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D E C I S I O N
of 10 July 1996

Case Number: T 0980/94 - 3.4.2

Application Number: 89401513.0

Publication Number: 0345164

IPC: G01N 1/10, G01N 1/28, G01N 1/22

Language of the proceedings: EN

Title of invention:
Method and apparatus for sampling a cryogenic liquid

Applicant:
CANADIAN LIQUID AIR LTD AIR LIQUIDE CANADA LTEE

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 84, 54, 56

Keyword:
"Clarity - after amendments - yes"
"Novelty - after amendments - yes"
"Inventive step - after amendments - yes"

Decisions cited:
-

Catchword:
-



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Boards of Appeal

Chambres de recours

Case Number: T 0980/94 - 3.4.2

D E C I S I O N
of the Technical Board of Appeal 3.4.2
of 10 July 1996

Appellant:

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Decision under appeal:

Decision of the Examining Division of the European
Patent Office posted 12 August 1994 refusing
European patent application No. 89 401 513.0
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. 89 401 513.0 (publication No. 0 345 164) was refused on the grounds that the submitted claim 1 was not clear, that the subject-matter of this claim, which concerned a method, and of the other submitted independent claim of the same category was not novel having regard to D1: US-A-3 673 871, and that the subject-matter of the submitted further independent claim, which concerned an apparatus, lacked an inventive step having regard to D1 and D2: US-A-3 938 391.

The Examining Division took the following view:

The claimed method of sampling a cryogenic liquid for determination of the molar composition of the cryogenic liquid comprises the steps of isolating a liquid sample of the cryogenic liquid in an environment at a sampling location, removing said environment, and removing only said collection vessel. Since any method has to be employed in an environment, the vague feature of removing said environment, in particular in combination with the further feature of removing the collection vessel, results in a claim which lacks clarity.

Moreover, insofar as the text of the claim is understandable, all the features of the claimed method are shown in D1, which is thus suitable for solving the problem mentioned in the application that is collecting a sample containing all impurities without changes. The submitted apparatus claim 9 differs from the apparatus of D1 only in a feature of the connection between chambers or vessels, which is a matter of normal design procedure because it relates to effects which are generally known in this technical field, for instance from D2, so that said claim lacks an inventive step.

- II. The appellant (applicant) lodged an appeal against this decision.
- III. During the oral proceedings of 10 July 1996 which the appellant had requested auxiliarily, it filed a new set of 10 claims with the only independent claims reading as follows:

"1. A method of sampling a cryogenic liquid for determination of the molar composition of the cryogenic liquid comprising:
taking a liquid sample of the cryogenic liquid and holding it in an environment at a sampling location which is at a temperature above the freezing temperature of the cryogenic liquid and not greater than the boiling temperature of the cryogenic liquid,
removing said environment,
allowing then the held liquid sample to completely vaporize in bulk into a collection vessel without change in molar composition of the gas, which correctly reflects the composition of the liquid, collecting the vaporized liquid in gas form in said collection vessel for analysis, and
removing only said collection vessel from said sampling location for said analysis."

"4. A sampling method for determination of the molar composition of a cryogenic liquid comprising the steps of:
connecting a liquid sampling vessel (12) to a gas collection vessel (14) with at least a first valve (40, 42) in the connection, closing said first valve (40, 42) to isolate said collection vessel (14) from said sampling vessel (12),
filling said sampling vessel (12) with said cryogenic liquid, maintaining the walls of said sampling vessel

(12) at a first temperature which is at least about the temperature of said cryogenic liquid, cutting off the flow of said cryogenic liquid into said sampling vessel (12), applying a second temperature, higher than said first temperature, around said walls of said sampling vessel (12) in order to completely vaporize in bulk said cryogenic liquid and to generate gas, opening said first valve (40, 42), filling said collection vessel (14) with said generated gas without change in molar composition of said gas, which correctly reflects the composition of the cryogenic liquid, closing said first valve (40, 42), and disconnecting said collection vessel (14) and said first valve (40, 42) from said sampling vessel (12) for transportation of said collection vessel (14)."

"9. An apparatus to carry out the process according to one of claims 1 or 4 (read "to") 8 for sampling a cryogenic liquid for determination of the molar composition of the cryogenic liquid, comprising: a sampling vessel (12) for housing a sample of the cryogenic liquid, first conduit means (18) for flow of the cryogenic liquid into said sampling vessel (12), a removable chamber (16) encompassing said sampling vessel (12), an outlet (24) in said sampling vessel (12) for flow of cryogenic liquid from said sampling vessel (12) to said chamber (16), a gas collection vessel (14), second conduit means (22) adapted to provide gas flow communication between said sampling vessel (12) and said gas collection vessel (14), wherein said gas collection vessel (14) is detachably connected to said sampling vessel (12) by said second conduit means (22)."

IV. The appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the new set of claims, and submitted the following arguments in support of its requests:

The present claims include all the important features of the present invention, in particular that the liquid sample of the cryogenic liquid, which is held at the sampling location which is at a temperature above the freezing temperature of the cryogenic liquid and not greater than the boiling temperature of the cryogenic liquid, is allowed to completely vaporize in bulk into a collection vessel without change in molar composition of the gas, which correctly reflects the composition of the liquid. Therefore, there is no confusion regarding the function of the removable environment, which relates to the conditions such as the temperature surrounding said sampling vessel, and the claims are clear. D2 represents the nearest prior art because it also concerns measurements of vaporized cryogenic liquids which correctly reflect the composition of the liquid, and comprises the means and method steps necessary therefor. The present claims are novel in that they include the removable environment and the removable gas collection vessel which are not derivable from D2. Starting from the technique of D2, which uses a movable apparatus with a plurality of chambers for the cryogenic liquid connected by valves and is thus complicated, the skilled person would not take into account in an obvious way the technique of D1, which is adapted for separation of the contaminants from the cryogenic liquid and separate inspection thereof. Therefore, the present claims involve an inventive step.

Reasons for the Decision

1. The appeal is admissible.
2. *Allowability of the amendments*

Present claim 1 is based on original claim 1; moreover, it includes the features of original dependent claim 2 of removing the collection vessel from the sampling location for the analysis and of the original description (see page 10, lines 18 to 21; page 3, lines 16 to 23) that the liquid in the sample chamber is completely vaporized in bulk into a collection vessel without change in molar composition of the gas, which correctly reflects the composition of the liquid.

Present claim 4 is based on original main claim 5 and has been amended substantially as claim 1; moreover, the term "the walls" of the sampling vessel (12) has been substituted for "a wall" by taking into account in particular the original description (see page 14, lines 1 to 8; the figure) which concerns the influence on the content of the sampling vessel (12) by conditions such as the temperature surrounding said sampling vessel. Present claim 9 is based on original independent claim 10; it is drafted as a claim dependent on some of the method claims and includes moreover the features of original dependent claim 11 that the gas collection vessel (14) is detachably connected to said sampling vessel (12) by said second conduit means (22).

Therefore, the European patent application satisfies 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.

3. *Clarity and conciseness of the claims*

3.1 According to the present application (see page 10, lines 18 to 21; page 3, lines 16 to 23; page 14, lines 1 to 8), an important feature of the present invention is that the liquid sample of the cryogenic liquid held at the sampling location which is at a temperature above the freezing temperature of the cryogenic liquid and not greater than the boiling temperature of the cryogenic liquid, is allowed to completely vaporize in bulk into a collection vessel without change in molar composition of the gas, which correctly reflects the composition of the liquid, this being done by modifying the environment of the sampling chamber and in particular by increasing the surrounding temperature. This important feature, in the form of, in particular, "removing said environment", "applying a second temperature" or "a removable chamber (16) encompassing said sampling vessel (12)", is comprised in all the main claims explicitly or by back-reference, the different wordings being adapted to the respective claims and leading to a similar, definite meaning.

3.2 It is to be noted that present claims 1 and 4 are independent claims of the same category. The appellant has argued convincingly in this respect that the content of claim 4 is less abstract than the one of claim 1 and nearer to the embodiment illustrated in the only figure, that it represents embodiments for which a specific protection is considered as necessary, but that an amendment to render it dependent from claim 1 could result in additional subject-matter because of the differences of wording used originally.

Therefore, the Board is satisfied that, since the invention to be protected is defined without ambiguity in the set of claims, the present claims are clear and concise in the sense of Article 84 EPC.

4. *Novelty*

4.1 A method of sampling a cryogenic liquid for determination of the molar composition of the cryogenic liquid is known from D2 (see column 1, lines 4 to 12; column 1, line 41 to column 2, line 20; column 3, line 27 to column 4, line 12; the figure); the known method comprises the following steps: taking a liquid sample of the cryogenic liquid and holding it in an environment (2, 5), i.e. in the sample-receiving container (2) and the surrounding concentric interspace (5), at a sampling location which is at a temperature above the freezing temperature of the cryogenic liquid and not greater than the boiling temperature of the cryogenic liquid, allowing then some part of the held liquid sample to completely vaporize in bulk into a collection vessel (1), i.e. in the compressed gas vessel (1), without change in molar composition of the gas, which correctly reflects the composition of the liquid, collecting the vaporized liquid in gas form for analysis, in particular in said compressed gas vessel (1), i.e. in said collection vessel (1).

However, contrary to the present method, it is not by removing said environment, i.e. the conditions acting on the sample-receiving container (2, 5), that the liquid sample is allowed to completely vaporize, but by allowing some of the cryogenic liquid to flow out of the sample-receiving container (2, 5) into the concentric, surrounding compressed gas vessel (1), that only that part of the liquid sample is allowed to completely vaporize in bulk. Moreover, contrary to the present

method, in the known method, the collection vessel (1) is not the only part removed from the sampling location for said analysis, but both the compressed gas vessel (1) and the sample-receiving container (2, 5), which are held together and form a structural unit, are to be transported.

Incidentally, it is to be noted that an alternative method is also described in D2 (see column 3, lines 65 to 68), wherein the gas is then withdrawn from the compressed gas vessel (1) via a valve (11) and subjected to immediate analysis. However, since the analysis is mentioned as immediate, there is no indication of a transportable vessel removable from the compressed gas vessel (1) and its outlet valve (11) and, moreover, since the necessary gas container receives the gas after the cryogenic liquid has vaporized, there is no step of vaporizing into said volume where the analysis is effected.

4.1.1 An apparatus for sampling a cryogenic liquid for determination of the molar composition of the cryogenic liquid is known from D2 (see the same text locations); however, contrary to the present apparatus, the known apparatus comprises neither a removable chamber encompassing a sampling vessel wherein the cryogenic liquid sample is held and then vaporized nor a removable gas collection vessel.

4.2 A method of sampling a cryogenic liquid is known from D1 (see column 1, lines 29 to 32; column 2, lines 1 to 3; column 2, line 38 to column 3, line 12; Figures 1 to 3); however, the known method is used to isolate and separate from a sample of said cryogenic liquid the solid contaminants (condensable and solid particulates) contained therein in order to analyze the remaining liquid gas sample and the contaminants separately; thus,

contrary to the present sampling method, it is not the liquid sample which is allowed to completely vaporize in bulk into a collection vessel without change in molar composition of the gas, so that what is measured does not correctly reflect the composition of the original cryogenic liquid. An apparatus for sampling a cryogenic liquid is known from D1; however, since as mentioned here above the known apparatus is to carry out a process which is used to isolate and separate from this sample the solid contaminants (condensable and solid particulates), it comprises apparatus means which are different from those of the present apparatus, wherein the liquid sample is allowed to completely vaporize in bulk into a collection vessel without change in molar composition of the gas, which correctly reflects the composition of the liquid. More in particular, the present apparatus cannot comprise means for separating and isolating solid contaminants such as the filter (Filter, 144) of the known apparatus.

4.3 Therefore, the subject-matter of each of the main claims, i.e. of claim 1, of claim 4, which corresponds in substance to claim 1, and of claim 9, is novel in the sense of Article 54 EPC.

5. *Inventive step*

5.1 Since contrary to the method of D1 both the present method and the method of D2 are methods wherein the liquid sample is allowed to completely vaporize in bulk into a collection vessel without change in molar composition of the gas, which correctly reflects the composition of the liquid, D2 is considered as representing the nearest prior art.

5.2 The statement, in the present description (see page 2, lines 1 to 8), that methods of the type known from D2 have a number of disadvantages to rapid repeated use for collection of a plurality of samples which is important for comparison of the analysis results, is credible for the following reason:

In the first form of this known method, the entire apparatus (1, 2, 5) is transported into a laboratory where the analysis is conducted. Indeed, in the second, alternative form of the method, the gas is then withdrawn from the compressed gas vessel (1) via the valve (11) and subjected to immediate analysis; however, it is to be noted that it is stated in D2 (see column 1, lines 41 to 46) that the invention therein is based on the problem of developing a simple device which can be used as a mobile unit for the sampling of a liquefied gas and for the total evaporation of the thus-withdrawn liquid wherein assurance is obtained that, after the phase of conversion, the thus-produced gas has the same composition as the liquid; thus, no removable, transportable gas collection vessel is provided and no indication about it is derivable from D2 either. Therefore, it is necessary to transport for analysis the whole sampling apparatus of D2, and this can be considered as a cause of the drawback of this prior art mentioned in the present application. Moreover, as convincingly argued by the appellant, the method of D2 uses a complicated appliance, with concentric vessels (1), (2) and (5) and valves therebetween.

5.3 The above-mentioned problems of the prior art are credibly solved by the present methods and apparatus, wherein the environment of the sampling vessel is removable and thus said vessel can be submitted to the influence of, in particular, changing surrounding temperatures, and the gas collection vessel is detachably removable.

5.4 Indeed, in the method of sampling a cryogenic liquid known from D1 (see column 2, line 39 to column 4, line 29; Figures 1 to 3), the environment of the cryogenic liquid is also removed for causing the liquid in the vessel (38) to vaporize and, moreover, there is also at least a removable sample bottle (44) for analysis of the vaporized gas.

However, the method of D1 is for isolating ice and other condensible gases on the one hand, and the cryogenic gas itself on the other hand, and for inspecting them separately. Thus, in particular, in one of said methods, two removable sample bottles (44) and (60) are used, the second sample bottle (60) being for transporting the other condensible gases considered as contaminants; both said bottles are then submitted to analysis of the gases. In the other described method of D1 (see column 3, lines 13 to 27), there is indeed only one removable gas bottle (44), but the method is such that the contaminants left in the liquid sample chamber (38) are analysed therein. In any case, contrary to the technique of D2, no liquid having the same composition as the original cryogenic liquid is analysed.

It is also to be noted that, contrary to the system of D2, wherein the gas collection vessel (1) surrounds and insulates the sampling vessel (5, 2) containing the original cryogenic liquid, the structure of the

apparatus of D1 comprises a sampling vessel (38) directly in contact with a surrounding environment and thus adapted to be submitted to the influence of, in particular, changing surrounding temperatures.

5.5 Therefore, as convincingly argued by the appellant, taking into account the different purposes of the techniques of D1 and D2 and the corresponding differences in structure of the systems for implementing them, it was not obvious, at the present priority date, for the skilled person starting from the complicated technique of D2 and intending to simplify it, to take into consideration isolated features of the different technique of D1, in particular the removable environment and the removable gas sample vessel. Therefore, the subject-matter of present claim 1 involves an inventive step in the sense of Article 56 EPC.

5.6 Since present claim 4 differs in substance from claim 1 only in that it is more closely directed to the embodiment described in relation with the only figure of the present application, and since present claim 9 in particular is drafted as a dependent claim and thus credibly comprises the set of technical means necessary in particular for the method of claim 1 and which is not obvious in view of the teachings of D1 and D2, their subject-matter also involve an inventive step.

6. Therefore, the claims are allowable and a patent can be granted on this basis (Articles 52(1) and 56 EPC).

Order

For these reasons it is decided that:

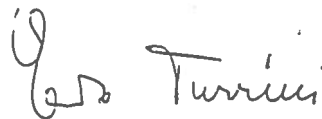
1. The decision under appeal is set aside.
2. The case is remitted to the Examining Division with the order to grant a patent on the basis of claims 1 to 10 submitted during the oral proceedings of 10 July 1996, the original description to be adapted and the figure as originally filed.

The Registrar:



E. Görgmaier

The Chairman:



E. Turrini

MCA

B. Sch.

