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Datasheet for the decision of 21 June 2012

Case Number:	T 0478/09 - 3.3.07		
Application Number:	04753255.1		
Publication Number:	1628766		
IPC:	B01J 37/26, B01J 37/00, C08G 65/10, C08G 65/26		

Language of the proceedings: EN

Title of invention: Nano-scale DMC catalyst particles

Patent Proprietors Dow Global Technologies LLC

Opponents: BASF SE

Headword:

-

Relevant legal provisions:

EPC Art. 54, 56, 84, 108

Keyword:

"Form of appeal - admissible (yes) - partial admissibility (no)"
"Claims - lack of clarity of a granted claim - need for
interpretation of the feature "average particle size
determined by TEM" (yes)"
"Novelty (yes) - Main Request (the known catalyst having a
bimodal distribution of particles does not make available a
catalyst in form of either of its subgroups of particles)"
"Inventive step (yes) - non obvious alternative (Main
Request)"

Decisions cited:

G 0010/91, T 0093/09, T 1127/02, T 0151/05, T 1819/07, T 1188/00

Catchword:

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Beschwerdekammern

Boards of Appeal

Chambres de recours

Case Number: T 0478/09 - 3.3.07

DECISION of the Technical Board of Appeal 3.3.07 of 21 June 2012

Appellants:	BASF SE	
(Opponents)	D-67056 Ludwigshafen	(DE)

Respondents:Dow Global Technologies LLC(Patent Proprietors)2040 Dow CenterMidland, MI 48674(US)

Representative:

Raynor, John Beck Greener Fulwood House 12 Fulwood Place London WC1V 6HR (GB)

Decision under appeal:

Decision of the Opposition Division of the European Patent Office posted 17 December 2008 rejecting the opposition filed against European patent No. 1628766 pursuant to Article 101(2) EPC.

Composition of the Board:

Chairman:	J.	Riolo
Members:	G.	Santavicca
	D.	T. Keeling

Summary of Facts and Submissions

- I. The appeal by the opponents lies from a decision of the Opposition Division, posted on 17 December 2008, rejecting the opposition against European patent No. 1 628 766.
- II. The patent was granted on European patent application n° 04 753 255.1, originating from international application PCT/US04/16394, published as WO 2004/105944 A1, and comprised 20 claims. Independent claims 1, 10 and 18 to 20 read as follows:

"1. A process for making a metal cyanide catalyst comprising

(A) forming an emulsion having a plurality of water droplets dispersed in an immiscible continuous phase, wherein the water droplets contain a transition metal cyanide compound and a metal salt that reacts with the transition metal cyanide compound to form a water-insoluble metal cyanide catalyst, and
(B) subjecting the emulsion to conditions such that the transition metal cyanide compound and the metal salt react in the water droplets to form the water-insoluble metal cyanide catalyst."

"10. A process wherein a metal cyanide catalyst is mixed with an alkylene oxide and the resulting mixture subjected to conditions including an elevated temperature sufficient to polymerize the alkylene oxide to form a poly(alkylene oxide), wherein the metal cyanide catalyst is the product of a process of any of claims 1-9." "18. A process wherein a poly(propylene oxide) polymer is EO-capped, comprising contacting the poly(propylene oxide) polymer with ethylene oxide under polymerization conditions in the presence of a catalytically effective amount of a metal cyanide catalyst in the form of particles having an average particle size, as determined by transmission electron spectroscopy, of from 5 to 500 nm, prior to being exposed to an alkylene oxide."

"19. A metal cyanide catalyst in the form of particles having an average particle size, as determined by transmission electron spectroscopy, of from about 5 to about 500 nm, prior to contact with an alkylene oxide."

"20. A process wherein a metal cyanide catalyst of claim 19 is mixed with an alkylene oxide and the resulting mixture subjected to conditions including an elevated temperature sufficient to polymerize the alkylene oxide to form a poly(alkylene oxide)."

- III. The patent was opposed to the extent of Claims 18 to 20 on the grounds of lack of novelty and lack of an inventive step (Article 100(a) EPC) having regard to documents D1 (WO 97/26080) and D2 (WO 01/44347).
- IV. In the decision under appeal, it was inter alia held
 that:
 - (a) D1 disclosed a DMC catalyst having a bimodal particle size distribution within the range of 0.1 to 10 µm measured by light scattering in polyether polyol dispersion of catalyst particles. Thus, D1 identified two groups of particles in a bimodal mixture, wherein the group of smaller particles was

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composed of particles having a size within the range of 100 to 500 nm, preferably within the range of 150 to 400 nm. In particular, the illustrated bimodal catalyst could contain a minor component having particle sizes comprised between 0.1 and 0.2 µm and a major component having particle sizes comprised between 0.9 and 4.0 µm. However, D1 was silent as to the form of the particle size distribution. Although physical means for separating those particle groups were available at the filing date of the patent, D1 neither disclosed nor hinted at such a separation, let alone that after separation one of the two groups of particles would inevitably have an average particle size within the range defined in Claim 19 as granted, i.e. 5 to 500 nm. Since the average size of the particles of the bimodal mixture disclosed by D1 was in the micrometric range, which was different from the nanometric average particle size of Claim 1, the catalyst of Claim 19 was novel. So did the processes of Claims 18 and 20, which were characterized by the use of the novel catalyst.

(b) As to inventive step, the closest prior art was disclosed in any of D1 and D2. Independently from the technical problem stated in the patent in suit, the problem solved over D1 and D2 was to provide a new catalytic composition form. Since D1 disclosed particle sizes in the micrometric range and D2 did not refer at all to the particle size of the catalyst used in the reactions, none of them, in isolation or in combination, would have led the skilled person to a catalyst as defined in Claim 19, which albeit in nanometric scale was appropriate for the processes of Claims 18 and 20. Thus, the subject-matter of Claims 18 to 20 was not-obvious.

- (c) Therefore, none of the grounds of opposition prejudiced the maintenance of the patent as granted.
- V. In their statement setting out the grounds of appeal, the appellants maintained both grounds of opposition, namely lack of novelty against Claims 19 and 20 and lack of an inventive step against Claims 18 to 20. Arguments were provided against Claims 19 and 20, whilst for Claim 18 reference was made to the arguments submitted during the opposition proceedings.
- VI. The patent proprietors (respondents) countered the objections raised in the statement setting out the grounds for appeal by letter of 21 August 2009, in which they also raised the objection that the appellants, by simply referring to the opposition proceedings, had not substantiated their reasons on why the decision made on Claim 18 should be reversed. Hence, the appeal should be limited to Claims 19 and 20.
- VII. In a communication of 7 May 2012, in preparation for the oral proceedings, the Board indicated the issues to be debated and decided.
- VIII. By letter of 21 May 2012, the appellants submitted arguments on the admissibility of their appeal to the extent of Claim 18 as well as further arguments on lack of novelty and inventive step. They also filed a further document (D3), i.e. Römpp Lexikon, Elektronenmikroskop, 1997, pages 1124 to 1126.

- IX. In their letter of 8 June 2012, the respondents enclosed an Auxiliary Request, in which Claims 18 to 20 specified that the average particle size was a volume average particle size.
- X. Oral proceedings were held on 21 June 2012. After deliberation by the Board, the decision was announced.
- XI. As far as relevant for the present decision, the appellants essentially argued as follows:

Procedural matters - Admissibility of the appeal

(a) The appeal complied with all the requirements of the EPC. The requests were clear. The fact that in relation to Claim 18 no new argument had been provided, i.e. in addition to the reference to the objections raised in the opposition proceedings, implicitly meant that the decision to maintain Claim 18 was wrong in view of those objections. For reasons of economy of procedure, the reference to previous objections was sufficient. Thus, the appeal was admissible.

Main Request

Lack of disclosure, lack of clarity, need to construe Claim 19

(b) The assertion by the proprietors that transmission electron spectroscopy (TEM) as such was known was not contested. However, as apparent from D3, TEM was a collective name encompassing different methods. The patent specification did not provide any details on how the average particle size was

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actually determined by TEM, e.g. how the samples were prepared, how many particles and what aspect of the particles had been considered. In particular, it was not clear how particles having irregular shapes, e.g. a cylinder, had been treated (the base or the lateral surface, if any, or the smallest or the biggest diameter, had been considered?). Since TEM was based on particle size measure from image analysis, which provided number-based distributions, it was not clear how these number-based particle sizes were then converted into volume-based particle sizes. These different ways of carrying out TEM greatly impacted on the results. Depending on the actual determination chosen, e.g. sampling, measurement, computation, it was likely that the same catalyst might fulfil or not the condition of Claim 1. Also, Claim 19 did not mention a volume average particle size, whilst the values given by D1 were based on volume distributions. Therefore, the feature of Claim 19 "having an average particle size, as determined by electron spectroscopy, of from 5 to 500 nanometres" had to be interpreted so broadly that it should be disregarded. It could not represent a distinguishing feature over D1.

Novelty

(c) The claimed subject-matter did not mention a volume average particle size. D1 dealt with DMC catalyst particles and mentioned, as a preferred option, the presence of a bimodal distribution of particles, a fraction of which (clearly defined as such in Claim 3 of D1) was made up of particles having volume average sizes ranging from 150 to 400 nm.

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This fraction fulfilled the definition of Claim 1, the subject-matter of which was thus not novel.

Closest prior art

(d) D1 and D2 belonged to the same technical field of the patent in suit, namely DMC catalysts for making polyols for polyurethanes. Thus, either of them might be considered as the closest prior art. D1 *inter alia* disclosed DMC catalyst particles having a bimodal distribution, wherein an individualized fraction of small particles had particle sizes in the range from 150 to 400 nm, determined by quasi elastic light scattering (QELS). D2 did not mention any DMC catalyst particle size but addressed the problem of controlling the EO-capping of the poly(propylene oxide) chain, as the patent in suit.

Problem and solution

- (e) D1 was not acknowledged in the patent in suit. It had not been proven that the claimed subject-matter attained a particular effect over D1. In fact, the patent in suit itself showed that the sought-for effect was not attained over the whole breadth of Claim 19. Hence, the problem solved over D1 can only be seen in the provision of DMC catalysts having smaller particle sizes.
- (f) D2, also not acknowledged in the patent in suit, addressed the same problem of the patent in suit but did not give any particle size of the applied DMC catalysts. In the method of Example 3 of D2 a dispersion of the catalyst particles was formed

with an Ultraturrax, which implied formation of small catalyst particles. Hence, the problem solved over D2 was still the provision of DMC catalyst having smaller particle sizes.

Obviousness

- The only difference between the subject-matter of (g) Claim 19 and the disclosure of D1, if any, lay in the claimed average particle sizes. However, a reduction to nanosize did not automatically implied an inventive step. It was known that smaller particles displayed a greater surface area, which played a role in catalysis processes. That smaller particle were better had also been admitted by the respondents. Since smaller particles were better, the skilled person starting from D1 would obviously try to separate the small fraction mentioned in D1 (having particle sizes of 100 to 200 nm, table of page 18 of D1) or, if unimodal distributions were used (which were encompassed by D1), to reduce the size of the particles, before using them in the polymerization of alkylene oxides.
- (h) For the same reasons, the skilled person starting from Example 3 of D2, with the aim of finding further suitable catalysts, would consider using the particles having the size of the small fraction contained in the catalyst of D1.
- (i) Thus, the claimed subject-matter was obvious over either of D1 and D2.

XII. As far as relevant for the present decision, the respondents argued essentially as follows:

Procedural matters - Admissibility of the appeal

(a) The statement setting out the grounds of appeal did not contain a party's complete case in relation to Claim 18, as reasons why the decision under appeal should be reversed had been given only in relation to Claims 19 and 20. As regards Claim 18, there was a mere reference to objections raised during the opposition proceedings. Thus, the appeal should be limited to Claims 19 and 20.

Main Request

Alleged insufficiency of the disclosure and/or lack of clarity Interpretation of Claim 19

Although insufficiency of the disclosure had not (b) been invoked, and lack of clarity was not a ground of opposition, these issues were actually argued by the appellants. In fact, transmission electron spectroscopy (TEM) was a known technique. Also, it was clear from the specification that, for the defined range of 5 to 500 nm as determined by electron spectroscopy, a volume particle size had been calculated. This calculation was based on the "equivalent sphere approximation", i.e. on the indication of the size of a sphere giving the same response as the particle being measured. The measurement techniques used in D1 and in the patent in suit were not identical but it had not been contested that the results were the same. Instead,

the appellants, who had the onus of proof in this respect, had not shown that TEM was not appropriate for the claimed determination, nor that it could not be carried out, let alone that it did not gave the same results as the technique of D1. Thus, the objections raised by the appellants under Articles 83 and 84 EPC should not be considered.

Novelty

(c) The patent in suit related to a new method for making nanometric particles of DMC-catalysts. D1 disclosed particle sizes in the micrometric range, a different mixture of particles, thus there was no disclosure in D1 of a nanometric mixture of particles as claimed. Hence, it was not realistic to compare the claimed subject-matter with the disclosure of D1. In nay case, the onus of proof was on the appellants, who had never proven beyond any reasonable doubts that the claimed subjectmatter was inherently disclosed in D1. Thus, the arguments on lack of novelty were not convincing.

Closest prior art

(d) According to the problem solution approach, the selection of the closest prior art document had to be made on objective criteria. D1 did not address the problem of catalyzing the EO-capping reaction on poly(propylene oxide) chains. Also, D1 disclosed nanoparticles contained in a paste, but the paste was made of bigger particles. As to D2, identified by the Board as possible closest prior art document, as it addressed the same problem as the patent in

suit, it did not mention any particle size. So D1 did not appear to be an appropriate starting point.

Problem and solution

- (e) Having regard to D1, the problem solved was to provide improved catalysts suitable for EO-capping of the poly(propylene oxide) chains. It had been demonstrated, e.g. by the examples in the patent in suit, that the sought-for EO end-capping was attained by the claimed DMC catalyst nanoparticles.
- The examples of D2 which used the catalyst of (f) Example 3, invoked by the appellants because of the allegedly small size, produced viscous polymers having a low content of primary hydroxyl groups (compared to the examples in the patent in suit). Example 10 of D2 did not use the catalyst of Example 3, but that of Example 2 (which had not been produced as dispersion by an Ultraturrax and which was solid, of unspecified size, possibly bigger than that of the catalyst of Example 3) and produced a clear polyol having a 46% content of primary hydroxyl groups. So, at least over the catalyst of Example 3 of D2, the claimed catalyst still provided an advantage. Hence, the problem solved over D2 was to provide further, advantageous catalysts for the process of EO end-capping.

Non obviousness

(g) D1 disclosed pastes containing catalyst particles that were bigger than those claimed and sieving of catalyst particles, which nevertheless were bigger than those claimed. D1 disclosed particle sizes in the range 0.1 to 10 micrometres and did not suggest any separation of the fraction containing small particles. Nor gave D1 any motivation to catalyst particles of the claimed size. Indeed, In D1 a comparison was made over prior catalysts having particle sizes ranging from 4.6 to 600 micrometres. So the methods of D1 would not lead the skilled person to the claimed subject-matter, which was obtained from a new method of preparation.

- (h) For the very same reasons, the combination of D2 and D1 would not lead the skilled person to the claimed subject-matter.
- XIII. The appellants (opponents) requested that the decision under appeal be set aside and that the patent be revoked to the extent of Claims 18 to 20.
- XIV. The respondents (patent proprietors) requested that the appeal be dismissed, or, in the alternative, that the patent be maintained on the basis of the Auxiliary Request filed with letter of 8 June 2012.

Reasons for the Decision

1. The appeal is admissible.

Procedural matters - Admissibility of the appeal

2. The respondents argued that the appeal was admissible only to the extent of Claims 19 and 20, as the grounds for reversal of the decision in relation with Claim 18

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had not been substantiated in the statement setting out the grounds of appeal.

- 2.1 It is not contested by the respondents that the appellants have sufficiently substantiated their position in regard to the alleged identity between the subject-matter of Claims 19 and 20 and the disclosure of D1, i.e. that they have dealt with the crucial point of the first ground of opposition, i.e. lack of novelty, invoked against Claims 19 and 20. The admissibility of the appeal in this respect is thus not in dispute.
- 2.2 Since the appeal is indisputably admissible to the extent of Claims 19 and 20, and since there is no support in the EPC for a notion of partial admissibility of an appeal (Case Law of the Boards of Appeal of the EPO, 6th edition 2010, VII.E.7), the appeal as a whole (i.e. to the extent of Claims 18 to 20) is admissible.

Procedural matters - Amendment to appellants' case

3. During the written proceedings, the parties argued that differences existed between the measurement method of the patent in suit and of D1, which however did not impact on the results achieved in a significant matter (statement of opposition of 17 October 2007, Point 2., second paragraph; response by the proprietors of 2 April 2008, page 2, second paragraph, second sentence). However, during the oral proceedings, the appellants argued that the difference significantly impacted on the results, to the extent that no limiting value could be attributed to the parametric feature of Claim 19, which thus should be disregarded. Since the appeal fails (*infra*), the Board need not consider whether that amendment to the appellants' case is allowable under Article 13 of the Rule of Procedure of the Boards of the Board of Appeal (RPBA) (T 93/09 of 19 April 2012, not published in the OJ EPO).

Procedural matters - Alleged insufficiency of the disclosure

- 4. It is not in dispute that Transmission Electron Microscopy (TEM) as such is known (letter by the appellants of 21 May 2012, Page 2, lines 21-22). However, with reference to D3, the appellants object that the patent specification is silent on how the TEM generally defined in Claim 19 is actually carried out, i.e. that it is not disclosed. This implied, depending on the specific TEM determination applied, that the same catalyst can or cannot fall under the scope of Claim 19, i.e. that the skilled person would not know whether that catalyst falls inside or outside the claimed scope (same letter, page 3, 4th paragraph).
- 4.1 Since no ground of opposition under Article 100(b) EPC has ever been raised, nor has the consent of the proprietors to deal with such a fresh ground of opposition (G 10/91, OJ EPO 1993, 420) ever been given, the Board cannot consider these arguments under the opposition ground of insufficiency of the disclosure.

Procedural matters - Alleged lack of clarity of granted claims

5. The appellants have objected that the feature of Claim 19 "average particle size determined by transmission electron spectroscopy of from about 5 to about 500 nm", having regard to the undisclosed method of measurement, is unclear as to its breadth, and cannot represent a distinctive feature of Claim 19 over D1 (letter by the appellants of 21 May 2012, page 3, 4th paragraph).

- 5.1 This objection of lack of clarity is against a claim as granted. However, lack of clarity cannot be challenged in opposition appeal proceedings, as the requirement that a claim of a European patent shall be clear is laid down in Article 84 EPC, which is not itself a ground of opposition under Article 100 EPC.
- 5.2 In the present case, the contested feature indisputably represents the only distinction over D1, if any. So the alleged lack of clarity may however affect the decision on issues under Article 100(a) EPC, such as novelty.
- 5.3 Thus, if the wording of Claim 19 does not allow a clear distinction of its subject-matter over D1 (if Claim 19 is not clear in itself), it will have to be interpreted in the described context.
- 5.4 Hence, the Board may not deal with clarity of granted Claim 19 but should decide whether Claim 19 is clear in itself, i.e. whether there is a need for interpretation.

Procedural matters - applicable rules of interpretation

6. As a general rule, an ambiguous text is construed in the broadest possible way (T 1127/02 of 14 September 2004, Point 7 of the Reasons, second paragraph), i.e. against the interest of the person responsible for its drafting (in the present case the proprietors, now respondents), and in favour of the person on whom it is

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imposed (in the present case the opponent appellants as member of the public) (T 0151/05 of 22 November 2007, Point 3.2.2 of the Reasons, last paragraph).

- 6.1 Some elements of this possibly broadest interpretation are set out in the Case Law of the Boards of Appeal of the EPO (6th edition 2010), as follows:
 - (a) Interpretations that are illogical or do not make technical sense should be ruled out. Interpretation of a claim should be technically sensible and take into consideration the whole disclosure of the patent (II.B.5.1).
 - (b) When assessing novelty of broad claims, there is no reason to use the description to interpret a broad claim more narrowly, if the question is merely that of examining a broad claim in relation to the state of the art (I.C.2.2 and II.B.5.3.2).
 - (c) The terms used in the claim should be given their normal meaning in the relevant art, unless the description gives them a special meaning (II.B.5.3.3).
 - (d) It is inconsistent with proper claim interpretation to read into a claim a particular meaning that only appears in the description (II.B.5.3.4). So in the course of the examination of an opposition/appeal, the patent proprietors cannot rely on Article 69 EPC as a substitute for an amendment which would be necessary to remedy a lack of clarity.

Main Request (patent as granted)

Need to construe Claim 19

- 7. The contested feature of Claim 19 ("average particle size, as determined by transmission electron spectroscopy, of from about 5 to about 500 nm") is made up of the following items of definition:
 - (a) (what) average particle size;
 - (b) (determined how) by transmission electron microscopy (TEM); and
 - (c) (expressed how) about 5 to about 500 nm.
- 7.1 As regards the first item, it comprises two notions, "average" and "size".
- 7.1.1 The notion "size" is based on the assumption that a single dimensional property is indicative of the particle size. However, particles are three-dimensional objects, for which at least three parameters (length, breadth and height) are required to provide a complete description. The measurement of some one-dimensional property of a particle and the reference to a sphere in order to derive one unique number, namely of the sphere having the same response (diameter, cross-sectional area, volume, weight, sedimentation rate, etc.) as the particle under examination, is known as "equivalent sphere approximation". In respect of this notion, Claim 19 does not specify which size is meant (larger/smaller dimension of the particle, diameter of a sphere having the same surface, cross-sectional area or volume, etc.).

- 7.1.2 As to the notion "average" (mean), it is not in dispute that transmission electron microscopy (TEM) generates a two-dimensional image of the particles contained in the sample, wherefrom the particle are characterized. On this image, a number of sizes can be measured, such as maximum or minimum diameter of the particle, which are then summed and divided by the number of particles, to obtain a number-length mean. It is also possible to mathematically convert the diameters measured to obtain a volume or mass mean, whereby however the conversion increases the error of the measurement. As regards the "average", Claim 19 does not specify what is meant (number-, volume-average, etc.). So it encompasses any of the common averages obtained from TEM.
- 7.2 As to the second item of information, Claim 19 does not specify how the method of measurement, i.e. TEM, is actually carried out, e.g. as regards sampling, determination, measurement, treatment of data, etc.
- 7.3 As regards the third item of information, it merely quantifies the concept of average particle size by a nanometric range. Since no statistical deviation is mentioned, the actual distribution of the particle sizes is thereby not defined.
- 7.4 It follows from the foregoing that the definition of Claim 19, in particular the "average particle size", is not as clear as possible. In this respect, the present decision is in line with decision T 1819/07 of 15 March 2011 (Reasons, in particular Point 3.5 thereof). Thus, Claim 19 is to be construed.

The construed subject-matter of Claim 19

- 8. Claim 19 concerns a metal cyanide catalyst in the form of particles having an average particle size, as determined by TEM, of from about 5 to about 500 nm, prior to contact with an alkylene oxide.
- 8.1 In relation to the general process of manufacture, the patent in suit mentions that "the DMC catalyst complex can be prepared at such small particle sizes by precipitating it in the dispersed aqueous phase of a water-in-oil emulsion" (Paragraph [0027]) and that "conditions are selected such that the aqueous phase forms droplets of about 500 nm or smaller in diameter" (emphasis added) (Paragraph [0029], third sentence; Paragraph [0052]). Since the particles are formed within those droplets, their maximum size (diameter) also lies within the upper limit of 500 nm. Hence, the feature "in the form of particles having an average particle size" is construed as implying that all particles have such an average size.
- 8.2 For the range of 5 to 500 nanometres, as defined in Claim 19, the patent in suit does not generally define what average and what size are meant.
- 8.3 Only in relation to the description of preferred embodiments does the patent in suit mention a volume average particle size, as follows:
 - (a) "The particles preferably have a volume average particle size from about 10 nanometres, such as from 40 nanometres, to 300 nanometres, more preferably 250 nanometres, especially to

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200 nanometres, most preferably to 150 nanometres" (Paragraph [0026]).

(b) "This catalyst has a volume average particle size of about 40 nanometres" (paragraph [0078], first sentence).

No details of how the volume average particle sizes have been computed from the measured values is disclosed.

- 8.4 It follows from the foregoing that:
 - (a) The interpretation invoked by the appellants (the objected feature is not suitable to impart any limitation, it should not be considered) cannot be retained, as it disregards the whole disclosure of the patent (case law, supra, II.B.5.1).
 - (b) The interpretation invoked by the respondents (the objected feature concerns a volume average particle size) cannot be accepted either. The limitation "volume average particle size" appears in the description only in relation to preferred embodiments. So it cannot be read into Claim 1 also for the generic range of 5 to 500 nm (case law, supra, II.B.5.3.4).
- 8.5 Hence, the average particle size defined in Claim 19 encompasses particles having any size ranging from 5 to 500 nm measured or computed and averaged from distributions commonly obtained with TEM equipment.

The disclosure of D1

9. D1 discloses (Claim 1) "a catalyst which comprises a paste of a double metal cyanide (DMC) compound, an organic complexing agent, and water, wherein the paste catalyst comprises at least about 90 wt.% of particles

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having a particle size within the range of about 0.1 to about 10 micrometers as measured by light scattering in polyether polyol dispersions of the catalyst particles.

- 9.1 D1 (Claim 12) also discloses a method of forming the catalyst, which comprises: (a) vigorously combining a powder DMC catalyst with an organic complexing agent to produce a reconstituted catalyst slurry; and (b) isolating a paste catalyst that contains DMC compound, organic complexing agent, and water.
- 9.2 D1 (page 11, lines 12-27) also discloses DMC catalysts prepared by sieving powder DMC catalysts and using only the smallest particles. In particular, D1 (Claim 15) discloses a powder DMC catalyst wherein at least about 90 wt.% of the catalyst particles can pass through a U.S. Standard Sieve of 230 mesh (63 µm).
- 9.3 The examples of D1 concern the preparation of paste (Example A), reconstituted paste (Example C), suspension (Example D) and powder catalysts (Examples B and E).
- 9.4 As regards the particle sizes and their distribution, D1 discloses that:
- 9.4.1 The paste can comprise at least about 90 wt.% of particles having a particle size within the range of about 0.1 to about 5 micrometers (page 6, lines 18-24).
- 9.4.2 The catalyst can comprise particles having a particle size within the range of 0.1 to 0.5 μ m as measured by

quasi elastic light scattering (QELS) in polyether polyol dispersions of the catalyst particles (Claim 3).

- 9.4.3 The particles can have a bimodal particle size distribution within the range of 0.1 to 10 μ m (Claim 4).
- 9.4.4 A major proportion of the catalyst particles have a size within the range of 1 to 10 μ m, and a minor proportion of the particles have a size within the range of 0.1 to 0.5 μ m (Claim 5). The larger particles can have a size of 1 to 5 μ m, and the smaller particles can have a size of 0.15 to 0.4 μ m (page 7, lines 1-3).
- 9.4.5 As regards the powder catalysts, Example B discloses no size. Example E illustrates sieved powder catalysts as shown in Table 6. The catalysts having the smallest particles (Ex.#25; retained on the sieve tray of 400 mesh; retained in the bottom pan) have a size of 63 to 44 µm (Ex.#25), of 38 µm (400 mesh), or are unspecified (bottom pan). Comparative Example C2 of Table 1, has a particle size ranging from 4,6 to 600 µm.
- 9.5 As regards the determination of particle sizes and distributions, D1 discloses that:
- 9.5.1 The particle sizes for catalysts of D1 are measured by first dispersing the paste catalyst in a polyether polyol (molecular weight less than about 1000, see Example G), and then measuring the size of the particles in this dispersion by light scattering, e.g. by using a Leeds & Northrup MICROTRAC X100 particle analyser, which measures static light scattering properties of the particles. This instrument can

approximate the relative amounts of small and larger particles in the paste catalysts (page 7, lines 14-22).

9.5.2 The very small catalyst particles (size less than 0.5 µm) are analysed with quasi-elastic light scattering (QELS), which measures the dynamic light scattering properties of the particles. This technique is used to verify the presence and particle size distribution of very small catalyst particles in a sample. QELS is performed on suspensions of catalyst particles in low molecular weight polyol. A suspension of 5 wt.% of DMC catalyst in dipropylene glycol is suitable for use in QELS measurements (paragraph bridging pages 7 and 8).

Novelty

- 10. Since the appellants maintain that the catalyst of Claim 19 lacks novelty over D1, it has to be decided whether any of the catalysts disclosed in D1 directly and unambiguously discloses the features of Claim 19.
- 10.1 Having regard to the analysis of the disclosure of D1 (*supra*), the Board cannot see a direct and unambiguous disclosure of a catalyst in powder form having the average particle size specified, as:
 - (a) The disclosure of D1 relating to a paste concerns a single mixture of particles of different sizes, for which D1 does not disclose any average value.
 - (b) The determination of the particle sizes is carried out by two methods, the first approximating the relative amounts of smaller and larger particles, the second determining presence and distribution of very small particle size.

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(c) Both methods are non-destructive, i.e. based on scattering of light by all particles present in the mixture, which thus remains as such. In fact, as indicated in Footnote 3 of Table 1, light scattering analyses are carried out on samples prepared according to Example G, which samples consist of a suspension including all of the measured particle sizes.

- (d) The fact that the distribution of the particles of the paste of D1 can be bimodal merely implies that the particles of the sample (subset of all particles of interest) to be analysed can be made up of heterogenous subgroups, giving rise to a set of data, the histogram of which has no normal distribution, but two maxima, which behave as distinct modes. Nevertheless, the subgroup of smaller particles remains mixed to the subgroup of larger particles in a single spread of particles. So, the disclosure of a bimodal distribution of particles, which is determined by non-destructive tests, during which no actual separation of the subgroups of smaller and larger particles is carried out, does not make available any of the physical subgroups as a distinct physical entity.
- (e) Hence, no separation of any fraction of particles of small and/or large sizes is illustrated. As a case in point, D1 does not illustrate the separation of the minor component of the bimodal paste of Ex.#1, the particles of which have sizes of 0.1 to 0.2 μm. So no physical fraction of catalysts in particle form having particle sizes of 0.1 to 0.2 μm is implicitly made available thereby.

- (f) Thus, in D1 there is no direct and unambiguous disclosure of any physically separated "fractions" of the catalyst in paste form.
- (g) As to the catalyst powders of D1, which are obtained by sieving, they have sizes that are larger than 500 nm. In this respect, D1 neither discloses a separation of any fractions thereof, nor their crushing to produce a powder.
- (h) As regards the undisclosed average particle size of the catalysts of D1, they can comprise up to 10% of particles having a size greater than 10 µm. In any case, they comprise a major proportion of large particles ("size up to 10 µm or more") and a minor proportion of particles having nanometric sizes. Thus, the average particle size is presumably greater than 500 nm. This plausible presumption has never been displaced by evidence by the appellants.

10.2 Thus, the subject-matter of Claim 19 is novel over D1.

The disclosure of D2

- 11. D2 has not been invoked against the novelty of the catalyst of Claim 19. However D2 is relevant for assessing inventive step (*infra*), so its disclosure is to be considered, *inter alia* in order to establish the distinguishing feature of the claimed subject-matter.
- 11.1 D2 (Claim 1) concerns a process for preparing polyether alcohols by catalytic addition of at least two alkylene oxides onto H-functional initiator substances, wherein at least one multimetal cyanide compound is used as catalyst and the addition of the alkylene oxides onto the initiator substance includes incorporation of at

least one oxyalkylene block, during whose formation at least two alkylene oxides are metered in together and the ratio of the alkylene oxides to one another in the mixture is changed during the joint introduction.

- 11.1.1 The oxyalkylene block, which can consist of two alkylene oxides (Claim 2), such as ethylene oxide and propylene oxide (Claim 10), can be incorporated at the end of the polyether chain (Claim 7).
- 11.1.2 Multimetal cyanide catalysts can be used for the molecular addition of the alkylene oxides (Claim 11).
- 11.2 According to D2 (Page 7, lines 28-45), polyether alcohols for use in flexible molded urethane foam or in polyurethane elastomers usually have an inner block consisting of propylene oxide or a classical random mixture of ethylene oxide and propylene oxide, which is adjoined by a pure ethylene oxide block, in order to provide a high content of primary hydroxyl groups. When using multimetal cyanide catalysts, this procedure gives highly viscous, turbid polyethers having a low content of primary hydroxyl groups. Although the content of primary hydroxyl groups can be increased by molecular addition of relatively long ethylene oxide chains, this also results in an undesirably high increase in the hydrophilicity of the polyether alcohols. However, when starting from a classical random block or a block of only one alkylene oxide, if a block having an increased ethylene oxide content is added on at the end of the polyether chain, by the dynamic procedure, at the end of the metered introduction, the desired contents of primary hydroxyl

groups can be obtained without long ethylene oxide blocks having to be added on at the end of the chain.

- 11.3 In contrast thereto, for polyols used in flexible slabstock foam, preference is given to using polyether alcohols having primary hydroxyl group contents of less than 10% (page 8, lines 1-3).
- 11.4 So the process of D2 is suitable for producing polyols having low and high primary hydroxyl groups contents.
- 11.5 The process of D2 is advantageously carried out using multimetal cyanide catalysts, as these have a very high activity and therefore ensure that the alkylene oxide introduced is immediately incorporated into the chain (Page 8, lines 23-26). These compounds are prepared by generally known methods, i.e. by combining the aqueous solution of a water-soluble metal salt with the aqueous solution of a hexacyanometalate, in particular a salt or an acid, and adding a water-soluble ligand while the solutions are being combined or afterwards (Page 9, lines 22-27). Thus, the method of preparation mentioned in D2 is different from the method of the patent in suit, which uses an emulsion of small droplets.
- 11.6 Since, when using multimetal cyanide catalysts, high contents of terminal primary hydroxyl groups can be achieved by means of ethylene oxide end blocks only at the price of high hydrophilicity and extremely high viscosities (Page 3, lines 1-4), the object of D2 is to provide polyether alcohols (polyols) which could be prepared by catalytic addition of at least two alkylene oxides onto H-functional initiator substances, which polyols have no turbidity and viscosities favorable for

processing and which can be processed without problems to give polyurethanes, e.g. flexible polyurethane foams. In addition, the content of alkylene oxides at the end of the polyether chain should be able to be adjusted in a targeted way. High molecular weight tails, as occur in the molecular addition of alkylene oxides by a customary block procedure using DMC catalysts for preparing the polyether alcohols, should be avoided (page 4, lines 1-13).

- 11.7 The polyether alcohols of D2 have a very narrow molecular weight distribution and surprisingly display no turbidity. Owing to the very high reaction rate in the molecular addition of the alkylene oxides by means of DMC catalysts, controlled addition of the alkylene oxides onto the initiator substance is possible (Page 12, lines 36-41).
- 11.8 D2 illustrates the preparation of 3 DMC catalysts (Examples 1 to 3), by the known method acknowledged above. In Examples 1 and 2, a dried solid catalyst is produced, whereas in Example 3 the moist solid cake was dispersed in water for 5 minutes by an Ultraturrax. D2 does not specify whether the Ultraturrax used in its Example 3 produces a dispersion of nano particles of the catalysts. Nor has any evidence in this respect ever been provided by the appellants, who own the patent of D2. Nevertheless, Example 3 is the only example of D2 mentioning a particle size reduction.
- 11.9 Thus, D2 does not disclose any DMC catalyst in particle form having an average particle size as claimed.

Closest prior art

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- 12. The patent in suit concerns nano-scale DMC catalyst particles.
- 12.1 According to the patent in suit (Paragraph [0005]), this DMC catalyst (nano)particles more efficiently catalyze the EO-capping reaction as well as efficiently polymerize propylene oxide.
- 12.2 As the closest prior art documents, the decision under appeal considered D1 and D2, not because they dealt with the same problem as the patent in suit, but merely because they aimed at a new catalytic composition form. The appellants considered D1 as the closest prior art document. The respondents dealt with D1 in the written proceedings but argued orally that D2 rather than D1 described the closest prior art. Hence, it has to be determined whether D1 or D2 deals with the closest prior art according to the problem solution approach.
- 12.3 D1 (*supra*) does not address the problem of catalysing an EO-capping reaction. Instead, D2 (*supra*) (page 4, first paragraph) addresses one of the problems stated in the patent in suit (paragraph [0005]), namely that the content of alkylene oxides at the end of the polyether chain should be adjustable in a targeted way, and *inter alia* discloses the use of DMC catalyst particles, e.g. in reduced size form.
- 12.4 Therefore, the Board considers that D2 rather than D1 discloses the closest prior art.

Problem and solution

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13. The technical problem stated in the patent in suit (Paragraph [0005]) was to provide a DMC catalyst that more efficiently catalyzes the EO-capping reaction (i.e. to polymerize poly(ethylene oxide) end-caps onto poly(propylene oxide) polyols, to form polyols with primary hydroxyl groups, as detailed in Paragraph [0004] of the patent in suit). Furthermore, the catalyst should efficiently polymerize propylene oxide as well.

- 13.1 According to the patent suit (Paragraphs [0006] to [0012]), the problem is *inter alia* solved by a metal cyanide catalyst as defined in Claim 19 as granted.
- 13.2 Since D2 is not acknowledged in the patent in suit, the problem stated in the patent in suit did not consider D2. Also, during the oral proceedings before the Board, the respondents argued that the claimed catalyst is still advantageous over the catalysts disclosed by D2.
- 13.3 Therefore, as established in the Case Law of the Boards of Appeal of the EPO (6th edition 2010, I.D.4.4, in particular the acknowledgement of T 1188/00), it should be plausible that the effect first alleged in appeal proceedings (improvement over D2) is achieved across the whole scope of Claim 19.
- 13.4 This effect concerns the first of two polymerizations that are of particular interest for the catalyst of the patent in suit (Paragraphs [0043] and [0044]).

The first of these is the polymerization of ethylene oxide onto a poly(propylene oxide) homopolymer or copolymer having mainly terminal secondary hydroxyl groups, in which using the claimed catalysts cause ethylene oxide to polymerize onto a surprisingly high proportion of those terminal secondary hydroxyl groups to provide an EO-capped polyol with a significant proportion of primary hydroxyls.

According to D2, the proportion of end-groups that are EO-capped (i.e. with primary hydroxyl groups) tends to decrease as the molecular weight of the poly(propylene oxide) starting material increases, as follows: - For starting materials of about 1500 molecular weight or less, greater than 45%, or even greater than 50%, of the end groups can be EO-capped. - For starting materials of 1500-3000 molecular weight, 33-50% of end groups can be EO-capped. - For starting materials of 3000-4000 molecular weight,

Results within those ranges are illustrated for the catalysts of Examples 9-12 (Table 2), having particle sizes ranging from 48 to 210 nm (Table 1), which have been prepared in water-in-oil emulsions having a disperse phase droplet size of below 500 nm (Paragraph [0052]), as follows:

20-43% of the end groups can be EO-capped.

The catalysts of Examples 10 to 12, evaluated in the production of polyols having a molecular weight (Mn) of 1500, respectively attain the following end-groups EOcapping contents: 48-49%; 43-46%; and, 46-48%. The catalyst of Example 12, used at a higher level (5000 ppm instead of 1000 ppm), produces a polyol of molecular weight 2500 Mn having 48% EO-capping or a polyol of 3200 Mn having 38% EO-capping. The catalyst of Example 24 (Paragraph [0078]), having a volume average particle size of 40 nm produces polyols as follows: a polyol having a molecular weight of 2800 and 51% primary hydroxyl groups; polyols having a moelcular weight of 2900 and 3600 with 45% primary hydroxyls; and, a polyol having a molecular weight of 3800 and 32% primary hydroxyls.

It is important to note that in all the examples of the patent in suit ethylene oxide is added only after completion of the propylene oxide polymerization (paragraph [0060], lines 13-16).

- 13.5 D2 illustrates the results obtained by the use of DMC catalysts in its processes as follows:
- 13.5.1 Apart from the comparative examples of D2 (Examples 4-5, 8-9, and 11-12), where the obtained polyol is not clear and too viscous, the catalyst of Example 3 of D2 is used in Example 6, to produce a clear polyol having a molecular weight Mw of 4107 g/mol, a dispersity of 1,13 (ratio between Mw and Mn) and a content of primary hydroxyl groups of 5.3%. However, the two alkylene oxides are dosed together, whereby the dosing of ethylene oxide is linearly reduced to zero after 87,5% of the whole amount of alkylene oxides has been supplied. Hence, this situation is not comparable with the dosing in the examples of the patent in suit.
- 13.5.2 Instead, the catalyst of Example 2 of D2 (which has not been prepared by using an Ultraturrax, i.e. which is solid but of undefined particle size) is used in Examples 7 and 10, according two different dynamic dosing procedures, as follows: In Example 7, where the dosing of ethylene oxide is started immediately after the starting of the dosing of

the propylene oxide, the polyol has a molecular weight of 4111 and 4,3% of primary hydroxyl groups; in Example 10, where the dosing of the ethylene oxide is started 1 hour after the dosing of the propylene oxide, the polyol has a molecular weight of 3890 and 46% of primary hydroxyl groups.

- 13.6 Since the dosing dynamic of Example 10 is the closest to that of the examples of the patent in suit, the attained content of primary hydroxyl groups, having regard to the molecular weight of 3890, can be compared to those attained with the catalysts illustrated in the patent in suit. The comparison shows that: Patent in suit Catalyst 17-I (Table 2): produces a polyol having a molecular weight Mn of 3798 and 22% of primary hydroxyl groups (hence, lower than 46%); Catalyst of Example 24 (Paragraph [0078]): produces two polyols, respectively having a molecular weight of 3600 and 45% of primary hydroxyl groups or a molecular weight of 3800 and 32% of primary hydroxyl groups.
- 13.7 Thus, the catalysts illustrated in the patent in suit do not represent an improvement over all of the catalysts used in the illustrated processes of D2, if polyols of similar molecular weights are compared. Nevertheless, at least for lower molecular weights, a comparable content of primary hydroxyls can be attained with the claimed catalysts.
- 13.8 Therefore, the problem solved over D2 is considered to be the provision of alternative catalysts, to be used in the implementation of the processes of D2.

Obviousness

- 14. According to D2 (page 4, lines 15-22; page 7, lines 4-9), the use of two alkylene oxides, the ratio of which is adjusted during their dosage, permits to end-cap the polyol chains with ethylene oxide. Thus, the solution envisaged in D2 relies on the control of process conditions rather than on the choice of the catalyst itself. As regards the catalyst used, neither Example 3 nor Example 2 of D2 disclose the size of the catalyst particles. Since in D2 the catalyst does not play a role which is as important as the control of the process, D2 itself does not suggest the claimed solution.
- 14.1 D1 does not suggest anything in relation to the EOcapping problem to be solved as stated in the patent in suit and in D2. On the contrary, D1 illustrates the use of its catalysts in the production of polyols having a molecular weight of 8000 (see the examples of D1), which is much higher than the molecular weights of the polyols illustrated in the patent in suit and in D2. So, D1 and D2 do not appear to deal with the same polyols.
- 14.2 As regards the size of the catalyst particles, D1 discloses that:
- 14.2.1 Improved results are obtained from powder DMC catalysts wherein at least about 90 wt.% of the catalyst particles can pass through a U.S. Standard Sieve of 230 mesh (63 µm). As shown in Table 6 of D1, an unsieved powder DMC catalyst (Comparative Example 7) initiates polymerization (becomes active) within 7 minutes under the standard reaction conditions (see

Example F), and gives an 8000 mol. wt. polyoxypropylene diol having a viscosity of 3480 cks containing visible particulates of catalyst suspended in a 8000 molecular weight polyol. In contrast, a sample of powder DMC catalyst that passes through 230 mesh (63 μ m) initiates faster (within 5 minutes), and gives a clear 8000 mol. wt. polyol of low viscosity (3260 cks) and narrow molecular weight distribution (Mw/Mn= 1.14). The catalyst that passes through 140 mesh (104 μ m) and is retained on the 200 mesh screen (74 μ m) (Comparative Example 24) is no better than the composite material in terms of initiation time or polyol quality.

- 14.2.2 Hence, D1 suggests reducing the particles to the size as defined in order to improve the activity of the catalyst. That high activity of the catalysts of D1 is measured when producing high molecular weight polyols.
- 14.3 It follows from the foregoing that even if (despite all evidence) it were obvious to use the catalysts of D1 in any of the processes of D2, the catalysts would still have a size as disclosed in D1, i.e. of micrometric average size. So this combination would not lead the skilled person towards the subject-matter of Claim 19.

Claims 18 and 20

15. Since the product of Claim 19 is novel and inventive, its use as defined in Claims 18 and 20 is likewise novel and inventive.

Conclusion

16. None of the invoked grounds of opposition prejudices the maintenance of the patent as granted.

Auxiliary Request

17. In view of the decision made for the Main Request, the Board need not decide on the Auxiliary Request.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

S. Fabiani

J. Riolo