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**Datasheet for the decision  
of 12 May 2022**

**Case Number:** T 1579/18 - 3.3.10

**Application Number:** 10768151.2

**Publication Number:** 2493836

**IPC:** C07C17/386, C01B7/19, C07C17/38

**Language of the proceedings:** EN

**Title of invention:**  
HYDROGEN FLUORIDE-HFC-254EB AZEOTROPE AND ITS USES

**Patent Proprietor:**  
The Chemours Company FC, LLC

**Opponent:**  
ARKEMA FRANCE

**Headword:**

**Relevant legal provisions:**  
EPC Art. 100(a), 100(b), 100(c)

**Keyword:**  
Grounds for opposition - extension of subject-matter (no) -  
insufficiency of disclosure (no) - lack of patentability (no)

**Decisions cited:**

T 0199/15

**Catchword:**



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Case Number: T 1579/18 - 3.3.10

**D E C I S I O N**  
**of Technical Board of Appeal 3.3.10**  
**of 12 May 2022**

**Appellant:** ARKEMA FRANCE  
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**Decision under appeal:** **Decision of the Opposition Division of the European Patent Office posted on 17 April 2018 rejecting the opposition filed against European patent No. 2493836 pursuant to Article 101(2) EPC.**

**Composition of the Board:**

<b>Chair</b>	P. Gryczka
<b>Members:</b>	R. Pérez Carlón
	F. Blumer

## Summary of Facts and Submissions

- I. The appellant-opponent lodged an appeal against the decision of the opposition division rejecting its opposition against European patent No. 2 493 836.
- II. Notice of opposition had been filed on the grounds of added subject-matter (Article 100(c) EPC), insufficiency of disclosure (Article 100(b) EPC) and lack of inventive step (Article 100(a) EPC).
- III. The documents filed include the following:

D1 WO 2009/105517 A2  
D6 WO 94/27940

- IV. The patent as granted contains four independent claims which read as follows.

*"1. A process for separating 1,1,1,2-tetrafluoropropane from a mixture comprising 1,1,1,2-tetrafluoropropane, 1,1,1,2,3-pentafluoropropane and hydrogen fluoride comprising:*

*subjecting said 1,1,1,2-tetrafluoropropane, 1,1,1,2,3-pentafluoropropane and hydrogen fluoride mixture to a distillation step, forming a column distillate composition comprising an azeotropic or near-azeotropic composition of said 1,1,1,2-tetrafluoropropane and hydrogen fluoride, and a bottoms composition of 1,1,1,2,3-pentafluoropropane containing less than 100 ppm (mole basis) of hydrogen fluoride, wherein said near-azeotropic composition is characterised by a difference between dew point pressure and bubble point pressure that is less than or equal to 3%, based upon*

*bubble point pressure.*

*3. A process for separating 1,1,1,2-tetrafluoropropane from 1,1,1,2,3-pentafluoro-propane comprising forming a mixture comprising 1,1,1,2-tetrafluoropropane, 1,1,1,2,3-pentafluoropropane and hydrogen fluoride; and subjecting said 1,1,1,2-tetrafluoropropane/1,1,1,2,3-pentafluoropropane/hydrogen fluoride mixture to a distillation step, forming a column distillate composition comprising an azeotropic or near-azeotropic composition of said 1,1,1,2-tetrafluoropropane and hydrogen fluoride, wherein said near-azeotropic composition is characterised by a difference between dew point pressure and bubble point pressure that is less than or equal to 3%, based upon bubble point pressure.*

*6. A process for the separation of 1,1,1,2-tetrafluoropropane from a mixture comprising 1,1,1,2-tetrafluoropropane and hydrogen fluoride, said process comprising:*

- a) subjecting said mixture to a first distillation step in which a composition enriched in either (i) hydrogen fluoride or (ii) 1,1,1,2-tetrafluoropropane is removed as a first distillate composition with a first bottoms composition being enriched in the other of said components (i) or (ii); and*
- b) subjecting said first distillate composition to a second distillation step conducted at a different pressure than the first distillation step in which the component enriched as a first bottoms composition in (a) is removed in a second distillate with a second bottoms composition enriched in the same component which was enriched in the first distillate composition.*

*7. An azeotropic or near-azeotropic composition consisting of from 35.2 mole percent to 78.4 mole percent 1,1,1,2-tetrafluoropropane, and hydrogen fluoride, wherein the vapor pressure is from 2.6 psia to 345.2 psia (17.9 kPa to 2.39 MPa) at a temperature of from -40°C to 100°C and said near-azeotropic composition is characterized by a difference between dew point pressure and bubble point pressure that is less than or equal to 3%, based upon bubble point pressure."*

- V. The opposition division concluded that the claims of the patent as granted found the required basis in the application as originally filed. It also concluded that the claimed subject-matter was sufficiently disclosed for it to be carried out by a skilled person.

Document D6 was the closest prior art for the processes of claims 1 to 5. The problem underlying the claimed invention was the provision of an alternative process for the separation of fluorinated compounds from a mixture of hydrogen fluoride (HF), 1,1,1,2-tetrafluoropropane and 1,1,1,2,3-pentafluoropropane. The solution proposed by claims 1 to 5 was characterised by forming an azeotropic or near-azeotropic composition of HF and 1,1,1,2-tetrafluoropropane. As azeotropes were not predictable, the claimed solution would not have been obvious for a skilled person. The claimed processes were thus inventive.

With respect to the azeotropic mixture of claim 7, document D1 was the closest prior art. The problem underlying the claimed invention was to provide an

alternative azeotropic composition. The claimed solution, characterised by containing 1,1,1,2-tetrafluoropropane and HF, would not have been obvious for a skilled person and was thus inventive.

Lastly, the separation of the components of the azeotrope by pressure-swing distillation would not have been envisaged by the skilled person having regard to D6 without the benefit of hindsight. Claim 6 was thus also inventive.

VI. The arguments of the appellant-opponent were as follows.

The features "containing less than 100 ppm (mole basis) of hydrogen fluoride" in claims 1 and 5; "near-azeotropic composition is characterised by a difference between dew point pressure and bubble point pressure that is less than or equal to 3%, based upon bubble point pressure" in claims 1, 3 and 7; and "consisting" in claim 7 did not find the required basis in the application as originally filed.

Examples 3 and 4 of the patent did not credibly show that the azeotrope of claim 7 existed. For this reason, the claimed invention was not sufficiently disclosed for it to be carried out by a skilled person.

Document D6 was the closest prior art. It disclosed mixtures containing 1,1,1,2-tetrafluoropropane 1,1,1,2,3-pentafluoropropane and HF. D6 taught to separate those mixtures by distillation. By doing so, the skilled person would have arrived at the claimed azeotrope and the claimed processes without requiring inventive skills. The claimed subject-matter was thus not inventive.



VII. The arguments of the respondent-patent proprietor were as follows.

The features objected to by the appellant-opponent found the required basis on page 10, lines 1-3; page 5, lines 6-9 and in claim 11 as originally filed.

The claimed azeotrope was sufficiently disclosed by examples 3 and 4 of the patent. There was no evidence on file which proved them wrong. The claimed invention could thus be carried out.

Document D6 was the closest prior art. The problem underlying the claimed invention was to separate HF from 1,1,1,2,3-pentafluoropropane. The claimed solution made use of the azeotrope of claim 7. Having regard to their close boiling points, the skilled person had no reason to distil the mixtures obtained in the examples of D6 to separate HF from 1,1,1,2,3-pentafluoropropane. The claimed solution was thus inventive.

VIII. Oral proceedings before the board took place on 12 May 2022.

IX. The final requests of the parties were as follows.

The appellant-opponent requested that the decision under appeal be set aside and that European patent No. 2 493 836 be revoked.

The respondent-patent proprietor requested that the appeal be dismissed or that the patent be maintained with the claims of any of auxiliary requests 1 to 17, all filed with its letter dated 15 January 2018.

- X. At the end of the oral proceedings, the decision was announced.

### **Reasons for the Decision**

1. The appeal is admissible.
2. Amendments
  - 2.1 The appellant-opponent argued that a number of features of the claims as granted did not find the required basis in the application as originally filed.

The board informed the parties in a communication dated 14 July 2020 that it was inclined to concur with the reasoning and conclusions of the opposition division that the patent as granted contained no added subject-matter.

The appellant-opponent filed no further arguments in this respect, and the board sees no reason to depart from its preliminary view.

- 2.2 The feature "1,1,1,2,3-pentafluoropropane containing less than 100 ppm (mole basis) of hydrogen fluoride" finds a basis in claims 1 and 5 as originally filed, which required "1,1,1,2,3-pentafluoropropane essentially free of hydrogen fluoride", and the definition of that feature on page 10, lines 1-3 and lines 31-31 of the application as originally filed.

The appellant argued that the amount of hydrogen fluoride (HF) required by claims 1 and 5 was only disclosed in the context of a different process.

However, the process at the bottom of page 9

corresponds to that of claim 1, even if worded as the separation from 1,1,1,2,3-pentafluoropropane of HF and not of 1,1,1,2-tetrafluoropropane. The process steps are identical.

The claimed invention seeks the separation of HF from 1,1,1,2,3-pentafluoropropane. In the context of the claimed invention, "essentially free of HF" cannot thus require the complete absence of HF as this is not technically possible. The skilled reader would thus seek clarification on the degree of purity meant by that feature and would find it on page 10 of the application. No other option is offered. The skilled reader would thus consider that definition to be of general application.

- 2.3 With respect to the feature in claims 1, 3 and 7 requiring a "near-azeotrope" to be characterised by "a difference between dew point pressure and bubble point pressure that is less than or equal to 3%, based upon bubble point pressure", a basis can be found on page 5, lines 6-9.

The appellant argued that this feature was not combined with the other features of claims 1, 3 and 7. However, the definition on page 5, lines 6-9 is of general value, and no other alternative is offered. Trying to determine the meaning of the vague term "near-azeotropic" in the context of the claimed invention, the skilled person would inevitably have resorted to that definition.

- 2.4 Lastly, the feature "consists" in claim 7 of the patent as granted finds a basis in the feature "consists essentially of" in claim 11 as filed as "consists of"

is the sole clear reading of "consists essentially of".

- 2.5 The ground for opposition in Article 100(c) EPC does not preclude the maintenance of the patent as granted.
3. Sufficiency of disclosure
- 3.1 The opposition division concluded that the claimed invention can be carried out by a skilled person.
- 3.2 The appellant-opponent argued that example 3 did not prove the existence of an azeotrope as defined in claim 7. The composition of the distillate did not correspond to that of a binary azeotrope as other components such as hydrogen were also present. In addition, the alleged azeotrope in example 3 differed from the azeotrope in example 4. For these reasons, the claimed invention was not sufficiently disclosed.
- 3.3 The distillate in example 3 of the patent (feed 140 in Table 4) contains, in addition to the claimed azeotrope, hydrogen, which is much more volatile, and a small amount of the product obtained as bottoms, 1,1,1,2,3-pentafluoropropane. Absent evidence to the contrary, hydrogen is expected to distil first, followed by the claimed azeotrope. Some entrainment of 1,1,1,2,3-pentafluoropropane would not be surprising and explains its presence in the distillate.

With respect to the difference between the composition disclosed in example 3 and the azeotrope in example 4, the variation is not large. At 94.7 psia, example 3 discloses that the azeotrope contains 49% 1,1,1,2-tetrafluoropropane (5.26 parts of HF and 5.04 parts of 1,1,1,2-tetrafluoropropane). Averaging the data at 11.9 and 18.1 psia in Table 4 of example 4, about 58% is

obtained.

In addition, example 4 discloses the results from calculations based on phase studies, i.e. it does not disclose experimental results. A difference with the empiric data is thus to be expected.

The appellant-opponent has provided no evidence that an azeotrope of 1,1,1,2-tetrafluoropropane and HF cannot be obtained that contradicts the experimental evidence in the patent, which credibly shows its existence.

For these reasons, the appellant-opponent's argument is not convincing.

3.4 Thus, the ground for opposition in Article 100(b) EPC does not preclude the maintenance of the patent as granted.

4. Inventive step

4.1 The claimed invention relates to an azeotropic composition consisting of 1,1,1,2-tetrafluoropropane and HF (claim 7), its use in the separation from 1,1,1,2,3-pentafluoropropane (claim 1, claim 3) and its separation by swing-distillation (claim 6).

4.2 Closest prior art

The parties agreed at the oral proceedings that document D6 was the closest prior art. This was also the board's view.

The parties also agreed that document D6 did not disclose an azeotrope of 1,1,1,2-tetrafluoropropane and

HF.

4.3 Problem underlying the claimed invention

The respondent-patent proprietor defined the problem underlying the claimed invention as to provide a method for separating a mixture of 1,1,1,2-tetrafluoropropane, HF and 1,1,1,2,3-pentafluoropropane, which allowed 1,1,1,2,3-pentafluoropropane and HF to be separated.

4.4 Solution

The claimed solution is characterised by involving the azeotropic or near-azeotropic composition of 1,1,1,2-tetrafluoropropane and HF of claim 7.

4.5 Success

By distilling off an azeotropic or near-azeotropic mixture of 1,1,1,2-tetrafluoropropane and HF, 1,1,1,2,3-pentafluoropropane can be separated from HF, despite their similar boiling points.

The data provided in example 3 of the patent (Table 4) shows that distilling off the claimed azeotrope allows obtaining a bottoms consisting of 1,1,1,2,3-pentafluoropropane with less than 0.0001 mol% of HF.

The problem as defined above is thus credibly solved by the azeotrope of claim 7 and the processes of claims 1 and 3.

4.6 It thus remains to be decided whether the proposed solution to the objective problem defined above would have been obvious for the skilled person in view of the

prior art.

- 4.6.1 It was not disputed that the mixtures of compounds obtained by the examples of D6 contained 1,1,1,2-tetrafluoropropane, 1,1,1,2,3-pentafluoropropane and HF (examples 1 and 2, step C).

At atmospheric pressure, the boiling points of 1,1,1,2,3-pentafluoropropane (24 °C) and HF (19 °C) are close. 1,1,1,2-tetrafluoropropane is more volatile: its boiling point is close to 0 °C at that pressure. This was not disputed.

The respondent-patent proprietor provided the boiling point of these components at 100 psia. 1,1,1,2,3-pentafluoropropane and HF boil at that pressure within 1 °C.

- 4.6.2 The appellant-opponent argued that document D6 taught to separate these mixtures by conventional means such as distillation (page 4, lines 29-31). By following the explicit instructions of D6 that the mixture be distilled, the skilled person would inevitably have obtained an azeotrope according to claim 7 and would have arrived at separation methods according to claims 1 and 3.

- 4.6.3 However, the mixtures obtained in step C of the examples of D6 do not contain 1,1,1,2-tetrafluoropropane, 1,1,1,2,3-pentafluoropropane and HF only. They also contain non-negligible amounts of 1,1,1,2,3,3-hexafluoropropane and minor amounts of other halogenated compounds.

It was undisputed that azeotropic behaviour is unpredictable, on the one hand, and unavoidable, on the

other.

D6 does not disclose any detail of the distillation of the mixtures obtained in the examples. Neither did the appellant-opponent provide any evidence on the behaviour of these mixtures.

It is in principle possible that distilling these mixtures could have led to obtaining the azeotrope of claim 7. However, it would not have inevitably been the case, either based on the general common knowledge of the skilled person or any available evidence. The reason is that the formation of other, different azeotropic mixtures cannot be excluded.

Without information on the distillation behaviour of the mixtures of D6, the argument that, by following the instructions in D6, the azeotrope of claim 7 and the processes of claims 1 and 3 would have been inevitably obtained is not convincing.

- 4.6.4 In addition, the skilled person would have expected difficulties in separating close-boiling 1,1,1,2,3-pentafluoropropane from HF by distillation. This is even more the case as document D6 discloses that they are expected to form an azeotrope (page 4, line 31).
- 4.6.5 The appellant-opponent further argued that an azeotrope was disadvantageous from an industrial viewpoint as it increased the number of steps required in a separation.

However, the claimed azeotrope is shown to be advantageous as it allows the separation by distillation of two compounds whose boiling points are very close. This argument is thus not convincing.



4.6.6 The appellant-opponent argued that the facts of the case under appeal and that underlying T 199/15 were equivalent. In T 199/15, the board concluded that it was obvious to arrive at the claimed azeotrope following a routine distillation step. The same conclusion should thus be reached in the case at hand.

As in the current case, the relevant state of the art in T 199/15 taught to distil a reaction mixture containing the azeotrope's components. There is, however, a relevant difference between the cases.

In T 199/15, the azeotrope was formed between HF and the product sought, which was the most volatile component of the mixture. By distilling off the product sought, it was unavoidable to obtain the claimed azeotrope. There was no link of the found azeotrope to any technical application.

In the current case, the product to be purified remains as bottoms of the reaction, and the formation of the claimed azeotrope allows the separation of close-boiling 1,1,1,2,3-pentafluoropropane and HF. The skilled person could have distilled off volatile components from the mixture but would not have resorted to distillation to separate HF from the less volatile component, 1,1,1,2,3-pentafluoropropane.

The facts of the case at hand are thus not equivalent to those underlying T 199/15.

4.6.7 The claimed azeotrope (claim 7) and the processes involving that azeotrope (independent claims 1, 3 and 6) are thus inventive (Article 56 EPC).

5. Conclusion

None of the grounds in Article 100 EPC precludes the maintenance of the patent a granted.

**Order**

**For these reasons it is decided that:**

The appeal is dismissed.

The Registrar:

The Chair:



C. Rodríguez Rodríguez

P. Gryczka

Decision electronically authenticated