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Bezeichnung der Erfindung: Process for removing water from surfaces of Title of invention: articles Titre de l'invention :

Klassifikation / Classification / Classement : B 01 D 12/00

ENTSCHEIDUNG / DECISION vom/of/du 28 October 1986

Anmelder / Applicant / Demandeur :

Daikin Kogyo Co. Ltd.

Patentinhaber / Proprietor of the patent / Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence :

EPU/EPC/CBE Articles 52(1) and 56

Kennwort / Keyword / Mot clé :

"Inventive step" (yes)

Leitsatz / Headnote / Sommaire

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Case Number : T 192/84

D E C I S I O N of the Technical Board of Appeal 3.4.1 of 28 October 1986

Appellant :

Daikin Kogyo Co., Ltd. Shin-Hankyu Building 12/39 Umeda-1-chome Kita-ku Osaka-Shi, Osaka Japan

Representative :

Low, Peter John Wilson, Gunn & Ellis 41 Royal Exchange Cross Street Manchester M2 7BD Great Britain

Decision under appeal :

Decision of Examining Division 031 of the European Patent Office dated 4 May 1984 refusing European patent application No. 80 301 002.4 pursuant to Article 97(1) EPC

Composition of the Board :

Chairman	:	K.	Lederer
Member	:	J.	Roscoe
Member	:	E.	Persson

Summary of Facts and Submissions

I. European patent application No. 80 301 002.4 filed on 31 March 1980 and published under publication number 0 017 459 was refused by a decision of the Examining Division 031 of the European Patent Office dated 4 May 1984. That decision was based on Claims 1 to 3 filed on 31 August 1983.

- II. The reasons given for the refusal were that
 - (1) the subject-matter of the claims did not involve an inventive step having regard to GB-A-1 402 042 (document 1) and GB-B-1 236 180 (document 2) (Articles 52, 56 EPC);
 - (2) the claims were not clear (Article 84 EPC);
 - (3) the claims and description did not comply with the provisions of Rules 29(1) and 27(1)(c) EPC respectively.
- III. On 13 August 1984 the appellant filed a notice of appeal which was not strictly in accordance with the provisions of Rule 64 EPC, together with an application for reestablishment of rights. The appeal fee was paid at the same time. An amended notice of appeal remedying the deficiencies in the first notice was filed on 24 August 1984. A statement setting out the grounds of appeal filed by a telex dated 14 September 1984 was confirmed by a letter received on 22 September 1984. The letter was accompanied by a new set of claims and amended pages of description.

- IV. In a decision dated 9 November 1984 the Board declared that the appellant's company never lost the rights it had sought to have restored and that the notice of appeal and appeal fee were to be treated as having been received in due time.
 - V. At the oral proceedings appointed following an exchange of correspondence between the rapporteur and the appellant, the appellant presented a new set of claims and a new description to replace those on file and requested that the decision under appeal be set aside and a patent granted on the basis of the Claims 1 and 2 presented at the oral proceedings.

Claim 1, the only independent claim, reads as follows:

"A process for removing water from the surface of articles using apparatus comprising a water separating zone including a dewatering tank containing a mixture of trichlorotrifluoroethane and ethanol, and a water separator, the dewatering tank and the water separator being in communication with each other through a space provided thereabove, which space is allowed to communicate with atmosphere through a communication channel provided in part of the space, a cooler in the communication channel, the apparatus also comprising an evaporator having an upper space also communicating with the space above the dewatering tank and the water separator, said process comprising immersing an article from which water is to be removed in the dewatering tank, separating removed water from the bath in the water separating tank by virtue of the difference in specific gravity, introducing bath from which water has been separated into the evaporator, condensing vapour in the cooled channel, returning the condensed vapour to the water separating zone and adding a mixture of trichlorofluoroethane and ethanol to replace losses thereof from vapour loss, characterised in that (1) the ethanol

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concentration of the bath is initially from 4.5 to 7.8% by weight, (2) the aqueous phase from the water separator is rectified in a rectifying unit for separating ethanol from the water in said aqueous phase, (3) the ethanol recovered in step 2 is recycled from the rectifying unit to the water separating zone, (4) the water separating zone and/or the evaporator and/or reservoir if present is topped up with a bath mixture of trichlorotrifluoroethane and ethanol having an ethanol concentration of 4.5 to 7.8% by weight and (5) part of the bath from which water has been separated is introduced into the dewatering tank through the evaporator and part directly."

VI. The appellant argues essentially as follows:

The problem which the invention sets out to solve is the unpredictable change in bath composition arising from vapour loss and the selective loss of the water-miscible ethanol in the water discharged from a system as described in document 1 when using a trichlorotrifluoroethane (hereafter TCTFE)-ethanol bath. Document 1 itself affords no hint of the existence of such a problem and contains no information which could lead the skilled man to tackle it by the use of a rectifier in the manner claimed in the present application.

Were the skilled man, despite the statements in document 1 that the only loss of solvent from the system is as vapour and is not substantial, to recognise the possibility of ethanol being removed with the discharged water he would, were he to follow the only teaching in the document on how to deal with solvent loss, simply periodically add fresh solvent, the composition of which would be chosen in an attempt to offset the assumed loss whereby to restore the original bath composition.

As to the composition of the bath the preferred bath proposed in document 1 was the TCTFE-isopropanol azeotropic mixture and that document points to the desirability of using an azeotropic mixture whenever using an evaporator and indicates the need for composition adjustment if other mixtures are used. It therefore does not suggest the use of the non-azeotropic TCTFE-ethanol mixture of the composition claimed either as starting bath or as replenishment nor does it give any hint that such a mixture might have advantages.

In fact, however, the appellant has recognised that such a mixture, as Fig. 2 of the application shows, exhibits substantially improved separation efficiency compared with the solvents containing isopropanol and other of the common water-miscible solvents proposed in document 1, and with the azeotropic TCTFE-ethanol mixture.

Reasons for the Decision

- 1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is therefore admissible.
- 2. There is no objection to the present claims on formal grounds since they are clear and adequately supported by the specification as originally filed. Apart from feature (5) of its characterising part Claim 1 corresponds in substance to the original Claim 1. The features of that claim have been rearranged to take account of the prior art and to distinguish the process features proper from the constructional features of the apparatus in which the process is performed, and amendments made on the basis of information contained in the description, to eliminate some minor obscurities. The feature (5) is supported by Figs. 4

and 6 and the associated description from which it can be seen that bath from the reservoir 7 flows back to the dewatering tank directly via pump 8 or passes along line 17 to the evaporator.

- 3. The amendments made to the description serve to adapt it to the new claims, to meet the requirements of Rule 27(1)(c) EPC and to facilitate the understanding of certain passages. None of the amendments introduces matter which was not contained in the description as originally filed. Therefore no objection arises under the terms of Article 123(2) EPC.
- 4. Novelty
- 4.1 A careful examination of the documents cited in the search report reveals that none of them discloses the use of a rectifier to separate ethanol from the aqueous phase leaving a dewatering bath filled with a mixture of ethanol and trichloro-trifluoroethane.
- 4.2 Therefore the subject-matter of Claim 1 is novel.
- 5. Inventive step.
- 5.1 The process described with reference to Figs. 9 and 10 of document (1), which the Board considers to be the closest prior art, exhibits all the features set out in the preamble of Claim 1. Thus dewatering tank 28, water separator 30 and evaporator 36 all communicate with a channel, bounded by cooler 39, which opens into the atmosphere. During the water removal process articles to be dewatered are immersed in the tank 28 from which bath overflows into the separator. There the water, on account of its lower density, rises to the top and is discharged through pipe 33. The denser bath component flows into the

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evaporator where it is boiled to generate vapour which, after condensation by the cooler, collects in trough 40. This condensate is said to be cycled to the dewatering tank. Such cycling, to quote the document (page 8, lines 76-82), "may be essentially direct, flowing only through miscellaneous auxiliary components, such as driers, reservoirs and the like, or it can be indirect by first flowing into water separating sump 30 which, of course, completes the cycle by feeding dewatering sump". This implies the provision of a pipe connection, not shown in the figures between the two sumps which would allow some of the solvent separated from the liquid overflowing from the dewatering sump to return to it without having to pass through the boiling sump. Thus when construing the quoted passage in this way the described process also implicitly includes feature (5) of the characterising part of Claim 1.

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In the part of the description specifically relating to 5.2 Figs. 9 and 10 the only reference to bath composition is in Example I where the azeotrope of 97% by weight TCTFE and 3% isopropanol is used. On the other hand in the extensive discussion on choice of solvent at page 2, line 112 to page 4, line 7 it is stated that for removal of water a particularly effective group of solvents are two component solvents consisting of a water-miscible and a waterimmiscible component in which the latter constitutes between 80 and 99.5% by weight of the mixture. 1,1,2trichloro-1,2,2 trifluoroethane is one of two materials identified as preferred water-immiscible component to be used in combination with one of a group of eight solvents, which includes ethanol, as water-miscible component. Again the preferred solvent within this group is stated to be the mixture of 85-98% by weight 1,1,2-trichloro-1,2,2 trifluoroethane and the rest isopropanol. It is said to be important, where a solvent mixture is heated to boiling to

generate vapours (as in Figs. 9 and 10) to use a constant boiling azeotropic mixture since otherwise the mixture fractionates on boiling and eventually composition adjustment is required.

Neither in document (1) nor in US-A-3 559 297 (document 3), nor in FR-A-2 213 788 (document 4) the relevant contents of which correspond essentially to that of document 1 is there however any proposal to use a mixture of ethanol and TCTFE containing ethanol at a concentration within the claimed range of 4.5 to 7.8%.

In the documents cited on the search form the only reference to a specific ethanol-TCTFE mixture is in document 2 where it is proposed to use a mixture containing 4% ethanol, which is essentially the azeotropic mixture, in a process which however does not involve the use of an evaporator.

- 5.3 From the foregoing discussion of prior art it emerges that the process of Claim 1 differs from that disclosed in documents 1, 2 and 3 in:
 - (a) starting with and subsequently topping up one or more parts of the system with an ethanol-TCTFE mixture containing ethanol in a concentration of from 4.5 to 7.8% by weight;
 - (b) the rectification of the aqueous phase from the water separator in a rectifying unit to separate the ethanol from the water in the aqueous phase; and
 - (c) recycling of the ethanol recovered from the rectifying unit to the water separating zone.

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- 5.4 The problem which these measures are intended to solve is that of automatically maintaining the composition of the bath in the dewatering tank within a certain range which is highly effective in dewatering the articles in the face of loss of solvent vapour through the space communicating with the atmosphere on the one hand and an unpredictable selective loss of ethanol with the water discharged from the water separator on the other.
- 5.5 In the Board's view it is plausible on the basis of the information contained in the application itself that the combination of the measures (a) to (c) listed above will in fact solve this problem.
- 5.6 The only references in document 1 to loss of solvent from the system as a whole are at column 5, lines 108-111 and column 9, lines 50-52 where it is stated that there is no substantial loss, except incidentally as vapour loss, but that solvent make-up can be added to the system where necessary.

It is, however, also pointed out at page 3, lines 3-6 in connection with one-component solvents that if the liquid (water) to be removed is more than about 5% by weight soluble in the solvent efficient separation of the liquid from the solvent is more difficult. In document 3 it is stated that under such circumstances sufficiently efficient separation of water from the solvent is not possible according to the described process and at column 5, lines 72-75 a warning is given against agitating the bath since that would promote solubility of the water in the solvent and complicate subsequent water separation.

In none of documents 1, 3 and 4 is there however any discussion or recognition of problems caused by solubility of one component of a two-component solvent in water.

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5.7 Nevertheless the Board takes the view that the skilled man considering operating the process described in these documents, being aware of the substantial miscibility of ethanol with water, which is the very reason for its proposed use, and of the comparatively small proportion of it in the solvent mix (5-20%) would immediately appreciate, having regard to the above-mentioned references to other difficulties caused by miscibility of water and solvent, that when using the process intensively ethanol will be lost with the discharged water, thus leading to a change in the composition of the solvent composition even if it is initially azeotropic.

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- 5.8 The process known from document 1 already makes use of a water separator for separating the water to be discharged from the solvent which is to be retained and recirculated. Since the separator relies on gravity it can only be effective when the components to be separated are immiscible. Having regard to this it can only be seen as a logical response on the part of the skilled man to the problem of loss of ethanol, which is known to be miscible with water, to augment the gravity system with a system such as a rectifier, which is known to be effective in separating ethanol from water and to return the separated ethanol to the system just as the solvent from the gravity water separator is returned. In the known process a drier (43 in Fig. 10) is used in an analogous way to complete the task of separating the water from the solvent to be recirculated which gravity is unable to accomplish alone.
- 5.9 For these reasons the Board finds that the provision of characterising features (2) and (3) of Claim 1 does not in itself require an inventive step.

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5.10 In the appealed decision the Examining Division concluded that it would be obvious when using the TCTFE-ethanol bath proposed as one of a large number of possible alternatives in document 1, to use an azeotropic composition because of the preference for such compositions expressed in that document, and that since, as stated in the original description of the application in suit, an azeotropic TCTFE-ethanol mixture contains at least 4% of ethanol there could be nothing inventive in selecting mixtures containing from 4.5-7.8% ethanol.

This argument was based on the assumption that such mixtures were azeotropic, which is not the case. In fact only a single azeotropic mixture of TCTFE and ethanol exists, which according to CRC Handbook of Chemistry and Physics, 59th edition, D-21, contains only 3.8% ethanol.

Therefore, contrary to the opinion of the Examining Division, the expressed preference in documents 1, 3 and 4 for a constant boiling azeotropic mixture in the process using an evaporator leads the skilled man away from rather than towards use of non-azeotropic mixtures such as those claimed. Though the use of non-azeotropic mixtures is not ruled out in these documents there is nothing to suggest they might have any advantage to offset the need for eventual compositional adjustment. Furthermore, composition

adjustment as advocated in that document if it is decided to use such mixtures does not suggest the addition to the system of mixture having the same composition range as that originally in the system as required in Claim 1.

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5.11 On the other hand the appellant has recognised (see Fig. 2 of the application) not only that ethanol-TCTFE mixtures in general provide superior water separation to certain of the other mixtures, including the preferred one, proposed in documents 1, 3 and 4, but also that the separation efficiency of the claimed composition is greater than that of the azeotropic mixture and rises sharply within ethanol concentration with the range.

The vapour generated by the evaporator which as seen from Fig. 3 will have a composition which is essentially constant at about 4% ethanol for a wide range of ethanol concentration in the evaporator bath, will fill the space over the dewatering bath and water-separator and thus suppress evaporation from these which might otherwise lead to a build up of ethanol concentration. Such a build-up will however occur in the evaporator bath thus enabling more perfect drying to be obtained by finally dipping the articles to be dried in this bath.

5.12 In view of the above considerations the Board finds that the process claimed in Claim 1 does involve an inventive step. The claim is therefore allowable.

The dependent Claim 2 relates to a particular embodiment of the process of Claim 1 and is likewise allowable.

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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 grant a patent on the basis of the following items:
 - Description pages 1-19 as presented during the oral proceedings
 - Claims 1 and 2 as presented during the oral proceedings
 - Drawings Figures 1-4 and 7 as originally filed, Figures 5 and 6 filed on 31 August 1983.

The Registrar:

The Chairman:

F.Klein

K.Lederer