

Internal distribution code:

- (A) [] Publication in OJ
(B) [X] To Chairmen and Members
(C) [] To Chairmen
(D) [] No distribution

D E C I S I O N
of 1 December 2003

Case Number: T 0211/01 - 3.3.6

Application Number: 92907846.7

Publication Number: 0573578

IPC: C10L 1/22

Language of the proceedings: EN

Title of invention:

A process for preparing halogen-free, deposit-control fuel additives comprising a hydroxypolyalkene amine

Patentee:

Dover Chemical Company Inc.

Opponents:

BASF Aktiengesellschaft
Chevron Chemical Company LLC

Headword:

Hydroxypolyalkene amine/DOVER

Relevant legal provisions:

EPC Art. 54(3), 56, 114(2)

Keyword:

"New ground of opposition - not to be considered"
"Novelty (all requests): yes"
"Problem solution-approach - defective disclosure is not a promising starting point for the assessment of inventive step"
"Inventive step (all requests): no - obvious aggregation of known process steps"

Decisions cited:

-

Catchword:

A document which is obviously defective as would be readily recognised by those skilled in the art when trying to reproduce its disclosure cannot be taken as the most promising and appropriate starting point for the assessment of inventive step (points 2.1.3 - 2.1.5).



Case Number: T 0211/01 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 1 December 2003

Appellant: BASF Aktiengesellschaft
(Opponent I) D-67056 Ludwigshafen (DE)

Representative: Schweiger, Georg, Dr.
Reitstötter, Kinzebach & Partner
Postfach 86 06 49
D-81633 München (DE)

Appellant: Chevron Chemical Company LLC
(Opponent II) 2613 Camino Ramon
San Ramon, CA 94583-4289 (US)

Representative: Benedum, Ulrich Max, Dr.
Haseltine Lake Partners
Motorama Haus 502
Rosenheimer Strasse 30
D-81669 München (DE)

Respondent: Dover Chemical Company Inc.
(Proprietor of the patent) 1201 North Orange Street
Wilmington, Delaware 19801 (US)

Representative: Gille Hrabal Struck Neidlein Prop Roos
Patentanwälte
Brucknerstrasse 20
D-40593 Düsseldorf (DE)

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 13 December 2000
rejecting the opposition filed against European
patent No. 0573578 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: P. Krasa
Members: G. Dischinger-Höppler
C. Holtz

Summary of Facts and Submissions

I. This appeal is from the decision of the Opposition Division to reject the oppositions and to maintain European patent No. 0 573 578 on the basis of five claims as granted, the only independent claim reading:

"1. A process for preparing a halogen-free hydroxy-polyalkene amine composition by first reacting a polybutene having an average molecular weight of from 400 to 2200 or a polypropylene having a number of average molecular weight of from 170 to 2200 with hydrogen peroxide in the presence of an organic carboxylic acid and an acid catalyst and then reacting the obtained epoxidized polybutene or epoxidized polypropylene at a temperature of from 185 to 300°C in a closed vessel at a pressure of up to about 2.1 MPa (300 psi) with an excess of from 2 to 20 moles per mole of said epoxidized polybutene or epoxidized polypropylene of at least one type of an amine compound being a primary or secondary monoamine, a primary or secondary diamine or a primary or secondary polyamine."

The dependent claims refer to preferred embodiments of the process of Claim 1.

II. Two notices of opposition, based on lack of novelty and inventive step (Articles 100(a), 54(3) and 56 EPC) cited *inter alia* the following documents:

(1) WO-A-92/12221;

- (3) "Oxidations in Organic Chemistry", M. Hudlicky, ACS Monograph 186, American Chemical Society, Washington D.C. 1990, pages 10 to 12;
- (4) "Organic Peroxides, Vol. 1", D. Swern, Wiley-Interscience, J. Wiley and Sons, New York 1970, pages 340 to 369;
- (5) US-A-3 794 586 and
- (6) DE-A-2 520 267.

III. In its decision, the Opposition Division dismissed the objection under Article 123(2) EPC raised late during the opposition proceedings and found that the subject-matter of the claims as granted was based on the application as filed. An objection concerning validity of the priorities claimed by document (1) was held to be irrelevant to the question of novelty since document (1) did not disclose any use of an acid catalyst for epoxidation. Further it was found that a skilled person would not have used the amination conditions specified in document (6) in the process of document (5) in the expectation of improving the yield of amination since document (5) taught that using temperatures above 180°C would lower the yield. Therefore, the claimed subject-matter was held to be not obvious to a person skilled in the art.

IV. During the appeal proceedings the Respondent (Proprietor) filed amended sets of claims in three auxiliary requests under cover of letters dated 13 and 24 November 2003.

Claim 1 of auxiliary request I differs from that of the main request only in that the acid catalyst is specified as being "selected from the group consisting of methanesulfonic acid, toluenesulfonic acid, sulfuric acid and phosphoric acid and mixtures thereof".

Claim 1 of auxiliary request II differs from that of the main request in that the term "an acid catalyst" is replaced by "a mixture of phosphoric and sulfuric acid".

Claim 1 of auxiliary request III differs from that of auxiliary request II by substituting the range of temperature of "from 185 to 300°C" by a range of "from 230 to 285°C".

- V. Oral proceedings before the Board of Appeal were held on 1 December 2003 in the course of which the Board addressed the question of whether document (5) was a reasonable starting point for the assessment of inventive step and the Respondent filed a further set of amended claims in auxiliary request IV.

Claim 1 thereof differs from that of auxiliary request III by the features "wherein the amount of hydrogen peroxide is 1.5 to 2.0 moles per mole of polyalkene based upon the number of average molecular weight of the polyalkene, and the amount of the carboxylic acid is 0.15 to 0.5 moles per mole of polyalkene based on the number average molecular weight of the polyalkene" introduced between the terms "sulfuric acid" and "and then reacting".

VI. The Appellants (Opponents) in writing and at the oral proceedings submitted inter alia the following arguments:

- The claimed subject-matter was anticipated by document (1) since the acetic acid contained in the 40% peracetic acid (hereinafter PAA) solution was an acid catalyst within the definition given in the patent in suit.
- The subject-matter of Claim 1 was not inventive in view of document (5) which turned out to be unfeasible in respect of amination. It was obvious to those skilled in the art to apply the more severe conditions of document (6) in order to perform amination. If, alternatively, document (6) was used as the closest prior art, it was obvious to use a different olefinic substrate in order to provide an alternative hydroxyalkene amine product.

VII. The Respondent did not agree with the assessment of the late-filed ground of opposition under Article 100(c) EPC as regards the main request and argued in essence as follows:

- The subject-matter of Claim 1 was novel over the disclosure of document (1) since it did not cover the presence of a preformed PAA during the epoxidation but instead an acid catalyst which was not present in document (1). The acetic acid contained in the PAA was a solvent and did not act as a catalyst.

- Document (5) was the only prior art on file which related to the products aimed at in the patent in suit. Nevertheless, it was admitted that such products were known in the art and obtainable by prior art processes referred to in the patent in suit.

- Starting from document (5) as the closest prior art, the problem solved by the claimed process was not only to improve the yield of amination - which was achieved by the particular amination conditions - but also a simplified epoxidation step by omitting a separate step of preparing PAA from hydrogen peroxide.

- As regards the epoxidation step, the proposed solution was not obvious since in the process disclosed in document (5) no acid catalyst was present and both, documents (5) and (6) taught the use of preformed PAA. Further, the claimed *in situ* formation of PAA in the presence of polyalkylene was not suggested by the prior art.

- Regarding the amination step, it was not obvious to combine documents (5) and (6) since they related to different starting materials. Further, there was no reason to select from document (6) a reaction temperature of more than 185°C with a view to improving the amination yield while document (5) recommended not working at reaction temperatures above 180°C.

VIII. The Appellants requested that the decision under appeal be set aside and that the patent be revoked.

The Respondent requested that the appeals be dismissed, alternatively, that the decision under appeal be set aside and that the patent be maintained on the basis of either of the auxiliary requests 1 to 3 filed with the letters of 13 and 24 November 2003 or auxiliary request 4, filed in the oral proceedings.

Reasons for the Decision

1. *Novelty (all requests)*

The Board is satisfied that the claimed process is not anticipated by the cited prior art. While there is no need to give further details here since the appeals fail for lack of inventive step, the Board nevertheless deems it appropriate to deal briefly with that issue.

1.1 Lack of novelty had been objected only in view of document (1) which also discloses a two step process for producing hydroxypolyalkene amines by first reacting polyolefine with an oxidising agent to give an epoxide which is then reacted with an amine under particular conditions. It is uncontested by the parties that examples 7 to 11 of document (1) describe amination conditions corresponding to the second step in Claim 1 of the main request. Examples 7 and 8 start out from a commercially available polyisobutene epoxide and examples 9 to 11 start out from a polyisobutene epoxide derived from polyisobutene commercially available under the trade name "Ultravis 30". The

epoxidation of Ultravis 30 is disclosed in example 1, according to which 150.5 grams of 40% PAA in combination with 4.2 grams sodium acetate is used for oxidation. This corresponds to the statement in document (1) that the preferred oxidising agent is PAA, generally used as a 40% PAA solution together with about a 5% equivalent of sodium acetate as compared to the PAA (page 6, lines 24 to 27).

- 1.2 Solutions containing 40% wt of PAA are generally known in the art and commercially available. Typical solutions contain in addition 40% wt acetic acid, 5% wt of hydrogen peroxide and 1% wt of sulfuric acid, the latter as an acid catalyst which catalyses the formation of PAA in the equilibrium with acetic acid. It is further known that, for particular uses where the acid is undesirable, sodium acetate can be added to neutralise the acid catalyst in the solution (document (4), page 341, lines 9 to 17 and 32 to 35, page 342, second paragraph, and page 367, last paragraph, to page 368, line 14).

Document (1) does not specify the PAA solution it uses, but a person skilled in the art reading document (1) will realise that if the 40% PAA solution mentioned in document (1) ever contained any acid catalyst, this will have been neutralised by the addition of sodium acetate and will no longer be present.

- 1.3 The Appellants argued that the acetic acid present in the 40% PAA solution would also perform as an acid catalyst.

1.4 However, acetic acid is a carboxylic acid and the only one present in a conventional 40% PAA solution. In contrast, Claim 1 requires the presence of two acids, a carboxylic acid and an acid catalyst which are different as is corroborated by the description of the patent in suit (page 7, lines 22 to 26). It is nowhere suggested in the patent in suit that those acids might be represented by one and the same component nor does document (1) propose that any equilibrium acetic acid present in the PAA solution mentioned in document (1) might perform as an acid catalyst for whatever reaction.

1.5 The Board concludes, therefore, that the subject-matter of Claim 1 has to be regarded as being novel.

2. *Inventive Step (main request)*

2.1 Closest prior art and technical problem

2.1.1 The patent in suit and in particular the subject-matter of Claim 1 relates to a process for preparing a halogen-free hydroxypolyalkene amine composition (page 2, lines 5 to 11).

The object of the patent in suit is stated to consist in providing a further process for the preparation of halogen-free hydroxypolyalkene amines (page 2, lines 46 to 47). The Respondent admitted that such products were disclosed not only in document (5) but were also known from other prior art under Article 54(2) EPC referred to in the patent in suit under the section "Background of the Invention" (see in particular page 2, lines 25 to 29).

2.1.2 The Opposition Division and the parties in their written submissions took document (5) as the starting point for investigating inventive step. This document also discloses a two-step process for producing the same product as in the patent in suit, namely a hydroxyalkyl-substituted polyamine which is halogen-free, by first reacting a polyolefin with a 40% PAA solution containing 5% equivalent of sodium acetate as compared with the PAA and then reacting about equimolar amounts of the obtained polyolefin epoxide with a polyamine under atmospheric pressure at a temperature of 15 to 180°C (column 2, lines 28 to 60). Six examples (examples 3 to 8) indicate that the conversion of the amination reaction, based on the epoxide, is near 100%.

2.1.3 The Respondent, in the patent in suit (page 15, line 20, to page 16, line 7) and during the examining proceedings (Exhibit D filed under cover of a letter dated 14 August 1996), provided evidence that the process of document (5) did not furnish any amination reaction at all but merely an inhomogeneous mixture of starting materials. Since the Respondent's evidence was not contested by the Appellants, the Board concludes that document (5) is obviously defective as would be readily recognised by those skilled in the art when trying to reproduce its disclosure.

2.1.4 It is case law of the Boards of Appeal that the starting point for the assessment of inventive step should be one which is at least "promising" (see Case Law of the Boards of Appeal of the EPO, 4th edition, I.D.3.), in the sense that there was some probability of a skilled person arriving at the claimed invention.

2.1.5 Quite apart from the fact that a skilled person would normally not consider an obviously defective disclosure at all, the Board deems it in particular artificial to select such defective disclosure as a starting point for evaluating inventive step, when there exists other prior art which is not doubted with regard to its disclosure but is also directed to the same purpose or effect as the patent in suit. Therefore, the defective disclosure of document (5) cannot be taken as the most promising and, hence, appropriate starting point for the assessment of inventive step.

2.1.6 The Appellants, alternatively, relied on document (6) as the closest prior art. This document relates to a process for producing a mixture of alkanol amines obtained in a two-step process by first reacting a mixture of monoolefins in a known manner with PAA to form the epoxide and then reacting the epoxide with an equimolar amount or a surplus of up to 15 moles of polyamine per mole of epoxide at a temperature of 100 to 230°C at atmospheric pressure or in a closed vessel under elevated pressure (page 2, line 1, to page 3, last full paragraph).

The Respondent argued that the products obtained by the process of document (6) were totally different from those achieved in the patent in suit since the starting material in document (6) was a mixture of monoolefins having statistically distributed internal double bonds while the starting material in the patent in suit were polyolefins having a double bond in the end group. Therefore, so it argued, document (6) did not qualify as the closest prior art.

It is undisputed that the monoolefins in document (6) are derived from paraffins with up to 24 carbon atoms and enclose embodiments which are not necessarily distinguishable from a polypropylene in the lower part of the claimed molecular weight ranging from 170 to 2200. Further, the position of the double bond does not figure in Claim 1 of the patent in suit but it follows from the description that the preferred polyolefins have a double bond in position 1 or 2. Structures with a double bond in position 2 are, however, covered by the olefins used in document (6) as is apparent from the formula of the products given on page 1 wherein R_1 stands for an alkyl group having from 1 to 21 carbon atoms. Thus, the difference between the polyolefins of the patent in suit and the monoolefins of document (6) resides in the fact that in the latter case the double bonds are statistically distributed over the internal positions but not over position 1, the end position. The Board, therefore agrees with the Respondent in so far as the starting material of document (6) necessarily consists of a mixture of different olefin structures wherein those structures having a double bond in the end position are excluded.

- 2.1.7 Therefore, the Board concludes that, in the present case, the most suitable starting point is the prior art process mentioned in the patent in suit on page 2, lines 25 to 29, from which, according to the Respondent, the same products were obtainable as in the patent in suit via imination of an ozonolysis product of a polyolefin and final hydrogenation of the imine.

2.1.8 No comparative examples are on file in relation to this prior art. Therefore, the technical problem to be solved in view of such prior art cannot be considered to consist in an improvement of the yield of the final amines or a simplification of the epoxidation as was suggested by the Respondent in view of document (5) but remains what is stated in the patent in suit, i.e. providing an alternative for the preparation of halogen-free hydroxypolyalkene amine fuel additives.

The Board has no reason to doubt that this technical problem is actually solved by the two-step process of Claim 1. In particular, the examples of the patent show that almost 100% conversion into the epoxide (Example 1) and a yield of amination based on the available epoxide of as high as 81% (Example 8) can be achieved.

2.2 Evaluation of inventive step

It remains to be decided whether, in view of the cited prior art documents, it was obvious for someone skilled in the art to solve the above technical problem by the claimed means, i.e. by a process wherein an epoxide is formed in the first step by reacting the polyolefin with hydrogen peroxide in the presence of an organic carboxylic acid and an acid catalyst and the epoxide is reacted in the second step with an excess of 2 to 20 moles per mole epoxide of an amine compound in a closed vessel at a temperature of 185 to 300°C and a pressure of up to 2.1 MPa.

2.2.1 A skilled person faced with the above technical problem and looking for other processes suitable for the same object of providing halogen-free hydroxypolyalkene amines would come across the amination process of olefin epoxides disclosed in documents (5) and (6), since document (5) addresses exactly the same products to be achieved and document (6) relates to the production of alkanol amines of similar structure.

2.2.2 It would, however, disregard document (5) in respect of the amination procedure after realising that its disclosure is unfeasible and thereby also disregard the warning given therein not to apply reaction temperatures above 180°C to avoid side reactions and lowering of the yield, since it follows from the reworking of the examples of document (5) that no amination takes place even at much lower temperatures of about 110°C (see Duplications of Examples 3 and 4 in document (5) given in the patent in suit and Exhibit D filed during the examination proceedings).

On the other hand, it is apparent from document (6) that amination yields of generally above 80% are obtainable for various epoxide mixtures if a surplus of 6 or 8 moles amine per mole epoxide is used at temperatures of 115 to 230°C both in an open or closed vessel at atmospheric or elevated pressure (Examples 1 to 3) or if equimolar amounts of epoxide and amine are used at 200°C at atmospheric pressure and in the presence of glycerine as catalyst (Example 4). The best results, i.e. 90% conversion or more of the epoxide into the amine, are obtained under the amination conditions of Example 2 (mole ratio of epoxide to amine of 1:8 at 200°C and 5 to 20 atmospheres) for the same

products (A, B and E) as under the conditions of Example 1 (115 to 130°C and atmospheric pressure) or of Example 3 (25 to 30 atmospheres, i.e. 24.5 to 29.4 MPa). The Board notes that the conditions of Example 2 correspond to those presently claimed in the amination step.

No reason was given by the Respondent why the polybutene and polypropylene epoxides mentioned in Claim 1 of the patent in suit should not react in the same manner as those used in document (6). On the contrary, considering the higher sterical hindrance in the epoxides used according to document (6) due to the fact that they are derived from olefins with statistically distributed internal double bonds only, a person skilled in the art would have expected at least the same reactivity for the epoxides in the patent in suit.

The Board concludes, therefore, that a skilled person had a clear incentive for selecting from the various amination conditions mentioned in document (6) those of Example 2 in order to provide an alternative process of producing a halogen-free hydroxy polybutene or polypropylene amine composition.

Thus, no inventive merit can be attributed to the amination step of present Claim 1 alone.

2.2.3 None of the documents on file discloses the epoxidation of polybutene or polypropylene as presently claimed, i.e. with hydrogen peroxide in the presence of an organic carboxylic acid and an acid catalyst. Since no functional interaction exists between the epoxidation

and the amination, it has to be assessed whether an inventive merit can be based on the epoxidation step as such.

The Appellants argued that the advantage of this step resided in the fact that PAA was formed *in situ* which, compared to the common epoxidation routes applied in documents (5) and (6), rendered superfluous any formation of PAA in a separate step and required less amounts of carboxylic acid such as acetic acid since no equilibration with the PAA was necessary.

The *in situ* formation of PAA from hydrogen peroxide in the presence of both sulfuric acid as a catalyst and acetic is, however, known from document (3) as an alternative to a preformed PAA solution, such as the commercially available 40% PAA solutions (page 11, last full paragraph, to page 12, line 1). The advantages of avoiding separate PAA formation and savings in acetic acid as claimed by the Respondent follow as a self-evident consequence of this process.

The Respondent argued that a person skilled in the art would not have tried to carry out epoxidation of the highly reactive olefinic substrates with end group double bonds in the presence of acids due to the danger of explosion. It did not, however, support this allegation with evidence.

On the other hand, it is known from document (3) that the *in situ* formation can be carried out in the presence of the substrate to be oxidised, in which case the reaction drives to completion at moderate temperatures (page 11, loc. cit.). Further, epoxidation

of double bonds is said to be the most important application of PAA (page 12, lines 8 to 9).

Therefore, given the information in document (3), someone skilled in the art looking for alternative processes would have considered the *in situ* formation of PAA in the presence of an olefinic substrate in order to drive the reaction to completion under the most important application conditions of PAA.

Thus, no inventive merit can be attributed to the epoxidation step either.

2.2.4 The same applies to the aggregation of the amination and epoxidation steps, since no unexpected effect can be derived from it.

2.3 For these reasons, the Board concludes that, in order to solve the technical problem of providing an alternative process as set out under 4.1.8 above, a person skilled in the art would have tried the epoxide amination disclosed in document (6) with a reasonable expectation of success. It would further have considered the epoxide production disclosed in document (3) via *in situ* formation of PAA in the presence of the olefinic substrate as a variation which is suitable as an alternative for the common epoxidation with commercial PAA solutions.

Consequently, the Respondent's main request must fail since the subject-matter of Claim 1 lacks an inventive step and does not meet the requirements of Article 56 EPC.

3. *Inventive step (auxiliary requests)*

None of the additional features of the auxiliary requests 1 to 4 are suitable to support the presence of an inventive step since they are either already contained in the cited prior art and suggested for the same purposes as in the patent in suit, namely the formation of PAA and the amination of the epoxides (see documents (3) and (4) for the selection of sulfuric or phosphoric acid as the catalyst in auxiliary requests 1 and 2; document (6) for a temperature of 230°C during amination in auxiliary request 3), or are design options for which no particular advantage is apparent (amounts of hydrogen peroxide and carboxylic acid to be used in the epoxidation step according to auxiliary request 4).

Therefore, these requests must also be dismissed for lack of inventive step of the claimed subject-matter.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. European patent No. 573 578 is revoked.

The Registrar:

The Chairman:

G. Rauh

P. Krasa