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**Datasheet for the decision
of 1 October 2019**

Case Number: T 1952/16 - 3.3.10

Application Number: 02254650.1

Publication Number: 1275633

IPC: C07C51/09, C07C67/327,
C07C51/50

Language of the proceedings: EN

Title of invention:

Method for decomposition of Michael type adduct

Patent Proprietor:

NIPPON SHOKUBAI CO., LTD.

Opponent:

THE DOW CHEMICAL COMPANY

Headword:

Relevant legal provisions:

EPC Art. 56, 113(1)
RPBA Art. 13(3)

Keyword:

Decisions cited:

T 1606/11

Catchword:



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Case Number: T 1952/16 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 1 October 2019

Appellant: THE DOW CHEMICAL COMPANY
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Decision under appeal: **Interlocutory decision of the Opposition**
Division of the European Patent Office posted on
27 June 2016 concerning maintenance of the
European Patent No. 1275633 in amended form.

Composition of the Board:

Chairman P. Gryczka
Members: R. Pérez Carlón
F. Blumer

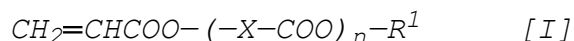
Summary of Facts and Submissions

- I. The appeal lies from the decision of the opposition division on the maintenance of European patent No. 1 275 633 in the form of the main request then pending. This is the second appeal on this case following T 1606/11.
- II. Notice of opposition had been filed on the grounds of added subject-matter (Article 100(c) EPC), insufficiency of disclosure (Article 100(b) EPC) and lack of novelty and inventive step (Article 100(a) EPC).
- III. The documents filed during the opposition proceedings include the following:
- D2 WO 00/53560
- D16 Experimental Report on European Patent No. 1275633, filed with a letter dated 8 April 2016
- D17 Declaration of Alan Sopchik dated 23 November 2016
- IV. In previous decision T 1606/11, the board decided that the subject-matter of the main request, which is also the main request in these appeal proceedings, had the required basis in the application as originally filed and was novel over D2.

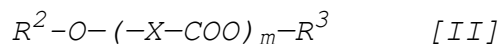
According to T 1606/11, the method of claim 1 differed from that disclosed in D2 by virtue of the relative amount of nitroxyl compound based on the total quantity of the adduct.

V. Claim 1 of the main request reads as follows:

"A method for decomposing an adduct of acrylic acid and/or an acrylic ester having the formula [I]



wherein n is an integer from 1 to 5, R¹ is a hydrogen atom or an alkyl group, and X is -CH₂CH₂- or -CH(CH₃)-, providing that X may be the same or different when n is greater than 1, or having the formula [II]



wherein m is an integer from 1 to 5, R² and R³ are each, independently, a hydrogen atom or an alkyl group, and X is -CH₂CH₂- or -CH(CH₃)-, providing that X may be the same or different when m is greater than 1, characterised in that the adduct of formula [I] or formula [II] is treated with a nitroxyl compound in an amount of 1-10% by weight based on the total quantity of the adduct in the presence of an alkali metal or alkaline earth metal salt at a temperature of 100°C to 180°C, thereby causing the adduct to decompose into acrylic acid and/or an acrylic ester and/or an alcohol."

VI. Following remittal, the opposition division concluded that said subject-matter was also inventive. This is the decision contested in the present appeal proceedings.

The opposition division agreed with the parties that document D2 was the closest prior art. The problem underlying the claimed invention was to provide a method for decomposing an adduct of formula [I] or [II]

with improved conversion and selectivity. This problem was credibly solved having regard to D16. The claimed solution, characterised by the amount of nitroxyl compound used in the process, would not have been obvious having regard to the prior art.

VII. The arguments of the appellant (opponent) on the issue of inventive step were as follows.

The contested decision defined the problem underlying the claimed invention as that of providing a process that allowed improved conversion and selectivity. However, such a formulation could not be allowed, since the application as originally filed did not link any of these effects to the amount of nitroxyl compound which was considered to be the solution to this problem.

However, if that formulation of the technical problem were allowable, document D16 did not show that it had been credibly solved. D16 was neither dated nor signed and thus had little probative value. It contained errors, for example the relative amount of nitroxyl compound in entries (A) to (C). It was not credible that essays carried out a decade later could have used the same starting feed. The formula provided on paragraph [0047] was not accurate with respect to selectivity coefficient. Lastly, D17 showed that no nitroxyl compound at all could have been present in example (J) of document D16, so that it should not represent the process of the closest prior art.

The appellant was not in a position to rework D16 as it lacked a detailed protocol and the chemical composition of 34% by weight of the feed was not disclosed.

Even if the problem as defined in the contested

decision were credibly solved, the claimed solution would have been obvious for the skilled person, who would have expected to improve the conversion and selectivity of the claimed method by increasing the amount of radical stabiliser. For these reasons, the claimed subject-matter was not inventive.

VIII. The arguments of the respondent (patent proprietor) were as follows.

Document D16, in particular examples (D) and (J), showed credibly that the problem as formulated in the contested decision, namely the provision of a method of decomposition of an adduct of formula [I] or [II] with improved conversion and selectivity, had been solved by the method of claim 1. The appellant's argument that D17 showed that no nitroxyl compound was present in the example (J) of document D16 could not be followed. D17 merely disclosed that 4H-TEMPO reacted under the conditions tested. However, D17 failed to show, as argued by the appellant, that no nitroxyl compound was present in the example (J) of document D16, as the product obtained had not been identified and could still contain a radical moiety. For these reasons, D16 was a valid comparison with the closest prior art.

The claimed solution, which was characterised by the relative amount of nitroxyl compound in the process, known to be a radical inhibitor, would not have been obvious for the skilled person, as the decomposition of adducts [I] or [II] did not proceed via a radical mechanism.

IX. Oral proceedings before the board of appeal took place on 1 October 2019.

X. The final requests of the parties were as follows:

- The appellant requested that the decision under appeal be set aside and that European patent No. 1 275 633 be revoked.
- The respondent requested that the appeal be dismissed or, subsidiarily, that the patent be maintained in the form of one of auxiliary requests 1 to 11, auxiliary requests 1 to 7 as filed before the opposition division with a letter dated 8 April 2016, auxiliary requests 8 and 9 as filed with a letter dated 18 March 2019, auxiliary requests 10 and 11 as filed with a letter dated 24 September 2019.

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

Reasons for the Decision

1. The appeal is admissible.

Inventive step, main request

2. Claim 1 relates to a method for decomposing an adduct of acrylic acid or an acrylic ester of formula [I] or [II]. These adducts are formally obtained by a Michael-type addition of a nucleophile (acrylate, R²O-) to the conjugated system of acrylic acid or ester. They will be referred to in this decision as "Michael adducts".

The process of claim 1 requires 1-10% by weight of a nitroxyl compound, relative to the total quantity of the adduct. It further requires an alkali metal or alkaline earth metal salt and a temperature of 100°C to

180°C.

3. Closest prior art

The opposition division and the parties considered that document D2 was the closest prior art. The board sees no reason to differ.

In previous decision T 1606/11, the board concluded that the method of claim 1 differed from that of example 1 of document D2 only in the amount of nitroxyl compound based on the total quantity of the adduct, which was between 0.24% and 0.57% by weight.

4. Technical problem underlying the invention

4.1 The respondent defined the technical problem underlying the claimed invention as providing a method for decomposing an adduct of formula [I] or [II] which made it possible to improve conversion and selectivity.

4.2 The appellant argued that this formulation of the technical problem was unallowable, since the application as originally filed did not link conversion or selectivity to the relative amount of nitroxyl compound.

However, the application as filed explicitly refers to the problem of improving conversion and selectivity in the context of decomposition of Michael adducts [I] and [II]. This problem was allegedly solved by using nitroxyl compounds (page 8, lines 22-27; page 3, last paragraph).

If a document such as D2, which discloses the use of nitroxyl compounds in the claimed process, is cited

against the patentability of the claimed invention, the board sees no reason why the respondent could not rely on the very same effect in connection with the relative amount of nitroxyl compound, which is the distinguishing feature over that document. In fact, the technical problem underlying the claimed invention remains the same, what has changed is the claimed solution to that problem.

This argument of the appellant cannot be followed.

5. Solution

The solution to this technical problem is the claimed method, characterised in that it is carried out by treating Michael adducts [I] and [II] with 1-10% by weight of nitroxyl compound based on the total quantity of the adduct.

6. Success - D16

6.1 The respondent relied on the experimental report filed as D16 before the opposition division to prove that the problem formulated above had been credibly solved.

6.2 The appellant argued that D16 did not bear either a date or signature and for this reason could not have any evidential value.

This objection was raised for the first time at the oral proceedings before the board, and is an amendment of the appellant's case (Article 13 RPBA).

The respondent argued that it was not in a position to provide a duly signed and dated version of D16 during the oral proceedings.

This late amendment to the appellant's case is thus not to be admitted following Article 13(3) RPBA, as the respondent could not address it without an adjournment of the oral proceedings (Article 113(1) EPC).

- 6.3 Examples D and J of experimental evidence D16 show the decomposition of an adduct of formula [I], after 4 hours at 140°C, in the presence of Na₂CO₃ and 4H-TEMPO. These examples differ in the amount of 4H-TEMPO: 0.8 wt% (J) and 5 wt% (D). According to D16, by increasing the amount of 4H-TEMPO the conversion went from 50% (J) to 74% (D) and the selectivity coefficient from 90% (J) to 98% (D).

The data provided thus reflect an improvement due to the relative amount of nitroxyl compound added to the reaction.

- 6.4 The appellant relied on document D17, which the board admits into the proceedings in its favour, to show that example (J) of D16 could not reflect the process of D2, as it had in fact been carried out in the absence of 4H-TEMPO.

At the oral proceedings before the board, the appellant did not wish to rely on document D17a. The issue of its admissibility can thus remain undecided.

According to D17, in a mixture of hydroquinone and 4H-TEMPO in acrylic acid, the amount of 4H-TEMPO becomes negligible after 15 minutes, even at temperatures below those used in D16. The appellant argued that this was due to the reaction of 4H-TEMPO with hydroquinone. The feeds of D16 contained hydroquinone (paragraph [0046] of the patent in suit), which inevitably reacted with

4H-TEMPO. For this reason, example (J) of D16 had not been carried out in the presence of 0.8% by weight of 4H-TEMPO, as all of it would have been consumed.

D17 does in fact disclose that the amount of 4H-TEMPO becomes minute after a short time. However, the author of D17 fails to investigate what product has been formed. If, as argued by the respondent, it esterified, the resulting ester would still be a nitroxyl compound, as required by claim 1. Thus, the data in D17 do not necessarily indicate the absence of nitroxyl compound, only the absence of 4H-TEMPO as such. This argument of the appellant is thus not convincing.

- 6.5 The appellant argued that the relative amount of nitroxyl compound in the various feeds of D16 was wrong. For this reason, the respondent could not rely on the results put forward in D16.

The mistake, acknowledged by the respondent, relates to the examples of D16 already present in the patent as granted, namely (A) to (C). However, there is no evidence that the concentration of 4H-TEMPO in the examples which could prove an effect with respect to the process of the closest prior art, namely (D) and (J), could have been erroneous.

- 6.6 The appellant further argued that it was not credible that examples (A) to (C) of D16, which are those of the patent in suit, and (D) to (J), carried out more than a decade later, could have used the same starting material. For this reason, D16 could not provide a valid comparison to the closest prior art.

However, examples (D) and (J), reflecting the effect of the claimed method, have been carried out at the same

point of time. This argument of the appellant is not convincing.

- 6.7 The appellant also argued that the formula in the patent in suit [0047] for calculating the selectivity coefficient was not sound. For this reason, the values in D16 relating to that parameter could not prove any effect.

However, it is not only clear that the formula in paragraph [0047] of the patent in suit is not correct, but also that it should relate to how much of the desired product was obtained relative to how much of the Michael adduct had reacted.

- 6.8 The appellant further argued that neither paragraph [0047] of the patent in suit nor D16 disclosed whether the values put forward related to molar quantity (in mol) or to mass ratio.

However, D16 shows an effect in terms of conversion and selectivity regardless of how the relative amount has been calculated, as both processes referred to the conversion of the same reagent into the same products. Thus this argument cannot be followed.

- 6.9 The appellant also argued that document D16 lacked a detailed protocol, which did not allow the data obtained to be reproduced.

However, D16 has been carried out according to the patent in suit. The examples of the patent required heating a feed as defined in paragraph [0046] at 140°C for 4 hours, in the presence of nitroxyl compounds and/or hydroxides. The feed contained [0046] 60% by weight acrylic acid dimer, 4% acrylic acid and 2%

hydroquinone. The board sees no reason why the appellant could not have attempted to reproduce D16, despite lacking information on the chemical nature of 34% of the feed beyond being "other high boiling impurities". Selectivity could have been calculated in the usual manner, the procedure to follow merely requires heating a round flask at 140°C for 4 hours and analysing the mixture obtained.

- 6.10 The evidence provided by the respondent as D16 is thin at best. It does not state who, when or where it has been carried out, lacks any experimental detail, does not provide the chromatograms of the mixtures obtained, let alone the eluting conditions or chromatograph used. In fact it is no more than some numbers on a table.

However, if the results of D16 were not accurate, the appellant could have proved them wrong by simple minimal experimentation. The appellant has however chosen not to provide any evidence in this respect. The appellant has also failed to convincingly cast doubt on the inadequacy of the evidence provided as D16, it merely referred to its obvious weakness.

Opposition appeal proceedings are adversarial, and it is up to the parties to make their cases. The respondent has provided evidence showing an effect derived from the distinguishing feature, whereas the appellant has provided none in that respect.

Under these circumstances, the board concludes, in agreement with the decision under appeal, that the problem as formulated by the respondent (see point 4.1 above) has been credibly solved by the method for decomposing an adduct of claim 1.

7. It thus remains to be decided whether the proposed solution to the objective problem defined above would have been obvious for the skilled person in view of the prior art.

It is not disputed that nitroxyl compounds are known stabilisers against radical processes, such as polymerisation.

This mode of action could explain, as argued by the appellant, that selectivity towards acrylic acid or acrylic ester would have been increased, as higher amounts of nitroxyl radicals would have prevented loss of product due to subsequent polymerisation more efficiently than a smaller quantity.

However, there is no reason why the conversion of Michael adduct could have been increased by enhancing the amount of nitroxyl compound in the reaction, as the decomposition reaction defined in claim 1 is not expected to follow a radical pathway.

Both reagents [I] or [II] and reaction products acrylic acid and acrylic esters are unsaturated and prone to polymerisation in the same manner. The stabilisation due to nitroxyl compounds against polymerisation would thus have affected both the starting material and the product and should not have changed the reaction conversion.

The skilled person would thus have found no reason to increase the relative amount of nitroxyl radical used in the method of D2 in order to achieve a higher conversion.

8. There is no argument on file relating to use claim 11 beyond those already put forward with respect to claim 1, nor is any objection immediately apparent. Claims 2 to 10, dependent on claim 1, are inventive for the same reasons as the latter.

For these reasons, the subject-matter claimed in the main request is inventive (Article 56 EPC).

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



B. ter Heijden

P. Gryczka

Decision electronically authenticated