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D E C I S I O N
of 6 November 1996

Case Number: T 0479/95 - 3.4.2

Application Number: 86100941.3

Publication Number: 0189862

IPC: G01N 30/88, G01N 30/46

Language of the proceedings: EN

Title of invention:
On-line coupled liquid and gas chromatography system with an interface capillary tube interposed between a pair of capillary chromatographic columns

Applicant:
THE DOW CHEMICAL COMPANY

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 123(2), 54, 56 and 113(1)

Keyword:
"Main request: novelty: no"
"First auxiliary request: subject-matter extended: yes"
"Second auxiliary request: inventive step: no"

Decisions cited:
-

Catchword:
-

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Case Number: T 0479/95 - 3.4.2

D E C I S I O N
of the Technical Board of Appeal 3.4.2
of 6 November 1996

Appellant:
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 19 January 1995 refusing European patent application No. 86 100 941.3 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: E. Turrini
Members: M. Chomentowski
B. J. Schachenmann

Summary of Facts and Submissions

I. European patent application No. 86 100 941.3 (publication No. 0 189 862) was refused on the grounds that, in particular, claim 1 submitted by the appellant lacked novelty having regard to D1: Journal of Chromatography, vol. 295, 1984, pages 55 to 61. Said claim 1, which is now the main request, reads as follows:

"1. A method for conducting an uninterrupted, in-line chromatographic analysis of a sample aliquot containing organic species of interest and employing an analytical system comprising, in combination, a liquid chromatography column and a gas chromatography column, a heater for said gas chromatography column, and detection and display apparatus for providing visual indicia of species of interest present in a sample aliquot to be analyzed, said method comprising the steps of employing as the liquid column, a packed or wall-coated capillary column and physically connecting the liquid and the gas columns in an in-line communication configuration;
connecting in inter-fluid flow communication with and interposing between said liquid and said gas columns a vaporizing chamber for receiving a liquid effluent from the liquid column;
directing the eluent containing the species of interest to the vaporizing chamber which is initially heated to a predetermined temperature for vaporization of the liquid eluent thereby converting the liquid eluent into a vapor and into free species of interest present in the eluent;

raising the temperature of the vaporizing chamber in order to vaporize the species of interest after the major portion of the eluent has been vaporized and discharged and delivering the species to the gas column to effect separation thereof for detection and recordation."

II. The Examining Division took the following view:

The applicant had stressed the feature that, according to the invention, the retention gap would be heated to a temperature above the boiling point of the solvent under the pressure conditions within the GCC (gas chromatography column), whereas according to D1 the retention gap temperature was below the boiling point of the solvent under the pressure conditions within the GCC; in particular, a temperature of 115EC was mentioned at page 15, line 8 of the application as filed. However, this feature put forward by the applicant could not support the heating of the retention gap to a temperature above the boiling point of acetonitrile-water since there was no mention in the application as filed of any pressure condition within the GCC, in particular of any "typical operating pressure within the GCC"; thus, the applicant's arguments could not be considered as persuasive since the feature of heating the solvent to a temperature above its boiling point was neither defined in the claim, nor was it supported by the original disclosure, so that the method of claim 1 was not distinguished from the method known from D1.

Moreover, D5: Journal of Chromatography, vol. 299, 1984, pages 415 to 419 had also been introduced in the procedure by the Examining Division and mentioned in particular in the decision under appeal in relation with said feature for denying an inventive step even in case support for this feature could be found in the application as filed.

III. The appellant (applicant) lodged an appeal against this decision and requested that the decision under appeal be set aside and a patent be granted on the basis of a main request having a text identical with the text having formed the basis for the decision under appeal or according to a first or a second auxiliary request.

First Auxiliary request

Claim 1 of the first auxiliary request comprises in particular the additional feature that, during the step of converting the liquid eluent into a vapor and into free species of interest present in the eluent, the temperature in the vaporizing chamber is above the boiling temperature of the eluent under the respective pressure conditions, and reads as follows:

"1. A method for conducting an uninterrupted, in-line chromatographic analysis of a sample aliquot containing organic species of interest and employing an analytical system comprising, in combination, a liquid chromatography column and a gas chromatography column, a heater for said gas chromatography column, and detection and display apparatus for providing visual indicia of species of interest present in a sample aliquot to be analyzed, said method comprising the steps of employing as the liquid column, a packed or wall-coated capillary column and physically connecting the liquid and the gas columns in an in-line communication configuration;

connecting in inter-fluid flow communication with and interposing between said liquid and said gas columns a vaporizing chamber for receiving a liquid effluent from the liquid column;
directing the eluent containing the species of interest to the vaporizing chamber which is initially set at a predetermined temperature for vaporization of the liquid eluent thereby converting the liquid eluent into a vapor and into free species of interest present in the eluent, wherein the temperature in said vaporizing chamber is above the boiling temperature of the eluent under the respective pressure conditions. (read";")
raising the temperature of the vaporizing chamber in order to vaporize the species of interest which thereupon migrate through the gas chromatography column to effect separation thereof for detection and recordation."

Second Auxiliary request

Claim 1 of the second auxiliary request comprises in particular, as compared to the main request, the additional feature that, during the step of converting the liquid eluent into a vapor and into free species of interest present in the eluent, the time for vaporization of the liquid eluent in the vaporizing chamber is less than 10 min, and reads as follows:

"1. A method for conducting an uninterrupted, in-line chromatographic analysis of a sample aliquot containing organic species of interest and employing an analytical system comprising, in combination, a liquid chromatography column and a gas chromatography column, a heater for said gas chromatography column, and detection and display apparatus for providing visual indicia of species of interest present in a sample aliquot to be analyzed, said method comprising the steps of employing as the liquid column, a packed or

wall-coated capillary column and physically connecting the liquid and the gas columns in an in-line communication configuration;
connecting in inter-fluid flow communication with and interposing between said liquid and said gas columns a vaporizing chamber for receiving a liquid effluent from the liquid column;
directing the eluent containing the species of interest to the vaporizing chamber which is initially set at a predetermined temperature for vaporization of the liquid eluent thereby converting the liquid eluent into a vapor and into free species of interest present in the eluent, wherein the time for vaporization of the liquid eluent in the vaporizing chamber is less than 10 min;
raising the temperature of the vaporizing chamber in order to vaporize the species of interest which thereupon migrate through the gas chromatography column to effect separation thereof for detection and recordation."

IV. In a communication dated 7 August 1996 accompanying the invitation to oral proceedings which the appellant had requested auxiliarily, the Board of appeal expressed the opinion that the subject-matter of claim 1 of the main request appeared to lack novelty having regard to D1, that the amendments having led to claim 1 of the first auxiliary request appeared to result in the application containing subject-matter extending beyond the content of the application as filed and that the subject-matter of claim 1 of the second auxiliary request appeared to lack an inventive step having regard to D1 and D5.

V. Appellant's letter dated 16 October 1996 informed the Board that the applicant and appellant had decided not to attend the oral proceedings scheduled to take place on 20 November 1996 and that it was, therefore, requested that a decision on the record be taken on the basis of the main and auxiliary requests as on file.

VI. The oral proceedings were cancelled accordingly.

VII. The appellant argued substantially as follows in the statement of grounds of appeal:

Claim 1 of the main request clearly comprises the feature that the vaporizing chamber is at a temperature above the boiling temperature of the eluent under the respective pressure conditions even before introducing the eluent; the term "vaporizing temperature" has to be interpreted, in the light of the technical disclosure of the specification, as being synonymous with the term "boiling temperature"; an indication of a different interpretation cannot be found in the specification. Therefore, the method of claim 1 for conducting a chromatographic analysis of a sample aliquot containing organic species of interest is distinguished from the method known from D1 and is thus new.

Claim 1 of the first auxiliary request comprises the feature that, during the step of converting the liquid eluent into a vapor and into free species of interest present in the eluent, the temperature in the vaporizing chamber is above the boiling temperature of the eluent under the respective pressure conditions. This feature has been introduced as disclaimer for delimitation over D1, so that an explicit disclosure in the original documents is not necessary and the European patent application has not been amended in such a way that it contains subject-matter which extends beyond the content of the application as filed.

Claim 1 of the second auxiliary request comprises the feature that, during the step of converting the liquid eluent into a vapor and into free species of interest present in the eluent, the time for vaporization of the liquid eluent in the vaporizing chamber is less than 10 min, so that the claimed subject-matter is clearly delimited over D1 and uses a surprisingly lower retention time than in said document, wherein it is specified that the solvent evaporates in the flooded inlet for between 10 minutes and more than one hour. Therefore, the subject-matter of the claim involves an inventive step.

Reasons for the Decision

1. The appeal is admissible.

2. *Main request*

2.1 Novelty

2.1.1 A method for conducting a chromatographic analysis of a sample aliquot containing organic species of interest is known from D1 (see page 55, "Summary"; page 56, paragraph "Experimental and Results" to page 61, paragraph "Conclusions"; Figures 1 to 4); said method is an uninterrupted, in-line chromatographic analysis of said sample aliquot containing organic species of interest; said method employs an analytical system comprising, in combination, a liquid chromatography column (LC column) and a gas chromatography column (GC), a heater for said gas chromatography column (GC), and detection and display apparatus for providing visual indicia of species of interest present in said sample aliquot to be analysed; said method comprises the steps of: employing as the liquid column, a packed

capillary column and physically connecting the liquid and the gas columns in an in-line communication configuration;
connecting in inter-fluid flow communication with and interposing between said liquid and said gas columns a vaporizing chamber (fused-silica retention gap) for receiving a liquid effluent from the liquid column;
directing the eluent containing the species of interest to the vaporizing chamber (fused-silica retention gap) which is initially heated to a predetermined temperature for vaporization of the liquid eluent thereby converting the liquid eluent into a vapor and into free species of interest present in the eluent;
raising the temperature of the vaporizing chamber in order to vaporize the species of interest after the major portion of the eluent has been vaporized and discharged and delivering the species to the gas column to effect separation thereof for detection and recordation.

2.1.2 The appellant has argued that present claim 1 clearly comprises the feature that the vaporizing chamber is at a temperature above the boiling temperature of the eluent under the respective pressure conditions even before introducing the eluent, that, in the light of the technical disclosure of the present specification, the term "vaporizing temperature" has to be interpreted as being synonymous with the term "boiling temperature", and that an indication of a different interpretation cannot be found in the present specification. However, this argument is not convincing because, in particular, the term "above the boiling temperature of the eluent" is not comprised in present claim 1 and thus cannot constitute an explicit distinguishing feature; moreover, although it is derivable from a plurality of text locations of the present application (see page 7, lines 28 to 30; page 11, lines 25 to 32) that, in the vaporizing

chamber, the eluent is converted to a gas and the species of interest present in the eluent is freed, or the eluent is at a temperature such that it is transformed into a vapor, however, the indications in D1 (see in particular page 58, second paragraph, third to seventh line and Figure 3), according to which solvent evaporation takes place at 80EC, this being mentioned as the boiling point of the solvent at ambient pressure, do not allow to distinguish this known mode of evaporation or vaporization from the mode of vaporizing of the eluent of the present application. Since the appellant has not provided any further argument, and since the arguments mentioned above are not considered as being convincing, the method of present claim 1 cannot be considered as being distinguished from the method known from D1, so that the subject-matter of present claim 1 lacks novelty in the sense of Article 54 EPC.

2.1.3 Therefore, the appellant's main request is not allowable (Article 54 and 97(1) EPC).

3. *First Auxiliary request*

3.1 Claim 1 of the present first auxiliary request comprises in particular the feature that, during the step of converting the liquid eluent into a vapor and into free species of interest present in the eluent, the temperature in the vaporizing chamber is above the boiling temperature of the eluent under the respective pressure conditions. Indeed, claim 1 of the present main request does not comprise any explicit similar feature concerning the temperature of the vaporizing chamber. The appellant has submitted that this feature has been introduced as disclaimer for delimitation over D1, so that an explicit disclosure in the original documents is not necessary. However, according to D1 (see page 58, second paragraph, fifth to seventh lines;

Figures 3 and 4; see also Figure 4), the only explicit temperature mentioned with respect to the fused-silica retention gap, i.e. the part wherein evaporation of the solvent (eluent) takes place, is 80E, which is indicated as being the boiling point of the solvent at ambient pressure. Therefore, since no specific other temperature is derivable from D1 (see also page 56, penultimate paragraph), the feature that the temperature in the vaporizing chamber is above the boiling temperature of the eluent under the respective pressure conditions cannot be considered as a disclaimer, which would concern only the specific temperature of 80EC at a particular inlet pressure (hydrogen) of 1.7 atm., but is an added positive feature, for which no basis could be detected in the application as filed. Therefore, the European patent application does not satisfy the requirement of Article 123(2) EPC that it may not be amended in such a way that it contains subject-matter which extends beyond the content of the application as filed and, therefore, the first auxiliary request is not allowable.

4. *Second Auxiliary request*

4.1 Claim 1 of the present second auxiliary request comprises in particular the feature that, during the step of converting the liquid eluent into a vapor and into free species of interest present in the eluent, the time for vaporization of the liquid eluent in the vaporizing chamber is less than 10 min. Indeed, according to the present description (see page 15, lines 1 to 10; Figures 1), for particular conditions and a particular eluent, the temperature of the oven (150) used for setting the temperature in order to vaporize the eluent is maintained at 115EC for seven minutes. In D1 (see page 56, third paragraph, second to fourth line), it is specified that the solvent

evaporates in the flooded inlet for between 10 minutes and more than one hour (depending on the carrier gas flow-rate and the column temperature). Therefore, the subject-matter of present claim 1 differs with respect of the duration of the evaporation of the eluent from the method known from D1 and thus is novel in the sense of Article 54 EPC.

4.2 However, starting from the technique known from D1, the skilled person was prompted, in particular by D5 (see page 415, second paragraph, first to fourth lines; page 417, second paragraph, first to third lines and the penultimate paragraph; see also page 419, the last sentence; Figure 1) which belongs to the related technical field of capillary gas chromatography and which mentions that the solvent mixtures listed in the Tables I and II are of interest as eluents for high-performance liquid chromatography (HPLC) coupled to capillary GC, to accelerate the solvent evaporation, which is mentioned indeed as lasting 10-30 minutes even under optimized conditions, this being done for instance by increasing the temperature of the column during the sample introduction above the boiling point of the solvent. Therefore, the subject-matter of present claim 1 results from an obvious combination of the teachings of D1 and D5 and, thus, it lacks an inventive step in the sense of Article 56 EPC, so that the second auxiliary request is not allowable.

5. *Basis of the decision*

The objections mentioned in the present decision are in substance identical with the objections set forth in the communication of the Board dated 7 August 1996 accompanying the invitation to oral proceedings which the appellant had requested auxiliarily. It is to be noted that no comment has been provided by the appellant against the objections of the Board. The

appellant has indicated in its letter dated 16 October 1996 that it had decided not to attend the oral proceedings scheduled to take place on 20 November 1996 and that it was, therefore, requested that a decision on the record be taken on the basis of the main and auxiliary requests as on file. Since the objections have been considered again and have still been found relevant, the requests are refused on the same grounds (Articles 113(1) and 97(1) EPC).

Order

for these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

P. Martorana E. Turrini

MCH

B. Sch.