

Internal distribution code:

- (A) [] Publication in OJ
(B) [] To Chairmen and Members
(C) [X] To Chairmen

D E C I S I O N
of 19 July 1995

Case Number: T 0771/92 - 3.3.1

Application Number: 85306281.8

Publication Number: 0216966

IPC: C07C 102/06

Language of the proceedings: EN

Title of invention:

Improved process for the production of N-substituted (meth) acrylamides from (meth) acrylates & amines over a metal oxide or alkoxide catalyst

Patentee:

TEXACO DEVELOPMENT CORPORATION

Opponent:

Röhm GmbH

Headword:

(Meth)acrylamides/TEXACO

Relevant legal provisions:

EPC Art. 54(1)(2), 56, 100(b), 123(2)(3)

Keyword:

"Novelty (yes) - after amendment"
"inventive step (yes) - non-obvious alternative process"

Decisions cited:

G 0010/91, G 0004/92, T 0912/91

Catchword:

-

Case Number: T 0771/92 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 19 July 1995

Appellant: Röhms GmbH
(Opponent) Kirschenallee
D-64293 Darmstadt (DE)

Representative: -

Respondent: TEXACO DEVELOPMENT CORPORATION
(Proprietor of the patent) 2000 Westchester Avenue
White Plains
New York 10650 (US)

Representative: Winkler, Andreas, Dr
FORRESTER & BOEMHERT
Franz-Joseph-Strasse 38
D-80801 München (DE)

Decision under appeal: Decision of the Opposition Division of the European Patent Office dated 29 June 1992 rejecting the opposition filed against European patent No. 0 216 966 pursuant to Article 102(2) EPC.

Composition of the Board:

Chairman: A. J. Nuss
Members: P. P. Bracke
R. E. Teschemacher

Summary of Facts and Submissions

- I. European patent application No. 85 306 281.8, filed on 4 September 1985, was granted as European patent No. 0 216 966.
- II. The patent was opposed. Revocation of the patent was requested on the grounds of lack of novelty and lack of inventive step.

Of the cited prior art documents the following remained relevant during the appeal procedure:

- (1) DE-A-3 048 020 and
- (2) DE-A-3 123 970.

- III. By a decision of 29 June 1992 the Opposition Division rejected the opposition.

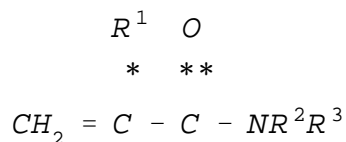
It was essentially held that the process claimed according to the set of claims as granted was novel, since it differed from the processes described in either document (1) or (2) by the fact that, prior to the reaction of the amine with the (meth)acrylate ester, the feedstocks were treated with a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium.

Moreover, since the claimed process was based on the idea that the Michael reaction could be suppressed by removing water from the feedstocks before bringing them together for reaction and it was not made plausible by the cited state of the art that such an idea was obvious to a skilled person, the process was considered also to involve an inventive step.

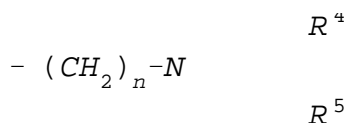
IV. The Appellant (Opponent) lodged an appeal against this decision.

During the oral proceedings held on 19 July 1995, at which the duly summoned Appellant was not represented, the Respondent (Proprietor) filed a set of 7 claims, with the only independent claim reading as follows (emphasis added):

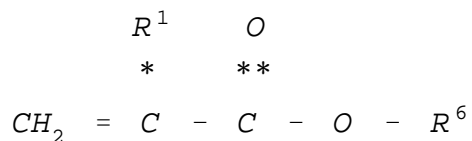
"1. A process for the preparation of N-substituted (meth)acrylamides of the formula:



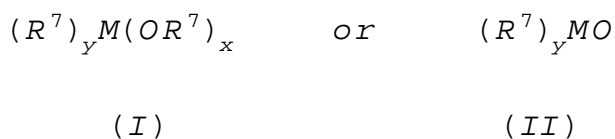
where R¹ is hydrogen or methyl, R² is hydrogen or alkyl having 1 to 4 carbon atoms and R³ is alkyl, aryl, alkaryl, aralkyl or alkoxyalkyl having 1 to 20 carbon atoms or



wherein n is 2 to 6, and R⁴ and R⁵, which may be the same or different are alkyl having 1 to 4 carbon atoms, or R⁴ and R⁵, together with the N atom to which they are attached, are a substituted or unsubstituted morpholine, pyrrolidine or piperidine ring, which process comprises reacting **a feedstock** of an acrylate ester of the formula:



wherein R¹ has the meaning given above and R⁶ is alkyl having 1 to 4 carbon atoms, with **a feedstock** of an amine of the formula HNR²R³, wherein R² and R³ have the meanings given above, in the presence of an alkyl metal oxide, alkyl metal alkoxide or metal alkoxide catalyst having the formula:



wherein M is lanthanum, niobium, tantalum, copper, zinc, tin, lead, antimony or bismuth, R⁷ is alkyl having 1 to 4 carbon atoms, x is 1 to 5 and y is 0 to 5 and the sum of x and y is 2 to 5, depending on the valence of the metal, for formula I and y is 1 to 3 depending on the valence of the metal, for formula II, characterised **in that the said feedstocks contain water, and have been treated, prior to the step of reacting the feedstocks**, with a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium, as a **drying agent**."

V. In his written submissions the Appellant argued that in the processes according to documents (1) and (2) the irreversible reaction of the catalysts with water is faster than the reaction of the amine with the acrylate

ester and that, therefore, the reactants are also dried, prior to the said reaction. He concluded that, consequently, the process according to the claim as granted was anticipated by those documents.

He also submitted that in the process according to the patent in suit as well as in the process according to documents (1) and (2) the formation of Michael adducts is suppressed by reacting the (meth)acrylate ester and the amine in the presence of the same mixture of two catalysts and that no inventive step could be seen in bringing the feedstocks into contact with one of those catalysts, namely, a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium, prior to reacting them in the presence of the second catalyst, instead of directly reacting the feedstocks in the presence of a mixture of both catalysts.

Moreover, the Appellant contended that the claimed process could not be carried out.

VI. Both in the written procedure and during the oral proceedings the Respondent contested the allegation that the claimed process was anticipated by either of documents (1) or (2), since a preliminary drying of the reactants, prior to the step of reacting the feedstocks, was not mentioned therein.

Additionally, the Respondent submitted that a skilled person would have no reason to believe that the undesired Michael reaction was catalysed by the presence of water and, consequently, that there would be no reason to suppose that it would be possible to improve the reaction by drying the feedstocks, prior to

reacting them, let alone by using a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium as drying agent of the feedstocks.

- VII. Though the Appellant did not make an express request, his submissions are to be interpreted as meaning that the setting-aside of the impugned decision and the revocation of the patent is being sought.

The Respondent requested that the appeal be dismissed and that the patent be maintained on the basis of the claims as submitted during the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. *Amendments*

Claim 1 differs from Claim 1 as granted (see the emphasised parts) in that it has been specified that

- (a) the pretreated feedstocks are those containing the acrylate ester and the amine respectively, and
- (b) the tetralkoxides of silicon, titanium or zirconium are used as drying agents in pretreating the said water-containing feedstocks, prior to the step of reacting them,

as was implicitly disclosed in the originally filed application (see, particularly, page 3, lines 10 to 17, page 8, lines 15 to 18, and page 9, lines 4 to 12).

Claims 2 to 7 correspond with originally filed Claims 3 to 8 (i.e. Claims 2 to 7 as granted).

Since those amendments do not add subject-matter extending beyond the content of the application as filed and also do not lead to an extension of the protection conferred by the patent in suit, the requirements of Article 123(2) and (3) EPC are met.

3. *Novelty*

According to the claimed process a water-containing feedstock of an acrylate ester and a water-containing feedstock of an amine are treated with a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium, as a drying agent, prior to the step of reacting the feedstocks to convert a (meth)acrylate ester with an amine into a N-substituted (meth)acrylamide in the presence of a metal oxide, an alkyl metal alkoxide or a metal alkoxide catalyst.

Documents (1) and (2) also describe processes for converting a (meth)acrylate ester with an amine into a N-substituted (meth)acrylamide. However, those documents, which only mention the bringing together of a mixture of a (meth)acrylate ester and an amine with a mixture of a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium and a metal oxide, an alkyl metal alkoxide or a metal alkoxide [see document (1), example 1 and document (2), examples 12 to 14)], are silent about the possibility of bringing the methacrylate-containing feedstock and the amine-containing feedstock together separately with one of those catalysts, prior to the step of reacting the said feedstocks, let alone about

using a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium as a drying agent for these feedstocks.

Consequently, the Board concludes that Claim 1 is novel over the teachings of documents (1) and (2) (Article 54(1) and (2) EPC).

4. *Inventive step*

4.1 The Board considers that both documents (1) and (2) qualify as closest state of the art. This has also been accepted by the Opposition Division, the Appellant and the Respondent.

4.2 Both documents (1) and (2) are concerned with a process for preparing N-substituted (meth)acrylamides by converting a (meth)acrylate ester with an amine in the presence of a catalyst, wherein the formation of Michael adducts, obtained by the addition reaction of an amine to the (meth)acrylic double bond, is suppressed. See document (1), page 1, lines 4 to 16, and document (2), page 1, lines 3 to 6, and page 3, lines 24 to 30.

According to document (1) the Michael reaction is suppressed by using a catalyst consisting of an alkyl titanate and a dialkyltin oxide (see page 2, lines 1 to 16, the examples and Claim 1), and according to document (2) such formation is suppressed by using as catalyst compounds of metals of Group IV, lead, tantalum or zinc, for example, by using a tetralkoxide of titanium in combination with a dialkyltin oxide as catalyst (see page 4, lines 6 to 15, page 9, lines 18 to 20, and Examples 12 to 14).

4.3 In view of this, and in the absence of any advantages shown for the claimed process over that described in either document (1) or (2), the problem underlying the invention must be seen in the development of an alternative process for preparing N-substituted acrylamides by converting a (meth)acrylate ester with an amine, wherein the amount of by-product formed by the Michael reaction is substantially reduced (page 1, lines 35 to 36).

This problem is to be solved by the process as defined in the present Claim 1 (see point IV above).

The experimental data disclosed in Examples IV and VI of the patent in suit provide sufficient evidence that the stated problem has indeed been solved by the process with the features required by the present Claim 1. This was not contested by the Appellant.

4.4 It remains to be decided whether, in the light of the teachings of documents (1) and (2), a skilled person would have brought the water-containing amine feedstock and the water-containing acrylate ester feedstock into contact with a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium prior to reacting the said feedstocks, with a view to suppressing the Michael reaction.

4.5 The Appellant is of the opinion that a skilled person would have done so (see point V above). However, as will be apparent from the following considerations, the Board cannot find any support for this.

4.6 First of all, document (1) is completely silent about the possibility of bringing the feedstocks into contact with any of the catalysts as well as about the possibility of using water-containing feedstocks. The only information provided by this document is that to be found in Example 1 (see page 4, lines 16 to 20) and Example 2 (see page 5, lines 17 to 18) referring back to Example 1, namely that dibutyltin oxide together with a titanate are added **to the reaction mixture**.

As regards bringing the feedstocks into contact with any of the catalysts it is only stated in document (2), page 11, lines 8 to 10, and in Example 1, page 14, lines 10 to 14, that the catalyst may be present in the ester-containing feedstock.

4.7 Consequently, it was nowhere suggested in the two prior art documents that the amine-containing feedstock as well as the ester-containing feedstock be brought together with any of the catalysts prior to reacting them in order to reduce or suppress the Michael reaction due to the presence of water.

4.8 Furthermore, Appellant's view that no inventive step can be seen in bringing the feedstocks into contact with a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium prior to reacting them in the presence of a second catalyst instead of bringing the feedstocks into contact with such a tetralkoxide and the said second catalyst only in the reaction medium itself, can only be considered as an argument based on hindsight, because it is the essence of the invention that the Michael reaction is suppressed by drying each feedstock by removing water, prior to reacting them, with a

specific group of known drying agents and it was nowhere suggested in any of the available prior art documents that the presence of water would have an influence on the formation of Michael reaction adducts, let alone that by treating each feedstock with a C₁ to C₄ tetralkoxide of silicon, titanium or zirconium high yields of N-substituted acrylamides with only minor amounts of undesirable Michael adduct would be obtained.

- 4.9 The Board therefore concludes that since the process claimed in Claim 1 is not obvious in the light of the cited state of the art, it involves an inventive step in the sense of Article 56 EPC.
- 4.10 The patentability of the process Claims 2 to 7 follows from that of Claim 1 on which they depend.
5. Therefore, the two stated grounds for revoking the patent in suit do not prejudice the maintenance of the patent on the basis of Claims 1 to 7 submitted during the oral proceedings.
6. The Appellant's objection in the "Statement of the Grounds of Appeal", page 3, last paragraph, that the invention is technically not feasible, was not supported by any experimental evidence and, consequently, cannot be taken into consideration. Moreover, if this statement were to be interpreted as an objection under Article 100(b) EPC, this statement would have to be considered as a fresh ground for opposition, which might only be considered with the approval of the Patentee (G 10/91 OJ EPO, 1993, 420). The latter, however, considered this to be an objection

the Appellant was not entitled to raise (see Respondent's letter dated 14 April 1993, in particular, page 3, first paragraph).

7. The Board holds that the present decision to maintain the patent on the basis of a set of claims amended during oral proceedings in the absence of the Appellant does not conflict with the principle described in the decision of the Enlarged Board of Appeal G 4/92 (OJ EPO 1994, 149), whereby a decision may not be based on new facts put forward for the first time during the oral proceedings. It has been held that the submission of restricted claims is neither a fact nor can it be evidence within the meaning of the above decision (T 912/91, dated 25 October 1994, not for publication in OJ EPO). In any case the Appellant could not be taken by surprise by the amendments made. The Appellant had reasonably to expect that the Respondent would try to overcome the objections made. The amended set of claims is based on the wording of the set of claims according to the "first auxiliary request", filed with the letter of 14 April 1993, and the amendments result from the novelty objection made by the Appellant during the written procedure. Consequently, the absence of the Appellant at the oral proceedings did not prevent the Board from taking this decision.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent in the following version:
 - Description of the patent as granted,
 - Claims 1 to 7 as submitted during the oral proceedings.

The Registrar:

The Chairman:

P. Martorana

A. Nuss