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Bezeichnung der Erfindung: Preparation of alkyl glyceryl ether alcohols

Title of invention:

Titre de l'invention :

Klassifikation / Classification / Classement : C07C 43/13

ENTSCHEIDUNG / DECISION

vom / of / du 22 November 1988

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent / FMC Corporation
Titulaire du brevet :

Einsprechender / Opponent / Opposant : L'Oreal

Stichwort / Headword / Référence : Glyceryl Ether Alcohols/FMC

EPÜ / EPC / CBE Art. 56

Schlagwort / Keyword / Mot clé : "Inventive step (confirmed)"

Leitsatz / Headnote / Sommaire

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European Patent
Office

Boards of Appeal

Office européen
des brevets

Chambres de recours



Case Number : T 257 /87 3.4.1

D E C I S I O N
of the Technical Board of Appeal 3.4.1
of 22 November 1988

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Decision under appeal : Interlocutory decision of the Opposition Division of
the European Patent Office dated 05 May 1987
concerning maintenance of European Patent
No. 0 024 806 in amended form.

Composition of the Board :

Chairman : K. Jahn

Members : R. Spangenberg

G.D. Paterson

Summary of Facts and Submissions

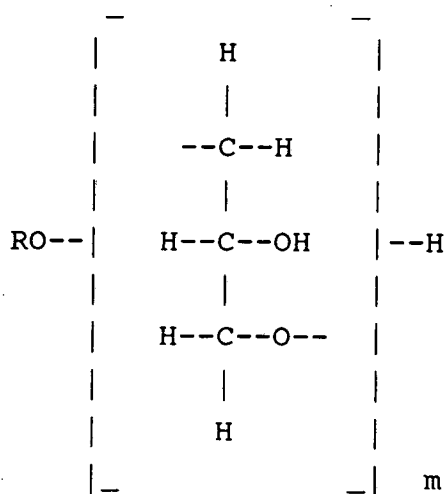
I. European patent No. 24 806 was granted on the basis of European patent application No. 80 302 443.9 filed 18 July 1980 claiming priority of two earlier applications in the USA of 27 July 1979 and 21 March 1980. The grant was published on 10 November 1982 (Bulletin 82/45).

A Notice of Opposition was filed by the Appellant on 8 August 1983. The grounds for opposition were lack of inventive step and insufficient disclosure (Article 100(a) and (b) EPC).

II. In an interlocutory decision dated 5 May 1987, the Opposition Division held that the patent in suit could be maintained in amended form on the basis of nine claims.

The independent claim read as follows:

"A process for preparing n-alkyl glyceryl ether alcohols of the general formula



wherein R is a C₁₀ - C₂₀ n-alkyl radical and m is a number from 1 to 10, by bringing together and reacting a C₁₀ - C₂₀

n-alkyl primary alcohol with crude glycidol in the presence of a basic catalyst in a reaction zone and maintaining the temperature of the reaction mass within the range from 125 to 180°C during the reaction period; characterised by extracting crude glycidol containing from 1 to 25% by weight of glycerine impurity with a non-polar non-reactive and miscible solvent selected from the group consisting of benzene, toluene, ortho-xylene, meta-xylene, para-xylene and mesitylene; obtaining from the solvent extract a crude glycidol solution containing not less than 1% by weight glycerine based on the total weight of glycidol and glycerine; reacting said alcohol and glycidol solution in a molecular ratio of alcohol to glycidol within the range of from 1:0.9 to 1:10, maintaining the resulting mass in an agitated condition at said temperature; separating said non-polar solvent from the reaction mass; and recovering a C₁₀ - C₂₀ n-alkyl glyceryl ether alcohol product."

The Opposition Division found the above claims being properly based on the application documents as filed, especially referring to page 4, lines 9 to 32 of the patent specification. The disclosure of the claimed invention was also found to comply with Article 83 EPC since the Appellant was able to repeat Example 6 of the specification without difficulty though he had obtained less favourable yields.

The Opposition Division further considered eight documents, the most important being:

- (1) US-A-3 719 636;
- (2) US-A-4 025 540;
- (3) US-A-2 089 569;
- (4) Römpps Chemie-Lexikon, 7. Auflage, Stuttgart (Franck),

1973, pages 1316 to 1319;

(5) US-A-4 105 580;

and concluded that the claimed process involved an inventive step. The closest prior art was found to be (1) which describes a process for reacting glycidol with alcohols. However, pure glycidol was reacted in polar solvents.

The problem to be solved by the claimed process was seen in suggesting a process in which crude glycidol containing large quantities of glycerine could be used as starting material without adversely affecting the yields and this problem was solved by using the non-polar solvents specified in the above Claim 1. The selection of these solvents was not rendered obvious by the disclosure of (2), relating to a different process, namely the reaction of carboxylic acids with glycidol in polar as well as non-polar solvents. Documents (3) and (5) were considered to be less relevant because (3) relates to a reaction of glycidol and alcohols in the absence of solvents and (5) teaches to use a polar solvent. Document (4) shows that it was common general knowledge that glycidol was soluble in non-polar solvents like toluene and glycerine was not.

III. On 24 June 1987 the Appellant filed a Notice of Appeal against the above decision and paid the appeal fee. A Statement of Grounds was received by telecopy on 7 September 1987 and confirmed in writing on 11 September 1987. The Appellant stated that the Opposition Division obviously has failed to consider

(9) Chemiker-Zeitung 99 (1975), pages 19 to 22,

a document submitted in the opposition proceedings. Also, further test results were submitted. In a communication,

the Board expressed its opinion that this submission was not sufficiently relevant to be admitted at this stage of the proceedings.

Oral proceedings were held on 22 November 1988.

At the oral proceedings the Respondent submitted amended claims and an amended description. The amendment consisted in deleting from Claim 1 and its counterpart on page 2 of the description the phrase "obtaining from the solvent extract a crude glycidol solution containing not less than 1% by weight glycerine based on the total weight of glycidol and glycerine" and modifying the following phrase "reacting said alcohol and glycidol solution" to read "reacting said alcohol and that glycidol extract".

- IV. The Appellant did not maintain his objection under Article 100(c) EPC with respect to this amended Claim 1, however, he maintained the objection relating to insufficient disclosure, substantially because the amount of crude glycidol to be added to the non-polar solvent is not specified and there is no possibility to repeat even the only example in the specification. He further observed that according to the only example no extraction can take place if the crude glycidol contains less than about 5% glycerine. If less glycerine is present, the "extraction" is a simple dissolution without separation of two phases.

The essential ground for appeal however was that the claimed subject-matter does not involve an inventive step.

According to the Appellant the technical problem to be solved was to find a process for making glycerine ether alcohols in which glycidol containing glycerine could be used as starting material and which is more efficient and

gives better yields than the known processes. This problem however was not solved by the claimed process as demonstrated by the Appellant's test results. The Appellant considered that the claimed process consists of two separate steps, the first being the treatment of the crude glycidol with the non-polar solvent in order to remove the major part of the contaminating glycerine and, if present, water and the second being the reaction of the solution of step one with the aliphatic alcohol. The first step was stated to be immediately obvious to a person skilled in the art in view of the common general knowledge, see (4), and the second step was a mere modification of the prior art (3) being obvious in view of (2) or (9) which both show that reactions of this type, i.e. addition reactions of compounds containing "active hydrogen" to glycidol, are normally performed in inert solvents, which may be polar or non-polar, toluene being mentioned as suitable in both documents. With respect to (2), the Appellant pointed out that the claimed process as well as that of (2) includes the production of 1-monoglycerides.

The Appellant also found the detergency performance of the product of the only remaining example in no way unexpected as a skilled person would have been aware of the adverse effect of glycerine to the formation of the desired products from glycidol and alcohols since glycerine, too, is an alcohol and reacts with the glycidol.

The Appellant further submitted that even if this effect could be regarded as surprising it would be the inevitable result of an obvious process and would therefore not render the claimed matter unobvious (see Decision T 21/81, OJ EPO 1/1983, 15). He also relied upon the earlier decision T 192/82 (OJ 9/1984, 415) of this Board and submitted that a skilled person knowing that the second

reaction step requires an inert solvent and that the crude glycidol requires purification would have been in a one-way street situation inevitably leading him to toluene and the other aromatic hydrocarbons mentioned in Claim 1 as the only possible solvents.

- V. The Respondent stated that the two steps of the claimed process are functionally linked together because the same solvent is used for performing both steps and that the claimed process should be considered as a whole when assessing inventive step. He submitted that the prior art documents relating to the reaction of alcohols with glycidol disclose that this reaction should either be performed without solvent (see (3)) or in a polar solvent as it is expressly stated in (1). Also (5), which relates to the reaction of crude glycidol recommends polar solvents. Regarding (9), the Respondent stated that the Appellant has only relied on a part of this article and submitted the missing pages 23 to 25 at the oral proceedings. He observed that (9) on page 22 only contains a very general statement that glycidol can be stabilised against self-condensation by inert solvents. A long list of inert solvents is given which contains benzene and toluene as well as ketones, ethers, secondary and tertiary alcohols. It is also generally stated on page 22 that for a great number of reactions of glycidol the presence of an inert solvent has advantages. However, on page 23 not submitted by the Appellant, a reaction scheme is shown involving 13 different addition reactions of glycidol with nucleophiles and there is no disclosure in (9) stating that benzene or toluene should be used as solvents specifically in the reaction of glycidol with alcohols. The only document recommending toluene as a solvent for a specific reaction of the type described in (9) is (2) which, however, relates to the reaction of acids and not of alcohols with glycidol. Thus, the Respondent submits

that all documents specifically relating to the addition of alcohols recommend to use polar solvents which are also included in the general statement of (9) and therefore the prior art rather teaches away from using benzene or toluene in the reaction under consideration.

Regarding insufficiency of disclosure the Respondent noted that obviously the Appellant has had no difficulties to perform the invention, even if the exact repetition of the example was not possible.

VI. The Appellant requested that the decision under appeal be set aside and the patent revoked.

The Respondent requested that the decision under appeal be set aside and the patent maintained with the text submitted at the oral proceedings.

At the end of the oral proceedings the Board's decision to maintain the patent as requested by the Respondent was announced.

Reasons for the Decision

1. The appeal meets the requirements of Articles 106 to 108 EPC and Rule 64 and is therefore admissible.
2. No objection under Article 123(2) and (3) arises against the present wording of Claim 1. The replacement of the expression "predissolving" by "extracting" is an admissible limitation clearly disclosed by the method given in Example 6 as originally filed and granted.
3. In the Board's judgement the Appellant's objection against the sufficiency of the disclosure in the patent in suit is

in contradiction to the fact that, being guided by a worked example, he was able to carry out the claimed process. The failure to reproduce the yield given in the worked example or to specify in that example the amount of crude glycidol to be extracted, which, in any case, can be easily determined by the skilled person, does not justify such objection since these are not necessary to enable the skilled person to carry out the claimed process.

Also the Appellant's statement that a crude glycidol containing from 1 to 5% by weight glycerine as the only impurity is completely miscible with toluene and cannot therefore be extracted with that solvent cannot form a basis for alleging that the claimed process cannot be carried out by a person skilled in the art since, in the Board's judgement, a process which does not involve an extraction step, i.e. phase separation, is not encompassed by the claims of the patent in suit. This will be explained later in more detail.

The patent in suit therefore meets the requirement of Article 100(b) EPC.

4. Claims 1 to 9 of the patent in suit relate to novel subject-matter since none of the documents cited discloses a process for reacting glycidol with aliphatic alcohols in the presence of a solvent selected from benzene, toluene, ortho-xylene, meta-xylene, para-xylene and mesitylene. As novelty has not been disputed, this needs no more detailed explanation.
5. Concerning the question of inventive step the Board considers document (1) as the closest prior art, because it is the only document relating to the reaction of crude glycidol with C₁₀-C₂₀-n-alkyl alcohols (cf. Example 2).

The yields and the detergency performance of such process were unsatisfactory if the crude glycidol contains substantial amounts of glycerine (cf. Comparative Example C of the patent in suit). However, it was abundantly clear to a skilled person that this drawback was caused by the presence of glycerine being capable to compete with the higher alcohol in the reaction with the glycidol. Therefore, it was obvious to remove a substantial part of the glycerine impurity before reacting the glycidol with the alcohol. Such purification is normally performed by distillation, see (1), Example 4, or by thin film distillation under reduced pressure, see (9), page 19, right column, last four lines. However, this distillation requires reduced pressure since glycidol decomposes at its boiling point under atmospheric pressure. Thus this process is economically unsatisfactory. Therefore, the problem underlying the claimed process may be seen in providing a more economical and efficient process for the preparation of alkyl glyceryl ether alcohols by reacting crude glycerine containing glycidol with alcohols.

According to the patent in suit the solution of the above problem essentially consists in providing a two-step process wherein crude glycidol containing from 1 to 25% by weight of glycerine impurity is at first extracted with certain non-polar aromatic hydrocarbon solvents and the extract so obtained (i.e. the aromatic solvent phase containing the glycidol after separation from the second (hydrophilic) phase containing a substantial amount of the glycerine) is then reacted with the alcohol in a specified molar ratio of alcohol and glycidol under specified reaction conditions.

Therefore contrary to the Respondent's submission in the Board's judgement the mere dilution of a crude glycidol with the aromatic solvent and the subsequent reaction of

the solution so obtained with the alcohol is no longer comprised by the claims of the patent in suit. Phase separation may however occur also in cases where the crude glycidol contains from 1 to 5% by weight of glycerine impurity since according to the description, page 4, lines 29 to 38 the crude glycidol may contain further impurities, especially water and in the presence of water it is well possible that two phases are formed.

In the Board's judgement the above problem is credibly solved since it is immediately apparent that the claimed process is efficient and straightforward.

- 6.1 The question now to be answered is whether or not a skilled person would have chosen the process of the patent in suit in order to solve the above problem.

In the Board's view a skilled person might have considered to replace the purification by distillation by another well-known purification method, e.g. liquid-liquid extraction by a solvent which dissolves glycidol but not glycerine.

For this purpose, however, a number of polar and non-polar solvents are available (see (4) and (9)). The aromatic hydrocarbons specified in the patent in suit, especially toluene, belong to that group. However, there is no guidance derivable from the prior art to use these solvents for that purpose.

- 6.2 It is further known from (1) that the reaction under consideration should be performed in a solvent. However, according to (1) this solvent should be polar. None of the other documents relating to the reaction of glycidol with alcohols specifically suggests a non-polar solvent. The information in (3) and (9) relates to addition reactions

of nucleophiles to glycidol in general. In this context it is known from (3), column 1, lines 46 to 47 that the application of solvents or diluents is often profitable. In (9), page 22, right column, last complete paragraph, it is disclosed that the presence of an inert solvent is advantageous for a great number of reactions of glycidol. In the preceding paragraph by way of example, a list of solvents including benzene and toluene, but also ketones and ethers as well as isopropanol and tertiary butanol is given which may be used to prevent self-condensation of glycidol. This self-condensation involves addition of the hydroxy group of a first molecule of glycidol to the epoxy group of a second one. If a skilled person therefore would consider this information as relevant with respect to the problem he seeks to solve he would rather be told not to use a solvent for the addition of a hydroxy compound to the epoxy group of glycidol since solvents would inhibit that reaction. Thus the disclosure in (9) does neither provide any reason why a skilled person should disregard the disclosure of (3) or (1) and either avoid to use a solvent or use polar solvents, e.g. ketones or ethers nor any incentive which would encourage a person skilled in the art to use aromatic hydrocarbons as solvents for the reaction under consideration if he wishes at least to maintain the yields reported in (1).

- 6.3 According to the Appellant, such information is provided by (2) relating to a similar reaction of the same type, i.e. the addition of carboxylic acids to glycidol. However, in the Board's judgement, a person skilled in the art would not have considered this document when looking for the envisaged improvement of the reaction of alcohols with glycidol containing glycerine impurity, since it does not relate to the problem of employing impure glycidol at all. Even if, however, the skilled person would have considered that document, it would not have been regarded

as relevant because, in the Board's view, a solvent found particularly suitable for the reaction of glycidol with acids having a different reactivity need not necessarily be suitable also for the reaction of glycidol with alcohols. The information of (2) on the one hand and (1) and (5) on the other hand, i.e. that toluene is preferred for the reaction of acids and polar solvents are suitable for the reaction of alcohols with glycidol does therefore not induce a person skilled in the art having in mind the above technical problem and the knowledge represented by (9) to use exactly toluene also for that latter reaction.

- 6.4 The claimed process, as the Respondent rightly submits, must be regarded as a whole. This means that the decisive question is not whether the two steps of the claimed process individually might be obvious having regard to the prior art but whether the two steps in combination were obvious in the light of the underlying problem. On the basis of the prior art cited against the patent in suit a person skilled in the art faced with the problem of providing a more economical and efficient process for obtaining glycerine ether alcohols could not predict that this solution lies in changing the reaction medium from a polar to a specific non-polar solvent which is at first used to produce an extract containing purified glycidol by liquid-liquid extraction, because he could not expect that this sequence of process steps would provide a good yield of glycerine ether alcohols with good detergency performance.

According to the Appellant's submission the claimed process should nevertheless be regarded as obvious since the prior art taken as a whole does not exclude it from consideration and a skilled person therefore could have found it. However when assessing inventive step the

essential question is not whether a skilled person could have performed the invention under consideration but rather whether he would have done so in the expectation to solve the underlying problem. It is very often possible to show after an invention has been made that a skilled person could have found it on the basis of the prior art but such considerations are the result of an ex-post-facto analysis and must be disregarded.

Therefore in the Board's judgement the process of Claim 1 of the patent in suit involves an inventive step within the meaning of Art. 56 EPC. Claims 2 to 9 which relate to preferred embodiments of the process of Claim 1, derive their patentability from that claim.

- 6.5 In the Board's judgement the decision T 21/81 relied upon by the Appellant is not relevant to the present case because it relates to an apparatus which a skilled person would have made having regard to the cited prior art even if he was not aware of all the advantages which were inherent to it. As has already been explained in detail the claimed process in the Board's judgement would not have been found by a skilled person and hence this decision is not applicable.

Also, the decision T 192/82 is not relevant because it was not plausibly demonstrated by the Appellant that a one-way street situation has existed in the specific circumstances of this case. In the Board's judgement a great number of different modifications to the known processes were possible both in respect of the purification and the reaction step.

- 6.6 It further follows from the considerations set out above that in the Board's judgement it was immaterial for the assessment of the inventive step whether or not a product with a detergency performance better than that of the

product of (1) has been produced by the claimed process. Therefore, there was no need to admit the test results belatedly submitted by the Appellant with respect to this question to the proceedings.

7. For the reasons set out in the preceding paragraphs, the grounds for opposition raised by the Appellant do not prejudice the maintenance of the patent in suit in the text submitted at the oral proceedings.

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 on the basis of the text submitted in the oral proceedings.

The Registrar

The Chairman

F.Klein

K.Jahn