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D E C I S I O N
of 1 August 2000

Case Number: T 0444/97 - 3.3.1

Application Number: 88101641.4

Publication Number: 0278407

IPC: C07C 31/125

Language of the proceedings: EN

Title of invention:
Alcohol mixture for plasticizer

Patentee:
MITSUBISHI KASEI CORPORATION

Opponent:
Exxon Chemical Patents Inc.
BASF Aktiengesellschaft, Ludwigshafen

Headword:
C_9-Alcohols/MITSUBISHI

Relevant legal provisions:
EPC Art. 83, 100(b)

Keyword:
"Sufficiency of disclosure (no) - no sufficient information to determine the group component percentages of the mixture claimed"

Decisions cited:
-

Catchword:
-

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Case Number: T 0444/97 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 1 August 2000

Appellant: (Opponent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 21 February 1997 rejecting the opposition filed against European patent No. 0 278 407 pursuant to Article 102(2) EPC.**

Composition of the Board:

Chairman: A. J. Nuss
Members: P. P. Bracke
S. C. Perryman

Summary of Facts and Submissions

I. The appeal lies from the Opposition Division's decision, dispatched on 21 February 1997, rejecting the opposition against European patent No. 0 278 407. The opposition was based on the grounds according to Article 100(a) and (b) EPC.

The decision was based on the granted set of 7 claims with the only independent claim reading:

"1. A C_9 alcohol mixture for plasticizer obtained by the hydroformylation and hydrogenation of a C_8 olefin mixture as the starting material obtained by the dimerization of a butene fraction, said alcohol mixture comprising from 10 to 50% by weight of a first group component having a retention time not longer than the retention time of isobutyl n-caprylate, from 10 to 40% by weight of a second group component having a retention time longer than that of isobutyl n-caprylate and not longer than that of methyl n-caprate and from 30 to 70% by weight of a third group component having a retention time longer than that of methyl n-caprate, as divided into such three components by gas chromatography using a capillary column packed with a polyethylene glycol having a number average molecular weight of 15,000 to 20,000 as an isomer separating agent."

II. In particular, the Opposition Division was of the opinion that the patent in suit provided sufficient information to enable a person skilled in the art to reproduce the invention, since the patent in suit described specific gas chromatographic conditions for using a capillary column packed with "polyethylene glycol 20M" and its use for analysing C_9 alcohol mixtures.

III. At the oral proceedings on 1 August 2000 the Respondent (Proprietor of the patent) provided as the first auxiliary request the following:

"It is requested that the proceedings are continued in writing in order to give the patent proprietor an opportunity to

(1) prove that the manufacturer Gasukuro Kogyo Inc. still exists, albeit under the changed name GL Sciences by certified translation of the trade register,

(2) prove that GL Sciences is still producing the polyethylene glycol 20M column today as at the time of priority by a more detailed declaration of GL Sciences and supporting written evidence,

(3) prove that all tests within the definition of the analysis conditions given in claim 1 of the second auxiliary request produce identical results within the confines of statistical significance,

(4) examine the evidence provided by the opponent to the extent that the board considers it relevant for the decision,

(5) in connection with item (4) we ask the board to request the opponent to provide all required information for a repetition and interpretation of their evidence,

(6) further, if the question of column length should be considered significant we ask for the opportunity to prove that a 50 m column and a 60 m column produce statistically identical results,

(7) as regard the new objection that the present catalogs of GL Sciences carries two types, namely "TC Wax" and "BC Wax" we request the opportunity to inquire into this fact."

As a second auxiliary request the Respondent provided a set of 7 claims with the only independent claim reading:

"1. A C_9 alcohol mixture for plasticizer obtained by the hydroformylation and hydrogenation of a C_8 olefin mixture as the starting material obtained by the dimerization of a butene fraction, said alcohol mixture comprising from 10 to 50% by weight of a first group component having a retention time not longer than the retention time of isobutyl n-caprylate, from 10 to 40% by weight of a second group component having a retention time longer than that of isobutyl n-caprylate and not longer than that of methyl n-caprate and from 30 to 70% by weight of a third group component having a retention time longer than that of methyl n-caprate, as divided into such three components by gas chromatography using a capillary column packed with a polyethylene glycol having a number average molecular weight of 15,000 to 20,000 as an isomer separating agent under the following conditions

(1) Column: Packing "polyethylene glycol 20M" (manufactured by Gasukuro Kogyo Inc.), FFS (flexible fused silika) capillary column having a wall thickness of 0.15 μm, a length of 50 m and a diameter of 0.25 mm.

(2) Detector: FID (hydrogen flame ionization detector).

(3) Carrier gas: Helium gas, capillary: 0.66 ml/min, purge: 43.56 ml/min.

(4) Combustion gas: Hydrogen: 0,59 bar (0.6 kg/cm²G)
Air: 0.78 bar (0.8 kg/cm²G)

(5) Temperature conditions:
Initial temperature of the column: 80°C,
temperature rise after 8 minutes: 4.5°C/min, final temperature: 180°C. The temperature at the inlet and in the detector: 230°C.

(6) Time for analysis: 40 minutes.

(7) Quantitative method: Internal standard method using n-undecane as the internal standard substance (IS).

C_9 alcohol mixture (sample)/IS = 10/1 (weight ratio), amount of sample = 0.2 μl, relative mol sensitivity of alcohol = 1.0."

As a third auxiliary request the Respondent filed a set of 5 claims with the only independent claim reading:

"1. A C_9 alcohol mixture for plasticizer obtained by the hydroformylation and hydrogenation of a C_8 olefin mixture as the starting material obtained by the dimerization of a butene fraction, said alcohol mixture comprising from 15 to 35% by weight of a first group component having a retention time not longer than the retention time of isobutyl n-caprylate, from 10 to 40% by weight of a second group component having a retention time longer than that of isobutyl n-caprylate and not longer than that of methyl n-caprate and from 30 to 65% by weight of a third group component having a retention time longer than that of methyl n-caprate, as

divided into such three components by gas chromatography using a capillary column packed with a polyethylene glycol having a number average molecular weight of 15,000 to 20,000 as an isomer separating agent under the following conditions

(1) Column: Packing "polyethylene glycol 20M" (manufactured by Gasukuro Kogyo Inc.), FFS (flexible fused silika) capillary column having a wall thickness of 0.15 μm, a length of 50 m and a diameter of 0.25 mm.

(2) Detector: FID (hydrogen flame ionization detector).

(3) Carrier gas: Helium gas, capillary: 0.66 ml/min, purge: 43.56 ml/min.

(4) Combustion gas: Hydrogen: 0,59 bar (0.6 kg/cm²G) Air: 0.78 bar (0.8 kg/cm²G)

(5) Temperature conditions: Initial temperature of the column: 80°C, temperature rise after 8 minutes: 4.5°C/min, final temperature: 180°C. The temperature at the inlet and in the detector: 230°C.

(6) Time for analysis: 40 minutes.

(7) Quantitative method: Internal standard method using n-undecane as the internal standard substance (IS).

C_9 alcohol mixture (sample)/IS = 10/1 (weight ratio), amount of sample = 0.2 μl, relative mol sensitivity of alcohol = 1.0."

As a fourth auxiliary request the Respondent filed a set of seven claims with the only independent claim reading:

"1. A C_9 alcohol mixture for plasticizer obtained by the hydroformylation and hydrogenation of a C_8 olefin mixture as the starting material obtained by the dimerization of a butene fraction, said alcohol mixture comprising from 10 to 50% by weight of a first group component having a retention time not longer than the retention time of isobutyl n-caprylate, from 10 to 40% by weight of a second group component having a retention time longer than that of isobutyl n-caprylate and not longer than that of methyl n-caprate and from 30 to 70% by weight of a third group component having a retention time longer than that of methyl n-caprate, as divided into such three components by gas chromatography using a capillary column packed with a polyethylene glycol having a number average molecular weight of 15,000 to 20,000 as an isomer separating agent under the following conditions

(1) Column: Packing "polyethylene glycol 20M" (manufactured by Gasukuro Kogyo Inc.), FFS (flexible fused silika) capillary column having a wall thickness of 0.15 μm, a length of 50 m and a diameter of 0.25 mm.

(2) Detector: FID (hydrogen flame ionization detector).

(3) Carrier gas: Helium gas, capillary: 0.66 ml/min, purge: 43.56 ml/min.

(4) Combustion gas: Hydrogen: 0,59 bar (0.6 kg/cm²G) Air: 0.78 bar (0.8 kg/cm²G)

(5) Temperature conditions:
Initial temperature of the column: 80°C, temperature rise after 8 minutes: 4.5°C/min, final temperature: 180°C. The temperature at the inlet and in the detector: 230°C.

(6) Time for analysis: 40 minutes.

(7) Quantitative method: Internal standard method using n-undecane as the internal standard substance (IS).

C_9 alcohol mixture (sample)/IS = 10/1 (weight ratio), amount of sample = 0.2 μl, relative mol sensitivity of alcohol = 1.0, whereby the alcohol mixture is superior to 2-ethylhexanol in cold resistance and electrical insulating properties of a vinyl chloride polymer plasticised with a phthalic acid ester of said alcohol mixture or 2-ethylhexanol, respectively."

IV. The Appellant (Opponent) essentially argued that in gas-chromatography the nature of the packing polyethylene glycol (PEG) material (*inter alia* the polarity) of the column strongly influences the retention times of alcohol components relative to the retention times of esters. Since the patent in suit claims a C_9 alcohol mixture comprising three groups of components, each defined by the relative retention time of the said components to the retention times of one or two reference-esters in a gas chromatogram obtained with a capillary column packed with a polyethylene glycol having a number average molecular weight of 15,000 to 20,000, without giving any further information about the nature of the packing PEG, the Appellant was of the opinion that the patent in suit did not provide enough information to enable a skilled person to verify in an unambiguous way whether a C_9 alcohol mixture was embraced within the claimed scope.

As support of his argument that the nature of the packing PEG material strongly influences the retention times of C_9 alcohol components relative to the retention times of isobutyl n-caprylate and methyl n-caprate, the Appellant provided as annex to the letter of 23 May 2000 an experimental report (Versuchsbericht III) D25 with the gas-chromatographic analysis data of one and the same C_9 alcohol mixture by using columns packed with different PEG's.

V. The Respondent submitted in particular that in gas-chromatography the nature of the packing PEG material of the capillary column does not strongly influence the retention times of alcohol components relative to the retention times of esters. As support thereof he provided as annex to the letter of 30 June 2000 an experimental report as Annex G with the gas chromatographic analysis data of two commercially available C_9 alcohol mixtures using various PEG capillary columns according to the patent in suit. Moreover, he argued that a specific gas chromatography-column as well as the specific conditions for analysing a C_9 alcohol mixture were described in the patent in suit. Therefore, the patent in suit disclosed at least one way for unambiguously verifying whether a C_9 alcohol mixture was embraced within the claimed scope.

VI. The Appellant requested that the decision under appeal be set aside and that the European patent No. 0 278 407 be revoked.

The Respondent requested as main request that the appeal be dismissed, as first auxiliary request that the proceedings be continued in writing to afford the patent proprietor an opportunity to deal with the matters listed in the 1st Auxiliary Request submitted in writing on 1 August 2000, or that the decision under

appeal be set aside and the patent be maintained on the basis of one of the set of claims submitted as 2nd, 3rd or 4th Auxiliary Requests at the oral proceedings on 1 August 2000.

Reasons for the Decision

1. The appeal is admissible.

2. *Main request*

2.1 Sufficiency of disclosure

In deciding whether the patent in suit discloses the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art, in the present case, it is essential to establish whether the patent in suit provides sufficient information in order to enable a person skilled in the art to determine in an unambiguous way the group component percentages as defined in Claim 1 for otherwise the claim would not represent a true definition of the alcohol mixture for which protection is sought.

2.1.1 The patent in suit teaches on page 3, lines 46 to 51, in general terms, that whether the alcohol mixture is one according to the claimed invention is determined by analysing it by GC using a capillary column packed with PEG having a number average molecular weight of from 15,000 to 20,000 and dividing the detected isomer components into three group components based on the retention times of two internal standard substances, namely isobutyl n-caprylate and methyl n-caprate, as the first respectively the second internal standard

substance. More specifically, the patent in suit mentions on page 3, line 52 to 56, that a suitable capillary column is packed with "polyethylene glycol 20M" (manufactured by Gasukuro Kogyo Inc.).

Therefore, it remains to be decided whether the indication "polyethylene glycol 20M" provided enough information in order to enable a skilled person to determine in an unambiguous way the group component percentages as defined in Claim 1. More particular, as it was undisputed that "polyethylene glycol 20M" stands for PEG having a number average molecular weight of 20,000, the question arises whether the C_9 alcohol components are divided into the **same** three group components based on the retention times of isobutyl n-caprylate and methyl n-caprate by using any PEG-packing material having a number average molecular weight of 20,000.

2.1.2 As support of his submission that the same three group components would be obtained with any PEG-packing material having a number average molecular weight of 20,000, the Respondent referred to the experimental report in Annex G (see point V above).

This report Annex G concerns the gas chromatographic analyses of two commercially available C_9 alcohol mixtures using seven different PEG capillary columns according to the patent in suit, of which two contain as stationary phase a PEG having a number average molecular weight of 20,000, namely ULBON HR20M and CBP-20. When analysing a C_9 alcohol mixture, indicated as sample A, by gas-chromatography and dividing the components in the gas-chromatogram into three group components according to Claim 1, the C_9 alcohol mixture was found to be composed of 4,10% by weight of the first group components, 17,77% by weight of the second

group components and 77,68% by weight of the third group components, if ULBON HR20M was used as stationary phase, whereas the same C_9 alcohol mixture was found to be composed of 3,53% by weight of the first group components, 18,14% by weight of the second group components and 78,33% by weight of the third group components, if CBP-20 was used as the stationary phase.

As, according to table 1 of Annex G the analysed sample and the gas chromatographic conditions are identical, the discrepancy in weight percentage measured, for example, 4,10 and 3,53% by weight, for the first group components can only be considered to result from the fact that the retention times of the used internal standards and of the C_9 alcohol mixture components are influenced by the nature of the PEG having a number average molecular weight of 20,000 used as stationary phase in a different way.

Moreover, since the Board does not have in particular any evidence that such difference is due to the inaccuracy of the determination and measurement methods, the Board cannot accept to consider both data lying within the confines of statistical significance.

2.1.3 The findings of the above experimental data are confirmed by the data provided by the Appellant in D25, from which it follows that a C_9 alcohol mixture was found to be composed of 8.02 % by weight of the first group components, 35.79 % by weight of the second group components and 56.07 % by weight of the third group components, when using a column supplied by GL Sciences Inc. as TCWAX, whereas the same C_9 alcohol mixture was found to be composed of 23.29 % by weight of the first group components, 32.71 % by weight of the second group

components and 43.86 % by weight of the third group components, when using a column supplied by Shinwa Chemical as ULBON HR 20M, both capillary columns containing as stationary phase a PEG having a number average molecular weight of 20,000.

2.1.4 From the foregoing it follows that the retention times of the alcohol components in relation to those of isobutyl n-caprylate and methyl n-caprylate are not only influenced by the number average molecular weight of the PEG but also by the nature of that PEG, such as, for example, its polarity. As a consequence, there is no basis for accepting that the C_9 alcohol components are divided into the same three group components based on the retention times of isobutyl n-caprylate and methyl n-caprate by using any PEG-packing material having a number average molecular weight of 20,000.

Therefore, the Board comes to the conclusion that the indication "polyethylene glycol 20M" does not provide sufficient information in order to enable a skilled person to determine in an unambiguous way the group component percentages as defined in Claim 1. Since a skilled person thus cannot get sufficiently clear and complete information from the patent in suit to determine whether a C_9 alcohol composition is embraced within Claim 1, the patent in suit according to the main request does not fulfil the requirement of sufficient disclosure. The Appellant's objection under Article 100 (b) EPC is thus well founded.

3. *First auxiliary request*

3.1 Points (1), (2) and (7)

As set out above, the essential reason for coming to the conclusion that the patent according to the main request does not fulfill the requirement of sufficiency

of disclosure is that the indication "polyethylene glycol 20M" does not sufficiently specify the stationary phase of the capillary column. Therefore, it is not relevant whether the company mentioned in the patent in suit, Gasukuro Kogyo Inc., still exists under the changed name GL Sciences Inc. or whether this company still produces the polyethylene glycol 20M column today.

3.2 Point (3)

Since it was shown with the experimental report provided by the Respondent as Annex G that under the gas-chromatographic conditions mentioned in the patent in suit by using two different capillary columns containing PEG having a number average molecular weight of 20,000 different results are obtained, any further showing that identical results could be obtained could only be seen as a proof that **under very specific circumstances** with different columns containing a stationary phase designated as "polyethylene glycol 20M" the same results are obtained. However, such showing could never be considered as evidence that all tests within the definition of the analysis conditions given in the patent in suit, page 3, line 54 to page 4, line 12 produce identical results within the confines of statistical significance.

3.3 Points (4) and (5)

Considering the fact that it was shown by the experimental report provided by the Respondent as Annex G that the retention times of the alcohols and those of isobutyl n-caprylate and methyl caprate are influenced in a different way by the nature of the PEG having a number molecular weight of 20,000, the evidence

provided by the Appellant is not essential for deciding the sufficiency issues. Therefore, the requests under points (4) and (5) are not relevant for the purpose of coming to a decision.

3.4 Point (6)

The length of the column not having any influence on the deciding about sufficiency of disclosure, this point too is not relevant for the purpose of coming to a decision.

3.5 The first auxiliary request must thus be refused as a whole as it relates only to matters unnecessary for the purpose of the decision to be taken.

4. *Second auxiliary request*

4.1 Article 123(2) and (3) EPC

The Board is satisfied that claims 1 to 7 are not amended in such a way that they contain subject-matter which extends beyond the content of the application as filed or that they are amended as to extend the protection conferred. In particular, Claim 1 is a combination of the features of Claims 1 and 2 and the gas-chromatographic conditions mentioned on page 7, line 20 to page 8, line 10 of the application as filed and the additional features of Claims 2 to 7 are supported by Claims 3 to 8 respectively as filed. This was not contested by the Appellant.

4.2 Sufficiency of disclosure

It is case law of the boards of appeal that sufficiency of disclosure within the meaning of Article 83 or Article 100(b) EPC must be assessed on the basis of the application, respectively the patent, as a whole,

including the description and the claims. As the incorporation of features disclosed in the description into the claims does not change the content of the application, respectively the patent, as a whole, lack of sufficiency of disclosure will not be overcome by incorporating features from the description into the claims, because the information given, whether in the description only or also in the claims, remains insufficient for carrying out the invention.

Consequently, the second auxiliary request lacks sufficiency of disclosure for the same reasons as the ones mentioned for the main request.

5. *Third auxiliary request*

5.1 Article 123(2) and (3) EPC

The Board is satisfied that claims 1 to 5 are not amended in such a way that they contain subject-matter which extends beyond the content of the application as filed or that they are amended as to extend the protection conferred. In particular, Claim 1 is a combination of the features of Claims 1, 2 and 4 and the gas-chromatographic conditions mentioned on page 7, line 20 to page 8, line 10 of the application as filed and the additional features of Claims 2 to 5 are supported by Claims 5 to 8 respectively as filed. This was not contested by the Appellant.

5.2 Sufficiency of disclosure

The third auxiliary request lacks sufficiency of disclosure for the same reasons as the ones mentioned for the second auxiliary request.

6. *Fourth auxiliary request*

6.1 Article 123(2) and (3) EPC

The Board is satisfied that claims 1 to 7 are not amended in such a way that they contain subject-matter which extends beyond the content of the application as filed or that they are amended as to extend the protection conferred. In particular, Claim 1 is a combination of the features of Claims 1 and 2, the gas-chromatographic conditions mentioned on page 7, line 20 to page 8, line 10 and the technical effect disclosed in the first sentence of page 19 of the application as filed and the additional features of Claims 2 to 7 are supported by Claims 3 to 8 respectively as filed. This was not contested by the Appellant.

6.2 Sufficiency of disclosure

Although the further specification in Claim 1 of the effect to be achieved can have an influence on the claimed scope, it does not provide any additional information on how to carry out the invention. Therefore, in the present case, the fourth auxiliary request lacks sufficiency of disclosure for the same reasons as the ones mentioned for the second auxiliary request.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar: The Chairman:

L. Martinuzzi A. Nuss

Beglaubigt/Certified Geschäftsstelle Registry/Greffe
Certifiée conforme: München/Munich 29. SEP. 2000

BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS ★ BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE ★ CHAMBRES DE RECOURS DE L'OFFICE EUROPÉEN DES BREVETS