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Datasheet for the decision of 5 September 2024

Case Number: T 0981/22 - 3.3.05

Application Number: 16717287.3

Publication Number: 3280525

IPC: B01J13/16, C11D3/50

Language of the proceedings: EN

Title of invention:

PROCESS FOR PREPARING POLYUREA MICROCAPSULES

Patent Proprietor:

Firmenich SA

Opponent:

Givaudan SA

Headword:

Polyurea microcapsules/Firmenich

Relevant legal provisions:

EPC Art. 54, 56, 84 RPBA 2020 Art. 13(2)

Keyword:

Novelty - main request (no) - auxiliary request 1 (no)
Inventive step - auxiliary requests 2-5 (no) - auxiliary
request 8 (yes)
Claims - clarity and conciseness - auxiliary requests 6, 7 (no)
Amendment after notification of Art. 15(1) RPBA communication
- exceptional circumstances (yes)

Decisions cited:

T 0261/19

Catchword:



Beschwerdekammern Boards of Appeal

Chambres de recours

Boards of Appeal of the European Patent Office Richard-Reitzner-Allee 8 85540 Haar GERMANY Tel. +49 (0)89 2399-0

Case Number: T 0981/22 - 3.3.05

DECISION
of Technical Board of Appeal 3.3.05
of 5 September 2024

Appellant: Givaudan SA

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 21 February 2022 rejecting the opposition filed against European patent No. 3280525 pursuant to Article

101(2) EPC.

Composition of the Board:

Chair E. Bendl

Members: S. Besselmann

R. Winkelhofer

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Summary of Facts and Submissions

- I. The opponent's appeal is against the opposition division's decision to reject the opposition against European patent EP 3 280 525 B1. The patent in suit concerns a process for preparing polyurea microcapsules.
- II. Claim 1 as granted (main request) reads as follows:

 "A process for the preparation of polyurea

 microcapsules comprising the following steps:

 a) dissolving at least one aromatic polyisocyanate

 having at least two isocyanate groups in a perfume
 containing oil to form an oil phase;

 b) dispersing the oil phase obtained in step a) into an

 aqueous solution comprising emulsifier to form an oil
 in-water emulsion;

 c) adding to the oil-in-water emulsion obtained in step

 b) a mixture of two polyamines in a respective molar

 ratio comprised between 95:5 to 5:95, to form a
 - d) optionally drying the slurry."

microcapsule slurry;

- III. Claim 1 of auxiliary request 1 differs from claim 1 of the main request in that the aromatic polyisocyanate in step a) has at least three isocyanate groups.
- IV. Claim 1 of auxiliary request 2 differs from claim 1 of the main request in that the following expression is added at the end of the claim:
 "wherein the polyamines of the mixture of two polyamines are selected from the group consisting of 1,2-diaminopropane, 1,2-diaminoethane, diethylenetriamine, guanidine, melamine, tris-(2-aminoethyl)amine, N,N'-bis(3-aminopropyl)-

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ethylenediamine, N, N, N, N'-tetrakis (3-aminopropyl)-1,4-butanediamine and 3,5-diamino-1,2,4-triazole."

- V. In claim 1 of auxiliary request 3, the amendments of auxiliary requests 1 and 2 have been combined.
- VI. Claim 1 of auxiliary request 4 differs from claim 1 of the main request in that the following expression is added at the end of the claim:

 "wherein the process is characterized in that is [sic] the mixture of two polyamines comprises 3,5-diamino-1,2,4-triazole."
- VII. Claim 1 of auxiliary request 5 differs from claim 1 of the main request in that the following expression is added at the end of the claim:

 "wherein the process is characterized in that the mixture of two polyamines consists of guanidine and 3,5-diamino-1,2,4-triazole."

Claim 1 of auxiliary request 6 differs from claim 1 of auxiliary request 5 in that the following expression is added at the end of the claim:

"in a respective molar ratio comprised between 30:70 to 20:80."

- VIII. Claim 1 of auxiliary request 7 differs from claim 1 of the main request in that the following expression is added at the end of the claim:

 "wherein the perfume comprises less than 20% of solvent, this percentage being defined by weight relative to the total weight of the perfume."
- IX. Independent claims 1 and 6 of auxiliary request 8 read as follows (with markings by the board to show the differences to the main request):

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- "1. A process for the preparation of polyurea microcapsules comprising the following steps:
- a) dissolving at least one aromatic polyisocyanate having at least two isocyanate groups in a perfume-containing oil to form an oil phase;
- b) dispersing the oil phase obtained in step a) into an aqueous solution comprising emulsifier to form an oil-in-water emulsion;
- c) adding to the oil-in-water emulsion obtained in step
- b) a mixture of two polyamines in a respective molar ratio comprised between 95:5 to 5:95, to form a microcapsule slurry;
- d) optionally drying the slurry; wherein the process is characterized in that the mixture of two polyamines consists of guanidine and 3,5-diamino-1,2,4-triazole in a respective molar ratio comprised between 30:70 to 20:80."
- "6. Polyurea core-shell microcapsules obtainable by a process as defined in any one of the claims 1 to 5 comprising
- a) an oil-based core comprising a perfume;
- b) a shell formed by the interfacial polymerization of at least one aromatic polyisocyante [sic] having at least two isocyanate groups and a mixture of two polyamines; and
- c) an emulsifier on the surface of the shell, wherein the mixture of two polyamines consists of guanidine and 3,5-diamino-1,2,4-triazole in a respective molar ratio comprised between 30:70 to 20:80."

Claims 2-5 of auxiliary request 8 relate to particular embodiments of the process of claim 1. Claims 7-10, relating to a perfuming composition or a perfuming consumer product, depend directly or indirectly on

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claim 6. Claim 11 relates to a use of polyurea microcapsules as defined in claim 6 or of a perfuming composition as defined in claim 7.

- X. The following documents are of relevance here:
 - D1 US 2013/0313734 Al
 - D6 US 2011/0071064 Al
 - D6a Lupranate M20 MATERIAL SAFETY DATA SHEET,
 March 2008, revised April 2011
 - D8 US 2014/0323376 Al
- XI. The opponent's (appellant's) arguments relevant to the present decision can be summarised as follows.

The subject-matter of claim 1 of the main request lacked novelty in view of D6.

The additional feature in auxiliary request 1 was also already known from D6.

Starting from document D6, the additional feature in auxiliary requests 2 and 3 was an obvious alternative.

The subject-matter of claim 1 of auxiliary requests 4 and 5 lacked an inventive step starting from D8.

Auxiliary request 6 did not meet the requirements of inventive step either. The claimed subject-matter was an obvious modification of Example 6 of D8, considering that the objective technical problem was that of providing an alternative.

The amendment in claim 1 of auxiliary request 7 gave rise to a lack of clarity.

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Auxiliary request 8 was late filed and should not be taken into account. The claimed subject-matter lacked an inventive step in view of D8 for the same reason as auxiliary request 6 (this was the only substantive objection against auxiliary request 8).

XII. The patent proprietor's (respondent's) arguments relevant to the present decision can be summarised as follows.

Document D6 did not take away novelty of the subjectmatter of claim 1 of the main request, *inter alia*, because D6 did not disclose a perfume-containing oil.

Claim 1 in all the auxiliary requests involved an inventive step. Document D1 was the closest prior art. Documents D6 and D8 were less relevant. They did not address the technical problem underlying the patent in suit and could not be used as alternative starting points to assess inventive step.

The additional feature in claim 1 of auxiliary request 7 was clear because the claim referred to a perfume-containing oil, not to a perfume per se.

Auxiliary request 8 should be admitted into the proceedings because it was a timely reaction to a fresh objection raised by the board. It was *prima facie* allowable.

XIII. The appellant requests that the decision under appeal be set aside and amended such that the patent be revoked.

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- XIV. The patent proprietor requests that the appeal be dismissed (main request) or that the patent be maintained in amended form on the basis of one of auxiliary requests 1-7 of 31 October 2022 or auxiliary request 8 of 27 May 2024.
- XV. In the alternative, both parties request remittal to the opposition division to deal with the auxiliary requests.

Reasons for the Decision

Main request

- 1. Novelty
- 1.1 According to the impugned decision, the subject-matter of claim 1 differed from Example 5 of D6 in that a perfume-containing oil was used. The fragrance emulsion of D6, which contained a fragrance mixture containing "Blue Touch Tom" fragrance and "Neobee" (80/20), did not anticipate a "perfume-containing oil", a perfume being a more complex mixture, including not only fragrances but also essential oils, aroma compounds, fixatives and solvents.
- 1.2 However, the terms "perfume" and "fragrance" are used interchangeably in D6. This is apparent, inter alia, from paragraph [0057] in which both terms are used, from paragraph [0061] in conjunction with the title of Table 1, and from the reference to "perfumery performance results" in Examples 9 and 10 of D6 (relating to the same fragrance mixture as Example 5).

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Furthermore, the function of the "fragrance" in D6, namely to provide the consumer with a pleasurable fragrance during and after using the product or to mask unpleasant odours from some of the functional ingredients used in the same product (paragraph [0098]), corresponds to the function of the perfume in the patent in suit (paragraph [0026]). Consequently, the presence of "Blue Touch Tom" fragrance anticipates the presence of a perfume.

There is consequently no need to consider other evidence submitted by the appellant to show that using both terms interchangeably corresponded to the usual practice.

- 1.3 D6 thus discloses a perfume-containing oil (i.e. the mixture of "Blue Touch Tom" and "Neobee"), also considering that D6 explicitly states that these compounds (in conjunction with Lupranate M20, which is an aromatic polyisocyanate, see D6a) form the oil phase.
- 1.4 The appellant provided proof that "Lupranate M20" was diphenylmethane diisocyanate (material safety data sheet D6a). D6a shows an "original date" of March 2008 (before the filing date of D6) and a "revision date" of April 2011 (after the publication date of D6). The respondent's assertion that the product name "Lupranate M20" might have referred to a different product when D6 was filed is therefore without basis.
- 1.5 Example 5 of D6 furthermore anticipates using a polyamine mixture, as required in step c) of claim 1. It is not contested that the 80:20 mixture (by weight) of HMDA (hexamethylenediamine) and Jeffamine EDR-176 falls within the claimed molar range of 95:5 to 5:95.

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There is no indication in D6 that further polyamines would be present. In any case, this would not be excluded by the wording of claim 1.

- 1.6 D6 explicitly states that the oil phase is emulsified into the aqueous phase, meaning that an oil-in-water emulsion is obtained, as is necessary to obtain the oil drops which are eventually encapsulated.
- 1.7 It was not contested that a surfactant (an emulsifier) is present in the aqueous phase.
- 1.8 For these reasons, the subject-matter of claim 1 lacks novelty in view of Example 5 of D6.

Remittal

2. Both parties were of the opinion that the case should be remitted to the opposition division to deal with the auxiliary requests. However, there is no room for a remittal to the opposition division in this case.

Under Article 11 RPBA, a board must decide a case itself unless there are "special reasons" for remittal. Such reasons do not exist here. Parties have no absolute right to have each and every matter examined at two levels of jurisdiction (Case Law of the Boards of Appeal of the EPO, 10th edn., 2022, V.A.9.2.1). Furthermore, remitting the case would mean that the board would not have an opportunity to deal with novelty and inventive step, which had been dealt with in the impugned decision. This would be contrary to the purpose of the appeal proceedings to review the decision under appeal in a judicial manner.

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Auxiliary request 1

- 3. Novelty
- 3.1 In claim 1, it is specified that the aromatic polyisocyanate has at least three isocyanate groups.
- 3.2 According to the respondent, D6 did not disclose the presence of three isocyanate groups but merely 2.7, having regard to paragraph [0046].
- 3.3 However, the value of 2.7 is an average and implies that compounds with more than two isocyanate groups, i.e. at least three, are present. This is also clear from paragraph [0019] and the formula shown in paragraph [0017] of D6.
- 3.4 The subject-matter of claim 1 therefore lacks novelty over Example 5 of D6.

Auxiliary request 2

- 4. Novelty
- 4.1 It was common ground that polyamines are used in Example 5 of D6 which are different from those specified in claim 1 of auxiliary request 2. Novelty was not contested.
- 5. Inventive step
- 5.1 The patent in suit relates to the production of oilcontaining core-shell microcapsules, in particular

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perfume-containing microcapsules with improved olfactive performance (paragraph [0001]).

5.2 D6 relates to polyurea microcapsules in which active materials have been encapsulated (paragraph [0001]). D6 thus relates to a similar purpose and is a suitable starting point for assessing inventive step.

In contrast to the respondent's view, it is not necessary that the technical problem addressed in the prior art which serves as the starting point be identical to the subjective technical problem indicated in the patent in suit.

Moreover, in a case in which inventive step is denied in view of a piece of prior art, "the choice of that prior art as the starting point for the assessment of inventive step needs no specific justification as the claimed invention must, as a general rule, be non-obvious having regard to any prior art" (T 261/19 Reasons 2.5).

- 5.3 The patent in suit addresses the technical problem of providing polyurea microcapsules having improved breakability.
- As a solution to this technical problem, the claimed process is proposed in which the polyamines are selected from the group specified in the claim.
- 5.5 However, it cannot be derived from the patent in suit that a technical effect, such as an improved breakability, can be obtained using this group of polyamines, compared with Example 5 of D6. For instance, it has not been shown that using 1,2-diaminopropane and 1,2-diaminoethane provides any

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advantage over the known use of hexamethylenediamine and polyetheramine as adjunct cross-linkers (Example 5 of D6).

- 5.6 The objective technical problem can thus merely be seen as providing an alternative.
- 5.7 The skilled person wishing to provide an alternative would by taught by D6 itself that, for instance, 1,2-diaminoethane (paragraph [0022]) and diethylenetriamine (paragraph [0023]) are suitable polyamines.
- 5.8 The skilled person would therefore arrive at a process within the scope of claim 1 without performing an inventive step.

Auxiliary request 3

6. Auxiliary request 3 is a combination of auxiliary requests 1 and 2 and thus also lacks an inventive step in view of D6 for the same reasons.

Auxiliary requests 4 and 5

7. In claim 1 of auxiliary request 4, it is specified that the mixture of two polyamines comprises 3,5-diamino-1,2,4-triazole (i.e. guanazole), based on claim 2 as granted. In claim 1 of auxiliary request 5, it is specified that the mixture of two polyamines consists of guanidine and 3,5-diamino-1,2,4-triazole (i.e. guanazole), based on claim 3 as granted.

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- 8. Novelty
- 8.1 Novelty was not contested. It was common ground that D6 does not mention the polyamine (or polyamines) of claim 1 of auxiliary requests 4 and 5.
- 9. Inventive step
- 9.1 As outlined above, the patent in suit relates to the production of oil-containing core-shell microcapsules, in particular perfume-containing microcapsules with improved olfactive performance (paragraph [0001]).
- 9.2 The general considerations regarding the choice of the starting point for assessing inventive step (see point 5.2) also apply here. D8 aims to provide waterdispersible core-shell type microcapsules capable of increasing the long-lastingness of the perception of the odour of fragrance aldehydes and ketones upon exposure to light (paragraph [0001]) and thus relates to a similar purpose. Capsules F4 and G4 of Example 6 are particularly relevant and constitute a suitable starting point for assessing inventive step.
- 9.3 The process of Example 6 of D8 comprises:
 - dissolving a polyisocyanate in 2-phenylethyl 2-oxo-2-phenylacetate (pro-fragrance) to form the organic (i.e. oil, see e.g. the footnote of Table V) phase
 - introducing the organic phase into an aqueous solution of PVOH, PVOH being an emulsifier
 - preparing an emulsion
 - adding a solution of guanidine carbonate and/or quanazole to the emulsion

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9.4 Moreover, and irrespective of whether the term

"perfume" includes a pro-fragrance (as indicated in the
patent in suit, see paragraph [0027], last sentence), a
minor amount of the corresponding fragrance (i.e. 2phenylacetaldehyde) is inevitably present, considering
that it is the function of the pro-fragrance to release
the fragrance upon exposure to light (paragraphs
[0004], [0017] and [0018] of D8).

For preparing Capsules F4 and G4, the polyisocyanate is Desmodur N100 (i.e. an aliphatic polyisocyanate), and a solution of guanidine carbonate and guanazole is used, these polyamines being present in different amounts (Table V). It was not contested that the resulting molar ratios fall within the broad range claimed.

- 9.5 The patent in suit addresses the technical problem of providing polyurea microcapsules with improved breakability.
- 9.6 As the solution to this technical problem, the claimed process is proposed in which the polyisocyanate is an aromatic polyisocyanate.

However, the examples in the patent show that samples A and B (according to the invention) are very similar to the respective samples obtained using the aliphatic polyisocyanate (I, J) in terms of perfume release after rubbing (Figure 1 in conjunction with Tables 2 and 3). Samples A and B are also similar to samples G and L in which only guanidine is used. Using only guanidine is already known from Example 6 of D8 (see e.g. Capsule E4). Any difference is within the error margin and would not be significant. This is even more so considering that the amount of polyamines (as the cross-linking agent) in samples A and B was less. This

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might contribute to higher breakability and thus higher peak intensity and might therefore also have affected the results.

- 9.7 For these reasons, the technical problem posed (see point 9.5) has not been solved across the whole scope claimed (of either auxiliary request 4 or 5). The objective technical problem is thus merely that of providing an alternative.
- 9.8 It is already known from Example 6 of D8 itself that Takenate D-110N, an aromatic polyisocyanate, may alternatively be used as the polyisocyanate.
- 9.9 The skilled person starting from Example 6 of D8, and faced with the technical problem of providing an alternative, would thus readily use Takenate D-110N in conjunction with a mixture of guanidine carbonate and guanazole. They would thus arrive at a process within the scope of claim 1 of auxiliary requests 4 and 5 without performing an inventive step.

Auxiliary request 6

- 10. Article 84 EPC
- 10.1 Claim 1 of auxiliary request 6, inter alia, specifies that the mixture of two polyamines consists of guanidine and 3,5-diamino-1,2,4-triazole in a respective molar ratio comprised between 30:70 to 20:80. Consequently, the feature "in a respective molar ratio comprised between 95:5 to 5:95", which is additionally present in the claim, is superfluous, the claim thus lacking conciseness. The same consideration

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- applies to product-by-process claim 6, in which a corresponding amendment has been made.
- 10.2 The respondent submitted that the potential issue was already present in the claims as granted. They were also of the view that even if the broader range was potentially superfluous, this should not give rise to an objection because the claim was nevertheless clear.
- 10.3 These arguments are not convincing. As follows from Article 84 EPC, clarity and conciseness are separate requirements. Moreover, the broader molar ratio in claim 1 as granted became superfluous only when the narrower molar ratio of claim 4 as granted was inserted in claim 1. This issue was not present in the claims as granted.
- 10.4 In summary, the amendments made in auxiliary request 6 have the consequence that the requirements of Article 84 EPC are not met.

Auxiliary request 7

- 11. Clarity
- 11.1 Compared with claim 1 as granted, an additional feature taken from the description has been inserted to specify that the perfume comprises less than 20% of solvent by weight, relative to the total weight of the perfume.
- 11.2 The respondent was of the view that the claim was clear because the skilled person would understand that the perfume-containing oil had to comprise less than 20 wt.% of solvent.

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- However, the claim refers to the total weight of the "perfume". It is not clear how the total weight of "perfume", which is present as a perfume-containing oil, is to be calculated. For instance, it is not clear if the total amount of the oil phase is to be taken into account. Furthermore, it is not clear on which basis the total amount of "perfume" and the associated solvents can be clearly determined when other ingredients are present (see paragraphs [0020]-[0025]) and when an ingredient provides more than one benefit (paragraph [0026]).
- 11.4 The requirements of Article 84 EPC are therefore not met.

Auxiliary request 8

- 12. Article 13(2) RPBA
- 12.1 As argued by the respondent, there are exceptional circumstances within the meaning of Article 13(2) RPBA justifying that auxiliary request 8 be taken into account.

Auxiliary request 8 was filed as a prompt response to the board's preliminary opinion in which a fresh objection of lack of clarity had been raised (point 21 of the communication pursuant to Article 15(1) RPBA). Auxiliary request 8 is based on auxiliary request 6 in which the superfluous expression has been deleted in claims 1 and 6. This amendment is straightforward and does not present the other party or the board with an unexpected situation.

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- 13. Inventive step
- 13.1 Reference is made to the remarks under point 9. above, in particular points 9.1 to 9.6.
- 13.2 The appellant was of the view that the claims of auxiliary request 8 also lacked an inventive step over D8, the objective technical problem being to provide an alternative.

However, in contrast to the conclusion reached on auxiliary requests 4 and 5 (see point 9.7), the more ambitious technical problem of providing polyurea microcapsules with improved breakability (see point 9.5) is considered solved by the process of claim 1 of auxiliary request 8 for the following reasons.

13.3 The proposed solution is the claimed process in which the mixture of two polyamines consists of guanidine and 3,5-diamino-1,2,4-triazole (i.e. guanazole) in a respective molar ratio comprised between 30:70 to 20:80 and in which the polyisocyanate is an aromatic polyisocyanate.

These distinguishing features were not contested. In the preparation of the known Capsules F4 and G4 of D8, the proportion of guanazole is lower than stipulated in the claim, and the polyisocyanate is an aliphatic polyisocyanate.

13.4 The examples of the patent in suit support that the distinguishing features contribute to an improved perfume release after rubbing, i.e. breakability. As shown in Figure 1, Capsule C, involving Takenate (i.e.

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an aromatic polyisocyanate) and a mixture of guanidine and guanazole in a molar ratio of 25:75 (75 mol% guanazole), has an improved perfume release after rubbing compared with a corresponding sample based on an aliphatic polyisocyanate (Desmodur) and compared with samples having a lower proportion of 25 or 50 mol% guanazole, these lower proportions of guanazole being representative of those known from D8. The effect is clearly outside the respective error margins shown in Figure 1.

In contrast to the appellant's arguments, the effect cannot be explained merely by the fact that the total amount of polyamines was low because some of the other – now comparative – samples related to a similarly low amount of polyamines. The samples represented in Figure 1 have been obtained following the same general procedure (paragraphs [0085]-[0087]), and there is no apparent reason why the data points within this figure could not be compared with each other.

In contrast to the appellant's view, the other figures in the patent in suit do not raise doubt as to the relevance of Figure 1. The peak intensity values in Figure 1 and Figure 2 are indeed different, but this is due to the peak intensity being shown in relative units. It is thus not permissible to directly compare the respective values. Figure 4 cannot be directly compared with Figure 1 either because Figure 4 relates to a different test, namely a sensory analysis with a low number of panellists (n=7) (paragraph [0095]) with low statistical significance, whereas the results of Figure 1 demonstrate a statistically significant effect determined by use of a quadrupole mass spectrometer.

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The appellant also submitted that Figure 2 showed that Capsules D, E and F - outside the scope of the claim - exhibited an even better breakability than Capsule C illustrating the claimed subject-matter. However, Capsules D, E and F do not reproduce the prior art. It is therefore irrelevant whether they constitute even better embodiments which are not claimed.

There is no proof for the appellant's assertion that the beneficial effect of combining guanidine carbonate and 3,5-diamino-1,2,4-triazole in the respective ratios would only be obtained with the specific aromatic polyisocyanate used in the examples and could not be generalised to other aromatic polyisocyanates.

- 13.5 In light of the above, the technical problem posed, namely to provide polyurea microcapsules with improved breakability, is thus to be regarded as solved.
- 13.6 There is no teaching in the prior art that would have prompted the skilled person, faced with the technical problem posed, to select a molar ratio of the two polyamines within the narrow range claimed and to combine this with an *aromatic* polyisocyanate.
- 13.7 The subject-matter of claim 1 therefore involves an inventive step.
- 13.8 The same conclusion applies to claim 6, relating to the polyurea core-shell microcapsules obtained by the process of claim 1. It also applies to claims 2-5 and 7-11, which directly or indirectly refer back to claim 1 or 6.

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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 Opposition Division with the order to maintain the patent on the basis of auxiliary request 8 as submitted on 27 May 2024 and the description to be adapted.

The Registrar:

The Chair:



C. Vodz E. Bendl

Decision electronically authenticated