible discrepancies observed for the measurement of the half life of the organic peroxide initiator would have prevented the skilled person to conduct the process, all the more so as documents D9, D23, D26 and D27 actually show that the skilled person can find sufficient guidance in the general prior art to select an initiator among the wide range of known initiators additionally satisfying the requirement concerning its half life at any given temperature as set out in claim 1 of auxiliary request 1. No further arguments were provided by respondent 1 at the oral proceedings before the Board in that respect.
- 2.2.6 As to the precision of the method of determination of the half life of an organic peroxide, respondent 1 alleged but did not show how that would prevent a skilled person from performing the claimed subject matter. The question of whether it could be determined whether a specific value of half life would, given the uncertainty affecting the precision of any measurement, fall under the claimed subject matter or not, is a question of clarity and not a question of sufficiency

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of disclosure.

- 2.2.7 Therefore, there is no reason in the light of the submission made by respondent 1 to conclude that the subject matter of auxiliary request 1 lacks sufficiency of disclosure.
- 3. Disclaimer
- 3.1 Claim 1 of auxiliary request 1 is defined by a disclaimer referring to a step of radio frequency dielectric heating and a disclaimer meant to exclude four examples of D8. While the first disclaimer was present in claim 1 of the application as originally filed, the second disclaimer is intended to exclude the examples of D8, a document according to Article 54(3) EPC (see point 1. above).
- The disclaimer made in view of the examples of D8 in claim 1 of auxiliary request 1 essentially defines the type of polymerization (suspension), its temperature (57°C), the presence of a specific protective colloid (1000 ppm of polyvinyl alcohol) and the metering of specific peroxides in the course of the polymerization process which contains two parts, a first part (i) intended to disclaim example 1 as well as its comparison example reported in Table 1 of D8 and a second part (ii) intended to disclaim example 2 and its comparison example as reported in Table 2 of the same document.
- 3.2.1 The disclaimer in claim 1 of auxiliary request 1 is crafted around the main features of the polymerization process according to D8, a polymerization process whereby an organic initiator and a protective colloid are metered at the polymerization temperature (claim 1

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and paragraph 6). It is formulated so as to exclude the aspects shared by the examples first (polymerization type, monomer, reaction temperature, presence of polyvinyl alcohol), followed by the aspect relating to the metering of the specific peroxide initiators (parts (i) and (ii)) as disclosed in Tables 1 and 2. As such, that disclaimer is not only concise, it is also specific, since it ultimately discloses polymerization processes performed at one temperature only (57°C), involving one monomer (VCM) and concerning specific organic peroxides (diisobutanoyl peroxide and 1-(2-ethylhexanoylperoxy)-1,3-dimethylbutyl-1-peroxypivalate).

3.3 Respondent 1 considered that the disclaimer added to claim 1 in view of D8 did not fulfil the criteria set out in G 1/03 (OJ EPO 2004, 413) and was therefore not allowable under Article 123(2) EPC on the grounds that the formulation chosen excluded more than what was disclosed in the examples of D8 and therefore more than necessary to restore novelty. In that respect, D8 disclosed a number of parameters that had not been included in the disclaimer and that were found to be necessary to the definition of the disclaimer by respondent 1: the volume of the reactor disclosed in D8, the mixing equipment used in the reactor, the repeated purges with nitrogen, the volume of water used in the polymerization process, specifics regarding the polyvinyl alcohol and vinyl chloride used (pages 8 and 9 of the reply to the statement setting out the grounds of appeal of respondent 1). Moreover, claim 1 of auxiliary request 1 lacked clarity since the formulation of the disclaimer did not indicate the starting point of the metering of the organic peroxide that was disclaimed and the number of disclaimers in

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claim 1 rendered that claim unclear.

- 3.3.1 With regard to the conditions that the disclaimer meets the requirements of clarity and conciseness and does not remove more than necessary to restore novelty, both explicitly indicated in G 1/03 (see headnote, points 2.2 and 2.4), the Board concurs with the positions expressed in T 2130/11, points 2.9 and 2.10. In particular, the difficulty for a patent proprietor in formulating an allowable disclaimer cannot justify an exception in the application of Article 84 EPC which is not foreseen in the Convention, not even with regard to the condition on the allowability of a disclaimer requiring that a "disclaimer should not remove more than is necessary to restore novelty". Rather, that condition should be applied while taking into consideration its purpose, namely that the "necessity for a disclaimer is not an opportunity for the applicant to reshape his claims arbitrarily" (G 1/03, supra, point 3 in the reasons, second paragraph, last but one sentence).
- 3.3.2 The consequence of this, as clearly set out in T 2130/11, is that there are situations in which, while fulfilment of the condition taken in a strictly literal way would not be possible, a definition of the disclaimed subject-matter which satisfies the requirements of Article 84 EPC and fulfils the purpose of the condition (i.e. to avoid an arbitrary reshaping of the claims) may be achievable. In other words, a disclaimer removing more than strictly necessary to restore novelty would not be in contradiction with the spirit of G 1/03, if it were required to satisfy Article 84 EPC and it did not lead to an arbitrary reshaping of the claims.

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- 3.3.3 It is the view of the present Board that the formulation of the disclaimer with respect to D8 as present in claim 1 of auxiliary request 1 corresponds exactly to this situation in that even if the disclaimer in its present form may be seen as formally removing more than is strictly necessary to restore novelty over D8, it avoids clarity problems and cannot be held to be in contradiction with the spirit of G 1/03 as it does not lead to an arbitrary reshaping of the claims. In particular, a formulation of the disclaimer including all the data and process steps as disclosed in the experimental part of D8 (paragraph 23) would indeed deprive the claim of its conciseness and render its wording unclear (Article 84 EPC). Besides, while it is not disputed that the features of the process of the examples according to D8 that have not been included in the formulation of the disclaimer in claim 1 may ultimately have an effect on the course of the polymerization, it has not been shown how the absence of these features in claim 1 of auxiliary request 1 may lead to an arbitrary reshaping of the claims, nor has the Board any reason to consider that it is the case.
- 3.4 With respect to the presence of separate disclaimers in claim 1 of auxiliary request 1, the subject matter they concern, radio frequency dielectric heating and the examples of D8, was not shown to be related in any way or create any uncertainty as to what is disclaimed. The Board does therefore not find that their presence in claim 1 of auxiliary request 1 infringes the requirements of Article 84 EPC.
- 3.5 The Board concludes from the above that the disclaimers present in claim 1 of auxiliary request 1 do not contravene the requirements of Articles 123(2)

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and 84 EPC.

- 4. Admittance of D18, D19, D20, D21 and the simulations provided with the letter of 11 June 2018
- 4.1 D19, D20 and D21 were filed by respondent 1 with the reply to the statement setting out the grounds of appeal dated 5 March 2014, i.e. pursuant to the requirements of Article 12(1) and (2) RPBA.
- 4.1.1 These documents were cited in reply to the reasoning of the opposition division in the contested decision not admitting D18 into the proceedings on the grounds that that document had been filed late and that it had not been shown that the computer simulations it was based upon were a sufficient proof to demonstrate that the addition of initiator after the pressure drop was a feature that was implicitly disclosed in examples 2, 3 and B of D6 (contested decision, point 5.6).
- 4.1.2 D19 to D21 are meant to provide evidence that the computer simulations provided in D18 were reliable since some of the simulations performed therein were confirmed by actual measurements provided in D19 to D21. Documents D19 to D21 thus address one of the main grounds which led to D18 not being admitted and can be seen as a reasonable reaction to the decision. The Board sees therefore no reason to hold D19 to D21 inadmissible pursuant to Article 12(4) RPBA. Since the information contained in these documents is based on and refers to the data provided in D18, D18 is admitted into the proceedings as well.
- 4.2 The simulations additionally provided by respondent 1 with letter of 11 June 2018 are meant to establish the influence of the water to VCM ratio on the result of

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the simulations of D18. These additional simulations were filed on 11 June 2018, in response to the point concerning an alleged discrepancy between the water to VCM ratio used in D18 and in the examples of D6 which was first raised in appeal by the appellant in its letter dated 8 May 2018. These simulations were therefore filed by respondent 1 at the earliest opportunity after it became aware of the objection against D18. The Board therefore finds it appropriate to make use of its discretion under Article 13(1) RPBA by admitting the simulations into the proceedings.

- 5. Novelty in view of D6
- 5.1 Novelty of claim 1 of auxiliary request 1 was contested by the respondent in view of examples 2 and 3 of D6. These examples disclose a process wherein diisobutanoyl peroxide is used as an initiator in the polymerization of vinyl chloride (VCM) at 57°C and the whole of the initiator is dosed over time in the course of the polymerization reaction.
- 5.2 Table II of D6 reports that the dosing time of the initiator was 2h in the case of example 2 and 4h in example 3. It was undisputed that it could not be concluded from D6 alone whether the initiator had been added to the polymerization mixture when the pressure in the polymerization reactor was dropping due to the depletion of the vinyl chloride monomer.
- 5.3 Respondent 1 argued in appeal that computer simulations of the polymerization processes of examples 2 and 3 of D6 were performed on the basis of the process parameters disclosed in D6 as well as data generally available in the art. These computer simulations were shown in two graphs of D18 ("Simulation of Example 2 of

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WO 00/17245" and "Simulation of Example 3 of WO 00/17245") and showed according to respondent 1 that the pressure in the polymerization reactor was already dropping in the course of the polymerization process before the end of the addition of the peroxide initiator at the reaction temperature, so that the condition in claim 1 was met.

- As a preliminary remark it is noted that the reference to paragraph 9 of the patent in suit, which concerns the definition of the wording "after the start of the pressure drop and/or during the pressure drop" is not applicable to the interpretation of the claimed subject matter, at least because a different wording is used in claim 1 ("when the pressure in the polymerization reactor is dropping"). The use of that definition in the context of the computer simulations provided by respondent 1 is thus irrelevant to the question of novelty of claim 1 of auxiliary request 1.
- Regarding the computer simulation of example 2 of D6 5.4.1 ("Simulation of Example 2 of WO 00/17245"), the graph provided in D18 showed that a drop pressure in the reactor was recorded starting after the mark 120 min (2 h) was reached, up until the end of the simulation at the mark 125 min, as evidenced by the curve P Reactor (bara) on the graph. The peroxide initiator according to the polymerization process disclosed in D6 was dosed to the reactor from the start of the heating up phase (D6, page 7, lines 26 and 27) which corresponds to the mark 0 min on the graph provided in D18. The dosage of the peroxide initiator simulated in D18 is based on the data provided in Table II (page 9) of D6, indicating that the dosing time of the peroxide initiator was 2 h. It can be readily concluded therefrom that the dosage of the peroxide initiator (up

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to the mark 2 h) was already completed when the pressure in the reactor started to drop (after 2 h). Under these circumstances, even if it is accepted that the simulation of example 2 of D6 provides an accurate reproduction of the example, no proof has been provided that the disputed condition of claim 1 is met (actually the contrary has been shown).

- 5.4.2 With regard to the computer simulation of example 3 of D6 ("Simulation of Example 3 of WO 00/17245"), the graph provided in D18 showed that a drop in pressure in the reactor was recorded starting after the mark 160 min (2 h and 40 min) was reached, up until the end of the simulation at the mark 175 min, when the conversion of the monomer reached 90% approximately, as evidenced by the curves P Reactor (bara) and mass conversion (%) on the graph. The data provided in Table II (page 9) of D6 indicate that the peroxide initiator was dosed to the reactor over 4 h, after which the conversion of the monomer reached 90% (Polymer yield in Table II). Both D6 and D18 report a monomer conversion of 90% at the end of the polymerization reaction in the case of example 3. However, the 90% monomer conversion was reached after dosing the initiator for 4 h in D6 whereas the same conversion was achieved after only 2 h and 40 min (175 min) in the computer simulation provided in D18. In that regard, the question that the Board had to answer was whether the computer simulation provided in D18 could be seen as an accurate representation of the polymerization process of example 3 of D6 in spite of the relevant difference in the duration of the polymerization to reach a monomer conversion of 90%.
- 5.4.3 The interpretation of the temperature profiles and monomer conversion in the course of the computer

simulation of the polymerization process of example 3 in the graph of D18, as provided by the appellant on pages 3 and 4 of the letter dated 8 May 2018 and at the oral proceedings, renders credible the argument that the simulations provided in D18 were based on a water to VCM ratio that is lower (1:1 was suggested) than that derived from D6 (4:1). That argument is also congruent with the shorter duration of the polymerization simulated in D18 (2 h and 40 min) as compared to the polymerization performed in D6 (4 h), since a more concentrated monomer solution (e.g. water to VCM ratio of 1:1) is expected to reach a given conversion in the course of its polymerization sooner than when the monomer solution is more diluted (e.g. water to VCM ratio of 4:1).

5.4.4 Respondent 1 did not contest the argument that the computer simulation of example 3 in D18 were carried out under the premise of a water to VCM ratio lower than that used in example 3 in D6. Instead, additional data was provided in a letter of 11 June 2018 containing three additional computer simulations of the polymerization of example 3 of D6 obtained at water to VCM ratios of 1:1, 2:1 and 4:1 (the reference to example 2 in that letter was corrected by respondent 1 in the course of the oral proceedings before the Board). While the graphs provided in that letter show that an increasing water to VCM ratio has little impact on the overall course of the computer simulation, the data made available by respondent 1 confirm more importantly that a monomer conversion corresponding to that reported in example 3 of D6 is consistently obtained after only 175 min of initiator addition, as already established in the simulation provided in D18.

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- 5.4.5 Whether the discrepancy regarding the duration reported for reaching a monomer conversion corresponding to the end of the polymerization process in D6 and in the computer simulations provided by respondent 1 is effectively due to the water to VCM ratio or to another parameter of the polymerization is not relevant to answer the question posed above under point 5.4.2. Rather, the profile of the monomer conversion, and in particular its value at 175 min, made available in the computer simulations of the polymerization process of example 3 of D6, establish that the computer simulations do not accurately represent the polymerization performed in D6 in view of a large discrepancy in one of the key features of the process. Under these circumstances, it cannot be ascertained whether the pressure drop observed in the graphs of the computer simulation corresponds to that of the polymerization process of example 3 of D6. In view of this, it can only be concluded that the computer simulations provided by respondent 1 are not a reliable reproduction of the example and therefore are not suitable to show that the polymerization process of example 3 of D6 falls under claim 1 of auxiliary request 1.
- 5.4.6 That conclusion is not altered by the simulation of the polymerization process of example F of D6 provided by respondent 1 in D19 or of the process disclosed in the examples of the patent in suit provided in the letter dated 11 June 2018. Indeed, if the data shown in the simulations of example F and of the examples of the patent in suit (dosing of the initiator, constant pressure phase, monomer conversion and overall duration of the polymerization) can be seen as fairly corresponding to the data disclosed in D6 (Table VII), D21 (page 12) and the patent in suit (Table 1), that is

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not the case for the data shown for example 3 of D6 as discussed above under points 5.4.2 to 5.4.5. Hence, even if it were accepted that the simulations of example F and of the patent in suit could ultimately be seen as fair representations of the corresponding polymerizations as performed in example F of D6 and in the patent in suit, that would in any case not be valid for the simulations of example 3 of D6 as provided in D18 and repeated in the letter of 11 June 2018.

- 5.4.7 The Board concludes therefore that claim 1 of auxiliary request 1 is novel in view of D6.
- 6. As the respondents did not present any further objections against auxiliary request 1 there is no need for the Board to decide on any other issue.

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Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the department of first instance with the order to maintain the patent in amended form on the basis of the claims of auxiliary request 1 filed with the statement of grounds of appeal and after any necessary consequential amendment of the description.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated