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**Datasheet for the decision
of 18 February 2014**

Case Number: T 0637/10 - 3.3.10

Application Number: 99929890.4

Publication Number: 1110936

IPC: C07C17/20, C07C17/21,
C07C19/08, B01J27/132

Language of the proceedings: EN

Title of invention:
PROCESS FOR PRODUCING FLUOROETHANE

Patent Proprietor:
DAIKIN INDUSTRIES, LIMITED

Opponent:
Ineos Fluor Holdings Ltd.

Headword:
PROCESS FOR PRODUCING FLUOROETHANE/DAIKIN

Relevant legal provisions:
EPC Art. 54, 56, 100(b), 114(2)

Keyword:
Sufficiency of disclosure - (yes)
Late-filed document - admitted (no)
Novelty - (yes)
Inventive step - (yes)

Decisions cited:

Catchword:



**Beschwerdekammern
Boards of Appeal
Chambres de recours**

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Case Number: T 0637/10 - 3.3.10

**D E C I S I O N
of Technical Board of Appeal 3.3.10
of 18 February 2014**

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Decision under appeal: **Interlocutory decision of the Opposition
Division of the European Patent Office posted on
11 February 2010 concerning maintenance of the
European Patent No. 1110936 in amended form.**

Composition of the Board:

Chairman: P. Gryczka
Members: J.-C. Schmid
F. Blumer

Summary of Facts and Submissions

I. The Appellant (Opponent) lodged an appeal against the interlocutory decision of the Opposition Division which found that the European patent No. 1 110 936 amended according to the then pending auxiliary request 1 met the requirements of the EPC.

II. Notice of opposition had been filed by the Appellant requesting revocation of the patent-in-suit in its entirety on the grounds of lack of novelty and inventive step (Article 100(a) EPC), of insufficient disclosure (Article 100(b) EPC) and of extending the subject-matter of the patent-in-suit beyond the content of the application as filed (Article 100(c) EPC. Inter alia the following documents were submitted in the opposition proceedings:

(2) US-A-5 334 787,

(4) EP-A-0 687 660 and

(5) EP-A-0 811 592.

III. According to the Opposition Division the patent-in-suit described the preparation of the fluorochromium oxide catalyst having the required fluorine content. The examples disclosed the preparation of HCF-125 as the main component. Hence, the specification of the patent-in-suit contained sufficient information to enable the skilled person to carry out the invention across the whole breadth of the claims.

Document (4) disclosed a two-stage process for preparing HCF-125 wherein HCFC-123 and /or HCFC-124 obtained by fluorination of perchloroethylene were

fluorinated with HF in the presence of a chromium oxide catalyst. In examples 1 and 5 the catalyst was a fluorochromium oxide catalyst having a fluorine content of 29%. Document (5) disclosed a process where HCF-125 and/or HCFC-124 were prepared by fluorination of HCFC-123 in the presence of a fluorinated chromium oxide. The fluorine content of the catalyst was, however, not disclosed. It was not possible to conclude without any doubt that the catalyst used in the process of the examples of document (4) or (5) reached a fluorine content of more than 30% by weight during the fluorination process, since the opponent did not provide the necessary experimental evidence showing the increase of the fluorine content of catalyst under the experimental conditions used. The claimed subject-matter was therefore novel with respect to documents (4) and (5).

Document (2) represented the closest prior art to the invention. The technical problem underlying the patent-in-suit was seen in the provision of an improved fluorination process for making HCF-125 with reduced CFC production. The solution was the process of claim 1 characterized by the fluorine content of the fluorochromium catalyst of not less than 30% by weight.

The examples and comparative examples in the patent-in-suit as well as the experimental evidence dated 10 September 2009 showed that this technical problem was credibly solved. None of the documents cited by the Appellant reported that the fluorine content of the catalyst had an effect on the selectivity of the reaction. Hence, it was not obvious for the skilled man to use a catalyst with a fluorine content of more than 30% by weight to improve the reaction selectivity. The

subject-matter of claim 1 therefore involved an inventive step.

- IV. During the oral proceedings held on 18 February 2014 before the Board, the Respondent (Proprietor of the patent) withdrew its previous main and auxiliary requests 1 to 11 and defended the maintenance of the patent in suit on the basis of the auxiliary requests 12 to 20 filed with the letter of 23 December 2013, auxiliary request 12 becoming the main request.

Claim 1 of the main request read as follows:

"1. A manufacturing method for fluorine-containing ethane characterized by that fluorochromium oxide with a fluorine content of not less than 30 wt.% is used as a catalyst wherein fluorine-containing ethane which contains 1,1,1,2,2-pentafluoroethane as the main component and 2-chloro-1,1,1,2-tetrafluoroethane and/or 2,2-dichloro-1,1,1-trifluoroethane as reaction products is obtained by fluorinating at least one selected from the group composed of tetrachloroethylene, 2,2-dichloro-1,1,1-trifluoroethane and 2-chloro-1,1,1,2-tetrafluoroethane with hydrogen fluoride, wherein 2-chloro-1,1,1,2-tetrafluoroethane and/or 2,2-dichloro-1,1,1-trifluoroethane from the reaction mixture are mainly circulated in said fluorination reaction, wherein the chromium oxide is fluorinated to a fluorine content of not less than 30 wt.% at least at the step immediately before the fluorination reaction of the above starting materials to give the fluorochromium oxide and subsequently submitted to the reaction."

- V. According to the Appellant, the requirement of claim 1 that 1,1,1,2,2-pentafluoroethane (HFC-125) was the main

component meant that it was the largest single component entity in the composition resulting from the claimed method. Furthermore claim 1 specifically covered methods in which tetra-chloroethylene (PCE) was the only starting product. The requirement that 2-chloro-1,1,1,2-tetrafluoroethane (HCFC-124) and/or 2,2-dichloro-1,1,1-trifluoroethane (HCFC-123) from the reaction mixture were mainly circulated in said fluorination reaction meant that those compounds were recycled. The invention could not be reproduced starting with PCE as the only starting product as revealed by examples 7 and 8 of the patent specification which showed that the fluorination of PCE did not give HFC-125 as the largest single component entity. None of the examples of the patent-in-suit fell within the scope of the claims, since the recycling of HCFC-123 and/or HCFC-124 was missing. Furthermore, the specification of the patent-in-suit did not teach which benefit was associated with the examples. The invention was therefore not sufficiently disclosed. It was general knowledge for the skilled person that a sufficiently high fluorination level of the catalyst would be achieved if the hydrofluorination conditions were maintained for a sufficient time. The reproduction of the fluorination process disclosed in example 7 of document (5) with a fluorochromium catalyst having an initial fluorine content of 17.9% by weight showed that after 442 hours the fluorine content of the catalyst had increased to 42.9% by weight, i.e. well above the claimed requirement of above 30% by weight. Hence, example 7 of document (5) was novelty-destroying for claim 1.

With the statement setting out the grounds of appeal the Appellant further submitted document

(9) US-A-5 571 770

to object to novelty of the claimed subject-matter, since the catalyst disclosed in example 1 therein had a fluorine level of 44.1% by weight.

As regards inventive step, document (5) represented the closest prior art. The objective of document (5) was to minimise the production of undesirable by-products. Example 7 disclosed a process where 42.0% HCF-125, 28% HCFC-124 and 30.0% HCFC-123 were obtained, showing that if present, less than 0.05% unwanted CFC by-products were produced. None of the examples of the patent-in-suit showed that when carrying out fluorination with the catalyst of claim 1 and recycling HCFC-123 and/or HCFC-124, HCF-125 was obtained as the major component. Furthermore, elevated HCF-125 levels and reduced CFC levels, if indeed they did occur, were scientifically intuitive and part of the common general knowledge of the skilled worker. Hence the claimed subject-matter lacked an inventive step.

VI. The Respondent objected to the admission of document (9) in the appeal proceedings. It was not shown that the catalyst disclosed in example 1 of this document was a fluorochromium oxide catalyst. Furthermore, there was no evidence that the fluorine content of the prepared catalyst was the sum of the fluorine content of the reactants. Thus, document (9) did not provide a direct and unambiguous disclosure of the catalyst of the claimed process.

The expression "1,1,1,2,2-pentafluoroethane as the main component" in claim 1 did not mean that this compound should be the major product in terms of weight in the reaction mixture, but rather that it was the most

important compound in the sense of being the target compound of the claimed process. The invention could also be carried with PCE as the sole starting material. In example 7, the selectivity reflected by the ratio CFCs/HFC-125 was 0.443, while it was only 0.662 in comparative example 6. The invention was therefore sufficiently disclosed to be carried out by a skilled person over the whole claimed scope.

None of the cited documents disclosed a process comprising the step of fluorinating the chromium oxide to a fluorine content of not less than 30 wt.% at least at the step immediately before the fluorination reaction of PCE, HCFC-123 or HCFC-124. The claimed subject-matter was therefore novel. The technical problem underlying the patent-in-suit was the provision of a manufacturing method for fluorine-containing ethane having an improved selectivity to HFC-125 (over chlorofluorocarbons contaminants (CFCs)). The technical problem was solved over the whole scope of the claim, as sufficiently proved by the examples and comparative example of the patent-in-suit, even by example 7 objected to by the Appellant, since it showed also an improved selectivity reflected by the CFCs/HFC-125 ratio. There was no pointer in the cited prior art to the proposed solution, i.e. to fluorinate the catalyst to a level of at least 30% by weight before the reaction, in order to improve the selectivity toward HFC-125. Accordingly, the claimed subject-matter involved an inventive step.

VII. The Appellant requested in writing that the decision under appeal be set aside and the patent be revoked.

The Respondent requested that the decision under appeal be set aside and the patent be maintained upon the

basis of claims 1 to 4 of the main request, filed as twelfth auxiliary request with letter dated 23 December 2013, or, subsidiarily, on the basis of any one of the auxiliary requests 1 to 8, filed as auxiliary requests 13 to 20 with the letter dated 23 December 2013.

VIII. Oral proceedings were held in the absence of the Appellant, which after having been duly summoned, informed the Board that it would not attend. At the end of the oral proceedings the decision of the Board was announced.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments*

Compared to claim 1 of the patent-in-suit as granted claim 1 of the main request comprises the additional feature that "the chromium oxide is fluorinated to a fluorine content of not less than 30% by weight at least at the step immediately before the fluorination reaction of the above starting materials to give the fluorochromium oxide and subsequently submitted to the reaction" according to the disclosure of page 9, lines 5 to 10 of the application as filed. Furthermore, the term "when" has been amended into "wherein" in order to render the technical feature following this term mandatory in the claimed process.

The amendments carried out in claim 1 restrict the protection conferred by the patent as granted. Therefore, there are no objections to the amendments made in present claim 1, which finding was not

contested by the Appellant. Dependant claims 2 to 4 are identical to claims 2 to 4 of the patent-in-suit as granted.

The requirements of Article 123(2) and (3) EPC are thus satisfied.

3. *Sufficiency of disclosure (Article 100(b) EPC)*

The claimed invention relates to a manufacturing method for fluorine-containing ethane which contains HFC-125 as the main component and HCFC-123 and/or HCFC-124 as reaction products.

The method of claim 1 comprises the steps of

- fluorinating chromium oxide to a fluorine content of not less than 30 wt.%,
- subsequently using that fluorinated chromium oxide as a catalyst to fluorinate at least one compound selected from the group composed of tetrachloroethylene (PCE), HCFC-123 and HCFC-124 with hydrogen fluoride and,
- circulating in said fluorination reaction HCFC-123 and/or HCFC-124 from the reaction mixture.

3.1 According to the Appellant the invention could not be reproduced when the feedstock was PCE as envisaged by claim 1, since examples 7 and 8 of the patent specification showed that the fluorination of PCE did not give HFC-125 as the major product, with the consequence that the invention could not be carried out in the whole scope of claim 1.

However, claim 1 does not require that HCF-125 is obtained as the major product by reaction of the feedstock with HF. Claim 1 stipulates that HCF-125 is obtained as the main component and HCFC-123 and

HCFC-124 as reaction products. In the Board's interpretation the wording "HCF-125 is obtained as the main component and HCFC-123 and R124 as reaction products" means that HCF-125 is the target product, which is recovered, while HCFC-123 and HCFC-124 are recycled in the fluorination reaction, what claim 1 expressly foresees, as acknowledged by the Appellant.

Example 7 of the patent-in-suit shows that the step of fluorinating PCE in the presence of the chromium oxide catalyst gives a reaction product which contains 22.3% HCFC-123, 13.6% HCFC-124 and 4.01% HFC-125. Recovering HFC-125 and, recycling HCFC-123 and HCFC-124 in the fluorination reaction, are within the normal ability of the skilled person. Hence the Board concludes that, contrary to the Appellant's assertion, example 7 of the patent-in-suit shows that the claimed process can be carried out by the skilled person also when PCE is the starting product.

- 3.2 According to the Appellant, the claimed invention was not sufficiently disclosed in the patent-in-suit, since none of the working examples disclosed therein was within the scope of claim 1.

In the present case, the processes described in the working examples of the patent-in-suit are not according to claim 1 only because they do not comprise the step of recycling HCFC-123 and/or HCFC-124. However, recycling compounds from a reaction mixture is common knowledge for the skilled person. Therefore, the disclosure of a process in the patent-in-suit, which does include the recycling step, is not a requisite for the skilled person to be able to carry out the process according to claim 1.

3.3 According to the Appellant the invention was not sufficiently disclosed, since the specification did not teach which benefit was associated with the inventive examples.

With respect to sufficiency of disclosure, the relevant question is whether the patent-in-suit provides sufficient information which enables the skilled person to perform the invention as defined in the claims. The Appellant's objection mainly concerns an alleged absence of effect of the claimed process. However, in the present case the effect achieved by the claimed process is not relevant for sufficiency of disclosure, as claim 1 only requires characteristics relating to the steps of the process without specifying any effect to be achieved. Thus, the Appellant's objection must also be rejected.

3.4 Consequently, the Appellant's objection to the sufficiency of the disclosure of the invention fails.

4. *Late filed document (9)*

The Appellant submitted document (9) with its statement of the grounds of appeal as a further novelty-destroying prior art document. The relevance of this document was contested by the Respondent, which requested not to admit it into the appeal proceedings, since it was late filed.

The Appellant relied on example 1 of document (9), which discloses the preparation of a catalyst by reacting an aqueous mixture of CrO_3 , MgF_2 and ZnF_2 with ethanol under reflux for a period of 16 hours, filtering the product and drying the product for 6 hours at 140°C , alleging that the chromia catalyst

obtained in this example had a fluorine content of 44.1 wt.%. In fact, the Appellant arrived to that result by speculating that the full fluorine content of all of the materials used to prepare the catalyst, i.e. of MgF_2 and ZnF_2 , will be present in the final catalyst. However, the Appellant did not provide any explanation, let alone any evidence, showing that the chromium oxide had been effectively fluorinated by this process and that the catalyst incorporated the full content of fluorine present in the reactants. Accordingly, document (9) does not appear *prima facie* relevant to the novelty of the subject-matter of claim 1.

Under these circumstances, the Board exercises its discretionary power conferred by Article 114(2) EPC to disregard this document.

5. *Novelty*

According to the Appellant the subject-matter of the claims maintained by the Opposition Division was not novel with respect to documents (4) and (5). The Appellant, however, made no novelty-objection based on these documents with respect to the claims of the present request, which comprise the additional feature that the chromium oxide is fluorinated to a fluorine content of not less than 30% by weight at least at the step immediately before the fluorination reaction of PCE, HFC-123 or HCFC-124. This feature is disclosed neither in document (4), nor in document (5).

The Board therefore concludes that the subject-matter of claim 1 is novel with respect to documents (4) and (5).

6. *Inventive step*

6.1 *Closest prior art*

The Board considers, in agreement with the opposition division and the Parties, that either document (2), (4) or (5) represents the closest state of the art to the invention, since each of these documents discloses a process for the preparation of HFC-125 which comprises all the features of the claimed process, except the step of fluorating the catalyst to a fluorine content of at least 30% by weight at least at the step immediately before the fluorination reaction of PCE, HCFC-123 and/or HCFC-124.

6.2 *Technical problem underlying the patent-in-suit*

According to the Respondent, the technical problem underlying the patent-in-suit was to provide a manufacturing method for fluorine-containing ethane having an improved selectivity to HFC-125 over chlorofluorocarbons contaminants (CFCs).

6.3 *Proposed solution*

The solution proposed by the patent-in-suit is the method of claim 1 characterized by fluorinating the chromium oxide to a fluorine content of not less than 30 wt.% before the fluorination reaction of PCE, HCFC-123 and/or HCFC-124.

6.4 *Success*

The Respondent *inter alia* referred to examples 1, 2, 3 and comparative examples 1 and 2 of the patent-in-suit

in order to show that the claimed process represented a solution to the above defined technical problem.

In these examples, fluorochromium oxide catalysts having different fluorine contents were prepared, and subsequently were used as the catalyst of the fluorination reaction of HCFC-124. The reaction gas was then analysed by chromatography to determine the selectivity ratio CFCs/ HFC-125. When the reaction was carried out with a catalyst having a fluorine content of 12 wt.%, 25 wt.%, 31.4 wt.%, 35.2 wt.% and 41.5 wt.%, the values of the CFCs/HFC-125 ratios were 0.281, 0.260, 0.221, 0.150 and 0.099 respectively.

These results reveal that there is a decrease of the CFCs/HFC-125 ratios when the fluorine content of the fluorochromium oxide catalyst increases, indicating that the selectivity towards HFC-125 over CFCs is enhanced by increasing the fluorine content of the fluorochromium oxide catalyst.

According to the Appellant, these experiments could not show a benefit in term of reduced CFCs/HFC-125 ratio of a process according to claim 1, since the experimental data provided related to single pass experiments, not any of which involved recycling.

The step of recycling a product does not form new by-products, since no chemical reaction takes place. Both in the comparative examples and in the examples according to the invention, the chemical fluorination reactions are operated without recycling. It is thus convincingly shown that the greater selectivity towards HCF-125 is caused by the characterising feature of the process, namely by the fluorine content of the chromium oxide of not less than 30% by weight at the beginning

of the fluorination reaction of PCE, HCFC-123 and/or HCFC-124. The Appellant's argument must therefore be rejected.

According to the Appellant, no problem was solved with respect to document (5), since the process of example 7 of that document already achieved a product composition having 42.0% by mole of HCF-125, 28.0 % by mole of HCFC-124 and 30.0% by mole of HCFC-123 suggesting that unwanted CFC by-products, if present, are present at a level of at most 0.05%, indicating a much better selectivity than that obtained by the processes according to the patent-in-suit.

The Appellant is, however, speculating on the absence or quasi absence of unwanted CFC by-products in example 7 of document (5), which discloses the relative proportions of HCF-125, HCFC-124 and HCFC-123 without reporting on CFCs. The amounts of HCF-125, HCFC-124 and HCFC-123 present in the product reaction are defined in this example in term of molar ratio. Unwanted CFC by-products, however, do not refer to a single compound, but to a mixture thereof. Accordingly, even following the Appellant's allegation that the CFCs compounds are present at a level of no more than about 0.05% by mole, that would not permit to conclude that the process disclosed in example 7 of document (5) achieves a greater selectivity towards HFC-125 over the CFCs than the process of present claim 1. In this respect, it must be reminded that the experimental data of the patent-in-suit, namely both the examples and the comparative examples, clearly show that unwanted CFCs are formed when HCFC-123, HCFC-124 and/or PCE are fluorinated with HF in the presence of a fluorinated chromium oxide. Accordingly, this Appellant's argument which is not supported by the facts must be rejected.

Consequently, the Board is satisfied that the technical problem underlying the patent in suit of improving the selectivity towards HFC-125, has been successfully solved by the proposed solution, i.e. by the process according to claim 1 characterized by fluorinating the chromium oxide to a fluorine content of not less than 30% by weight before the fluorination reaction of PCE, HCFC-123 and/or HCFC-124.

6.5 Obviousness

Finally, it remains to be decided whether or not the proposed solution to this objective technical problem is obvious in view of the cited state of the art. None of the documents (2), (3) and (5) teach that an improvement of selectivity towards HCH-125 can be obtained by fluorinating the chromium oxide to a fluorine content of not less than 30% by weight before the fluorination reaction of PCE, HCFC-123 and/or HCFC-124. The Appellant did not rely on any further documents, except on the late-filed document (9) which is, however, not admitted in the proceedings, and the Board is not aware of further documents relevant in this respect. Thus, the Board concludes that none of the cited documents renders the proposed solution obvious.

Therefore, the subject-matter of claim 1 of the main request, and for the same reason, that according to dependent claims 2 to 4 involve an inventive step within the meaning of Article 56 EPC.

7. Since the main request is allowable for the reasons set out above, there is no need for the Board to decide on the lower ranking auxiliary requests 1 to 8.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the opposition division with the order to maintain the patent on the basis of claims 1 to 4 of the main request, filed as the twelfth auxiliary request with the letter dated 23 December 2013, and a description yet to be adapted.

The Registrar:

The Chairman:



C. Vodz

P. Gryczka

Decision electronically authenticated