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Datasheet for the decision of 26 November 2015

Case Number: T 1911/13 - 3.3.10

Application Number: 08870617.1

Publication Number: 2229350

IPC: C07C41/36, C07C43/13

Language of the proceedings: EN

Title of invention:
PURIFICATION OF PROPYLENE GLYCOL MONOALKYL ETHER

Patent Proprietor:
Lyondell Chemical Technology, L.P.

Opponent:
The Dow Chemical Company

Headword:

Relevant legal provisions:
EPC Art. 100(a), 56
RPBA Art. 13(1), 13(3)

Keyword:
Inventive step - (no) main and first auxiliary request
Late-filed auxiliary request -
admitted (no) second auxiliary request

Decisions cited:

Catchword:

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Case Number: T 1911/13 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 26 November 2015

Appellant: (Patent Proprietor)	Lyondell Chemical Technology, L.P. Two Greenville Crossing 4001 Kennett Pike Suite 238 Greenville, DE 19807 (US)
Representative:	Gaverini, Gaetano Luigi Attilio Basell Poliolefine Italia S.r.l. Intellectual Property P.le Donegani, 12 44122 Ferrara (IT)
Respondent: (Opponent)	The Dow Chemical Company 2030 Dow Center Midland, MI 48674 (US)
Representative:	Chung, Hsu Min HGF Limited 140 London Wall London EC2Y 5DN (GB)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 4 July 2013 revoking European patent No. 2229350 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	P. Gryczka
Members:	R. Pérez Carlón
	T. Bokor

Summary of Facts and Submissions

I. The appellant (patent proprietor) lodged an appeal against the decision of the opposition division to revoke European patent No. 2 229 350.

II. Notice of opposition had been filed on the grounds of insufficiency of disclosure (Article 100(b) EPC) and lack of inventive step (Article 100(a) EPC).

III. The documents filed during the opposition proceedings included the following:

D1 US 2004/0211658
D2 US 3,367,847
D3 WO 99/58483

IV. With the statement setting out the grounds of appeal, the appellant filed a declaration by David W. Leyshon, which contained additional experimental evidence.

V. The opposition division concluded that the subject-matter of the two requests then pending was not inventive. Document D1 was the closest prior art, the problem underlying the claimed invention was to provide a further method of removing carbonyl impurities from propylene glycol monoalkyl ether fractions and the solution, which was characterised by contacting said fractions with a carbon adsorbent selected from activated carbons or charcoals, was not inventive having regard to D2 or D3.

VI. The main request of the appellant is to maintain the patent as granted. Since claim 1 of the European patent specification published as EP 2 229 350 contains a printing error, this decision would refer to the

wording of claim 1 according to the decision of the examining division to grant a European patent, which is the following:

"A method of purifying a propylene glycol monoalkyl ether containing carbonyl impurities which comprises contacting the propylene glycol monoalkyl ether in the liquid phase with a carbon adsorbent, selected from activated carbons or charcoals and recovering a purified propylene glycol monoalkyl ether product having a decreased carbonyl impurities content."

VII. The first auxiliary request is identical to the auxiliary request pending during the opposition proceedings. Claim 1 of this request contains all the features of claim 1 of the main request and, in addition, the following:

"*and a UV absorbance, at 245 nm, of 0.5 or less*".

VIII. During the oral proceedings before the board of appeal, which took place on 26 November 2015, the appellant filed a second auxiliary request, whose claim 1 includes, in addition to the features of claim 1 of the main request, the following:

"characterised by the fact that the activated carbon adsorbent is Fritrassorb(R) 600."

IX. The arguments of the appellant relevant for the decision were the following:

Document D1 was the closest prior art. The problem underlying the claimed invention was to provide a method of removing carbonyl impurities from a propylene glycol monoalkyl ether with improved efficiency and the

solution, which was characterised by using a carbon adsorbent, was not obvious having regard to any of documents D2 or D3, as these documents referred to glycols, which were a different class of compounds. Document D2 did not disclose the efficiency of the treatment with activated carbon and the process of D3 did not lead to an efficient separation. The appellant further relied on the additional effect of avoiding undesirable by-products, as shown in the experimental evidence filed as a declaration by David W. Leyshon, which should be admitted into the proceedings. Said experimental evidence had been filed as a reaction to the decision of the opposition division at the earliest possible moment during these appeal proceedings, thus allowing the respondent sufficient time to prepare its case. For all these reasons, the subject-matter of claim 1 of the main request and that of the first auxiliary request were inventive.

Claim 1 of the second auxiliary request merely restricted the subject-matter of claim 1 to the specific adsorbent which led to the best results. Such an amendment should have been expected, was simple and did not introduce any further issue into the proceedings. For these reasons, the second auxiliary request should be admitted into the proceedings.

X. The arguments of the respondent relevant for the decision were the following:

The respondent also considered document D1 as the closest prior art, but did not agree that the technical problem put forward by the appellant was effectively solved. If, nevertheless, the problem solved by the claimed invention were to be considered as providing an improvement over the purification method disclosed in

D1, such an improvement was obvious having regard to the teaching of either of documents D2 or D3. These documents disclosed a method for removing carbonyl compounds from glycols which, according to D1, could be purified by the same method as propylene glycol monoalkyl ethers, with activated carbon and achieving very good results. For these reasons, the subject-matter of claim 1 of the main request and of the first auxiliary request was not inventive.

The experimental evidence filed as a declaration by David W. Leyshon should not be admitted into the proceedings, as it was filed late and was not relevant.

Auxiliary request 2 had been filed during the oral proceedings before the board, and resulted from the combination of the subject-matter of claim 1 as granted with a feature taken from the description. The respondent could not have expected this and was not prepared for addressing it during the oral proceedings. For this reason, this request should not be admitted into the proceedings.

XI. The final requests of the parties were the following:

- The appellant requested that the decision under appeal be set aside, and the opposition be rejected (main request), or that the patent be maintained in an amended form on the basis of the auxiliary request filed with the grounds of appeal dated 14 November 2013, or on the basis of the second auxiliary request filed during the oral proceedings.

- The respondent requested that the appeal be dismissed.

XII. At the end of the oral proceedings, the decision was announced.

Reasons for the Decision

1. The appeal is admissible.

Evidence filed during appeal proceedings, admissibility

2. The respondent requested that the experimental evidence filed as a declaration by David W. Leyshon not be admitted into the proceedings, as said evidence was late-filed. D1 was known to the patentee during the opposition proceedings, and the appellant had thus ample opportunity to file comparative data with regard to D1 earlier.

The board considers, however, that the experimental data (hereafter D4) was filed as a reaction to the decision of the opposition division, at the earliest possible moment during these appeal proceedings (i.e with the statement of grounds of appeal), and that the opponent had ample opportunity to study it and had provided arguments on its content. For that reason, the board admits D4 into the appeal proceedings.

Main request, inventive step

3. Claim 1 of the patent as granted is directed to a method of purifying a propylene glycol monoalkyl ether containing carbonyl impurities which comprises contacting it with an activated carbon adsorbent

selected from activated carbons or charcoals.

4. Closest prior art

The parties and the opposition division considered that document D1 represented the closest prior art and the board sees no reason to differ.

Document D1 discloses a method of purifying solvents such as propylene oxide monoalkyl ethers [0012] by contacting said impure solvents, in the liquid phase [0010], with an acidic ion exchange resin [0004] which removes carbonyl impurities such as formaldehyde, acetaldehyde, acetone and propionaldehyde [0017].

Document D1 does not disclose contacting propylene oxide monoalkyl ethers with a carbon adsorbent selected from activated carbons or charcoals.

5. Technical problem underlying the invention

The appellant considered that the technical problem underlying the claimed invention was providing a method of removing carbonyl impurities from propylene glycol monoalkyl ether with improved efficiency, thus improving UV absorbance and colour properties (see grounds of appeal, page 3).

6. Solution

The claimed solution is the method of purifying propylene glycol monoalkyl ether according to claim 1, which is characterised in that it comprises contacting propylene glycol monoalkyl ether with a carbon adsorbent selected from activated carbons and

charcoals.

7. Success

In favour of the appellant, it will be considered that the technical problem underlying the claimed invention as formulated in point 4. above is solved in all aspects.

8. It remains thus to be decided whether or not the proposed solution to the objective problem underlying the patent in suit is obvious from the prior art.

8.1 Document D1 discloses a method of purifying propylene glycol monoalkyl ethers containing carbonyl impurities by contacting said ethers, in the liquid phase, with an ionic interchange resin such as Amberlyst resins.

Carbonyl impurities enhance the ultraviolet absorbance of glycols and thus worsen the quality of the final product (see paragraph [0005] of the patent in suit and column 5, lines 38-40 of document D2).

According to document D1, alkylene glycol monoalkyl ethers can be purified by the same process as the corresponding alkylene glycols [0011], [0012].

Lastly, D1 discloses that acidic resins can remove, at the most, 80% of the impurities present in the starting feed [0017]. The skilled person thus recognises that the method of D1 leaves room for improvement.

Trying to obtain an improved process, the skilled person would turn to documents relating to the purification of similar compounds removing, with good results, the same impurities.

As D1 teaches that alkylene glycols and alkylene glycol monoalkyl ethers such as propylene glycol monoalkyl ethers could be purified by the same procedures (see paragraph [0012]), the skilled person would then turn to documents relating to the purification of glycols containing these impurities with good results, and would thus be led to D2 and D3.

8.2 Document D2

Document D2 discloses that the filtration of ethylene glycol through activated carbon "removes trace amounts of impurities remaining in the ethylene glycol which affect its ultraviolet light transmission" (column 2, lines 26-36). Said impurities are aldehydes and esters (column 5, lines 38-40). Pure product, transparent at 220 nm, is obtained (column 7, lines 52-61).

The skilled person thus knows from D2 that activated carbon is a suitable material for purifying ethylene glycol very effectively, in the liquid phase, yielding a transparent product and from D1 that ethylene glycol can be purified analogously to propylene glycol monoalkyl ethers. Trying to provide a method superior to that of D1, the skilled person would consider that the method of D2, which implies contacting a feed with activated carbon, represents an improvement over that of D1, and would thus arrive at the claimed invention. Although the skilled person will not have complete certainty that the purification of propylene glycol monoalkyl ethers would be as good as that of glycol, he will have a reasonable expectation of success having regard to the results obtained in D2.

The subject-matter of claim 1 of the main request is

thus not inventive having regard to the teaching of documents D1 and D2.

8.3 Document D3

Document D3 discloses a process for treating organic liquids which achieves superior ultraviolet light transmitting characteristics (claim 1) by contacting said organic liquids with charcoal. Among the preferred organic liquids, D3 refers to alcohols, ethers and glycols (claim 7). On page 4, lines 12-14, D3 discloses that "by far the material having the most effect on the uv light transmittance is the activated carbon known as Norit(R)" and table II shows that "a wide variety of carbons may be used for improving the uv transmittance of glycols".

Propylene glycol monoalkyl ethers have both an alcohol and an ether functionality. According to D3, activated carbon is very effective for removing impurities which affect UV light transmittance from organic solvents of the alcohol, ether and glycol type, glycols being the most preferred type of solvents. As explained above, it is further known from D1 that glycols can be purified by the same type of processes as propylene glycol monoalkyl ethers.

Trying to find a process with improved efficiency, the skilled person would look at processes which achieve, as that of D3, excellent results for similar compounds, and would apply said process to propylene glycol monoalkyl ethers. For this reason, the skilled person will combine the teaching of document D3 with that of the closest prior art document D1 and thus arrive at the present invention. As in the case of D2, although the skilled person would not have complete certitude

that the method of D3 would lead to an improvement, he would have a reasonable expectation that it would be the case.

For these reasons, the board concludes that the subject-matter of claim 1 of the main request is not inventive, contrary to Article 56 EPC, also having regard to the teaching of documents D1 and D3.

8.4 The appellant argued that glycols and glycol ethers belonged to different classes of compounds and, for that reason, the skilled person would not have considered combining the teaching of D1 with that of D2 or D3.

However, document D1 discloses that glycols and glycol ethers such as propylene glycol monoalkyl ethers can be purified by the same techniques. For this reason alone, the skilled person would apply the methods of purification disclosed in documents D2 or D3 to the purification of propylene glycol monoalkyl ethers.

8.5 The appellant further argued that document D1 was directed to the purification of feeds different from those required by the claimed invention. D1 was concerned with the purification of solvents used in the extractive distillation of alkylene oxides and only aimed at reducing its level of impurities so that they could be reused in said extractive distillation. Thus, D1 did not aim at achieving a high level of purification. In contrast, the claimed invention was directed to the preparation of very pure compounds suitable for other applications and, for that reason, the skilled person would not have combined the teaching of D1 with either of D2 or D3 as they are aimed at different objectives.

However, the intended use of the propylene glycol monoalkyl ether is not a feature of claim 1. Further, the applicant cannot argue that document D1 is the closest prior art, that the problem underlying the claimed invention is providing a process which is more effective than that of D1 and, at the same time, that the skilled person would not have considered modifying it.

8.6 The appellant argued that the treatment with activated carbon disclosed in document D2 was merely optional, as D2 was concerned with the purification of glycols by fractional distillation. The table in column 7 did not indicate the level of impurities present before the purification and, for that reason, document D2 did not disclose the efficiency of the activated carbon treatment only.

However, document D2 explicitly mentions that the step of contacting ethylene glycol with activated carbon removes trace amounts of impurities which effect ultra-violet light transmittance of ethylene glycol (column 5, lines 37-40). Thus, D2 discloses that this step is efficient for the same purpose as that sought by the claimed invention. On lines 45-46, document D2 discloses that such an activated carbon treatment can also be used to update commercially available or off grade ethylene glycol, which indicates that treatment with activated carbon is suitable for removing impurities independently from their concentration in the starting product, and that this treatment is disclosed independently from the fractional distillation step. Lastly, claim 1 does not exclude further purification steps previous to contacting with a carbon adsorbent. For all these reasons, the argument

of the appellant is not convincing.

8.7 The appellant argued that the results summarised in Table I of D3 compared carbon adsorbents with adsorbents which were completely inefficient as they could not remove any impurity having UV absorption, and, for that reason, the skilled person would not have extracted from D3 the teaching that carbon adsorbents could provide an efficient purification method.

However, the best results in Table II of document D3 disclose an increase of the transmittance at 250 nm from 43.2% to 93.1%, which corresponds in terms of absorbance to a decrease from 0.36 to 0.03, and is thus comparable to the results disclosed in the examples of the patent in suit and in D4.

8.8 The appellant also relied on an additional effect, namely that of avoiding undesirable by-product formation, as mentioned in point 9. of D4.

Firstly, it is well known that Amberlyst type resins can be used as catalysts in various reactions due to their strong acidic properties. The skilled person would thus expect that an inert substrate such as activated carbon would not induce, in contrast to Amberlyst, any chemical decomposition of the product to be purified.

Secondly, acetone, acetaldehyde and methyl formate absorb in the ultraviolet due to the presence of a C=O group, and even methanol has ultraviolet absorbance. For this reason, the problem of reducing the amount of side products formed is only an aspect of that of improving the UV absorbance of propylene glycol monoalkyl ether as defined in point 5. above.

Lastly, the reasons for combining the teaching of documents D2 or D3 with that of D1 have already been explained. The board has concluded that the skilled person would combine their teachings with a reasonable expectation of success, and any further effect which might also have been achieved as the direct consequence of the non-inventive solution can only be considered as a bonus effect.

8.9 The appellant argued that, starting from D1, the skilled person would merely follow the teaching in paragraph [0018] and try to increase the efficiency of the separation by changing the acid resin, the concentration of carbonyl compounds in the feed, the presence of other impurities, the temperature, the flow rate and the age of the catalyst.

However, these variables are disclosed within the context of the invention of D1, and the skilled person knows that their optimisation would, at the most, remove 80% of the impurities. The skilled person, trying to improve the purification beyond the best result of D1, would thus necessarily look for the necessary information elsewhere.

8.10 The appellant further argued that document D1 was an improvement of the older technology represented by D2 and D3. For that reason, the person skilled in the art would not combine D1 with D2 or D3, since it would represent a step back, from which he would expect a decrease or, at the most, no gain in efficiency.

However, there is no reason why a more recent technology should necessarily be superior in every aspect than any older equivalent. This argument is thus

not convincing.

8.11 The board concludes that the subject-matter of claim 1 of the main request is not inventive, with the consequence that the ground under Article 100(a) EPC precludes the maintenance of the patent as granted.

First auxiliary request, inventive step

9. Claim 1 of the first auxiliary request limits the subject-matter of claim 1 of the main request by further including the feature "and a UV absorbance, at 245 nm, of 0.5 or less".

The appellant acknowledged that this feature reflects the inevitable consequence of the remaining features of claim 1 and that it does not change the inventive step analysis with respect to the main request. The board sees no reason to differ.

The subject-matter of claim 1 of the first auxiliary request is thus not inventive for the same reasons as that of the main request, with the consequence that the first auxiliary request is not allowable.

Second auxiliary request, admission

10. According to Article 12(2) RPBA, the statement of grounds of appeal must contain an appellant's complete case. If, at a later stage of the proceedings, the appellant wants other requests to be considered, admission of these requests into the proceedings is a matter of discretion for the board of appeal (Article 13(1) RPBA).

11. The first auxiliary request was filed at the oral proceedings before the board and the respondent objected to its admission into the proceedings. The amendment consisted on introducing into claim 1 a feature taken from the description of the patent in suit. Even though the amendment consisted of limiting claim 1 to the carbon adsorbent which provided the best result, such an amendment could not have been foreseen, and the respondent considered that it was not in a position to address this request during the oral proceedings.

In addition, the board notes that the activated carbon adsorbent required by claim 1 of the second auxiliary request is defined by a trademark, which is in general not allowable due to lack of clarity.

12. As the filing of the second auxiliary request is not a response to any new procedural situation, the respondent could not be expected to address it during the oral proceedings, and said request is not clearly allowable since it appears to introduce further deficiencies, the board does not admit this request into the proceedings (Article 13(1) and (3) RPBA).

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

C. Rodríguez Rodríguez P. Gryczka

Decision electronically authenticated