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**Datasheet for the decision
of 9 May 2025**

Case Number: T 0020/23 - 3.3.10

Application Number: 15862093.0

Publication Number: 3222607

IPC: C07C17/386, C07C19/08,
C07C21/18, B01D3/40

Language of the proceedings: EN

Title of invention:

METHOD FOR ISOLATING HFC-245CB AND (E)-HFO-1234ZE FROM
COMPOSITION CONTAINING BOTH COMPOUNDS

Patent Proprietor:

Daikin Industries, Ltd.

Opponent:

ARKEMA FRANCE

Headword:

Relevant legal provisions:

EPC Art. 83, 56

Keyword:

Sufficiency of disclosure - after amendment
Inventive step

Decisions cited:

Catchword:



Beschwerdekammern
Boards of Appeal
Chambres de recours

Boards of Appeal of the
European Patent Office
Richard-Reitzner-Allee 8
85540 Haar
GERMANY
Tel. +49 (0)89 2399-0

Case Number: T 0020/23 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 9 May 2025

Appellant:

(Opponent)

ARKEMA FRANCE
420, Rue d'Estienne d'Orves
92700 Colombes (FR)

Representative:

Arkema Patent
Arkema France
DRD-DPI
51, Esplanade du Général de Gaulle
CS 10478
92907 Paris La Défense Cedex (FR)

Respondent:

(Patent Proprietor)

Daikin Industries, Ltd.
Umeda Center Building
4-12 Nakazaki-Nishi 2-chome
Kita-ku
Osaka-shi, Osaka 530-8323 (JP)

Representative:

Hoffmann Eitle
Patent- und Rechtsanwälte PartmbB
Arabellastraße 30
81925 München (DE)

Decision under appeal:

**Interlocutory decision of the Opposition
Division of the European Patent Office posted on
19 October 2022 concerning maintenance of the
European Patent No. 3222607 in amended form.**

Composition of the Board:

Chair	P. Gryczka
Members:	R. Pérez Carlón
	L. Basterreix

Summary of Facts and Submissions

I. The appellant (opponent) lodged an appeal against the opposition division's interlocutory decision on the maintenance of European patent No. 3222607 in amended form, on the basis of the first auxiliary request in the opposition proceedings. That auxiliary request forms the respondent's (patent proprietor's) main request in the appeal.

II. Notice of opposition had been filed on the grounds of insufficiency of disclosure (Article 100(b) EPC) and lack of novelty and inventive step (Article 100(a) EPC).

III. The following documents are relevant to the present decision.

D9 WO 2008/054782 A1
D10 US 2004/0167366 A1
D11 WO 02/12153 A1
D12 US 2011/0270001 A1

IV. Auxiliary request 1 at opposition has only one independent claim, which reads as follows.

"A method for separating 1,1,1,2,2-pentafluoropropane (HFC-245cb) and (E)-1,3,3,3-tetrafluoropropene ((E)-HFO-1234ze), the method comprising subjecting a composition comprising the HFC-245cb and the (E)-HFO-1234ze to extractive distillation in the presence of at least one extraction solvent selected from the group consisting of halogenated hydrocarbons, halogenated unsaturated hydrocarbons, nitriles, ketones, carbonates, alcohols, hydrocarbons, esters,

and ethers;

wherein the halogenated hydrocarbon is at least one selected from the group consisting of 1,1-dichloro-3,3,3-trifluoro propane, 1,1,3-trichloro-3,3-difluoro propane, 1,1,2-trichloro-3,3,3-trifluoro propane, 1,1,1,3,3-pentachloro propane, and carbon tetrachloride;

wherein the halogenated unsaturated hydrocarbon is at least one selected from the group consisting of 1,2,3-trichloro-3,3-difluoro propene, 1,2,3,3-tetrachloro-3-fluoro propene, 1,3-dichloro-3,3-difluoro propene, 2,3-dichloro-3,3-difluoro propene, 1,3,3-trichloro-3-fluoro propene, and 2,3,3-trichloro-3-fluoro propene;

wherein the alcohol is at least one selected from the group consisting of methanol, ethanol, isopropanol, butanol, and 1-(trifluoromethyl)-2,2,2-trifluoro ethanol."

- V. The opposition division concluded that the claimed invention was sufficiently disclosed for it to be carried out by a person skilled in the art, and that the method in claim 1 of auxiliary request 1 was novel. Only D9 and D10 related to the separation of the compounds defined in claim 1 and were thus suitable starting points for examining inventive step. The problem underlying the claimed invention was to provide a further process for separating HFC-245cb and (E)-HFO-1234ze. The claimed solution was characterised by an extractive distillation using the solvents defined in claim 1; this would not have been obvious to a skilled person in view of the prior art and was thus inventive.

VI. With the reply to the statement of grounds of appeal, the appellant filed auxiliary requests 1 to 4 from the opposition proceedings and auxiliary request 5; auxiliary request 1 is the main request in the appeal. The present decision keeps the numbering of auxiliary requests 2 to 5; there is thus no request named "auxiliary request 1".

Claim 1 of auxiliary request 2 has the features of claim 1 of the main request, and adds the following.

"wherein the relative volatility of (E)-HFO-1234ze to HFC-245cb in the presence of the at least one extraction solvent is in a range of 0.2 to 0.8 or in a range of 1.2 to 3.0".

Claim 1 of auxiliary request 3 has the features of claim 1 of the main request, and further defines the extraction solvents as follows.

"wherein the halogenated hydrocarbon is at least one selected from the group consisting of 1,1-dichloro-3,3,3-trifluoro propane, 1,1,3-trichloro-3,3-difluoro propane, 1,1,2-trichloro-3,3,3-trifluoro propane, 1,1,1,3,3-pentachloro propane, and carbon tetrachloride;

wherein the halogenated unsaturated hydrocarbon is at least one selected from the group consisting of 1,2,3-trichloro-3,3-difluoro propene, 1,2,3,3-tetrachloro-3-fluoro propene, 1,3-dichloro-3,3-difluoro propene, 2,3-dichloro-3,3-difluoro propene, 1,3,3-trichloro-3-fluoro propene, and 2,3,3-trichloro-3-fluoro propene;

wherein the nitrile is at least one selected from the group consisting of acetonitrile, propionitrile, and

butyronitrile;

wherein the ketone is at least one selected from the group consisting of acetone, methyl ethyl ketone, methyl isobutyl ketone, diethyl ketone, acetylacetone, and cyclohexanone;

wherein the carbonate is at least one selected from the group consisting of dimethyl carbonate, diethyl carbonate, dipropyl carbonate, and dibutyl carbonate;

wherein the alcohol is at least one selected from the group consisting of methanol, ethanol, isopropanol, butanol, and 1-(trifluoromethyl)-2,2,2-trifluoro ethanol;

wherein the hydrocarbon is at least one selected from the group consisting of n-pentane, i-pentane, and n-hexane;

wherein the ester is at least one selected from the group consisting of methyl acetate, ethyl acetate, propyl acetate, butyl acetate, trimethyl orthoformate, dimethyl sulfate, and γ -butyrolactone; and

wherein the ether is at least one selected from the group consisting of dipropyl ether, dibutyl ether, tetrahydrofuran, tetrahydropyran, 1,3-dioxolan, and 1,4-dioxane."

VII. The appellant's arguments were as follows.

Claim 1 of the main request related to a separation by extractive distillation with one or more of the solvents defined in claim 1. However, not every solvent was suitable for the claimed method, and establishing

which ones were represented an undue burden to a skilled person. For that reason, the claimed invention was not sufficiently disclosed for it to be carried out by a person skilled in the art. The issue also applied to the invention as claimed in auxiliary requests 2 and 3.

Both D9 and D10 were suitable starting points for examining inventive step in the method in claim 1 of auxiliary request 3. In view of either D9 or D10, the problem underlying the claimed invention was to provide an alternative; and the claimed solution, characterised by an extractive distillation with the aid of defined solvents, was taught by D11 and D12 and would have been obvious to a skilled person. The claimed method was thus not inventive.

VIII. The respondent's arguments were as follows.

The claimed invention was sufficiently disclosed. There was no evidence that any of the solvents in claim 1 would not separate HFC-245cb and (E)-HFO-1234ze by extractive distillation, but even if some would not the patent still led a skilled person towards suitable ones.

Claim 1 of auxiliary request 3 included a limited number of solvents. Even if any of these were not suitable, for which there was no evidence, a skilled person would have no difficulty in finding those which would work, out of the forty-five chemicals in claim 1.

Only D10 was a suitable starting point for examining the method in claim 1 of auxiliary request 3, as D9 did not relate to the (E) isomer of HFO-1234ze. The problem underlying the claimed invention was to provide a

method for separating HFC-245cb and (E)-HFO-1234ze; and the claimed solution, characterised by an extractive distillation with the aid of the solvents defined in claim 1, would not have been obvious to a skilled person and was thus inventive.

IX. Oral proceedings before the board of appeal took place on 9 May 2025.

X. The parties' final requests were as follows:

The appellant requested that the decision under appeal be set aside and the patent be revoked.

The respondent requested that the appeal be dismissed, or that the patent be maintained with the claims of auxiliary requests 2 to 5 filed with the reply to the grounds of appeal.

XI. At the end of the oral proceedings, the decision was announced.

Reasons for the Decision

1. The appeal is admissible.

Sufficiency of disclosure

2. The opposition division concluded that the claimed invention was sufficiently disclosed for it to be carried out by a skilled person. The appellant contested this conclusion.

3. Claim 1 of the main request relates to a method for separating HFC-245cb and (E)-HFO-1234ze by extractive distillation in the presence of a solvent selected from

halogenated hydrocarbons, halogenated unsaturated hydrocarbons, nitriles, ketones, carbonates, alcohols, hydrocarbons, esters and ethers. Only halogenated hydrocarbons, hydrogenated unsaturated hydrocarbons and alcohols are restricted to specific chemicals in claim 1.

It is undisputed that the boiling points of HFC-245cb and (E)-HFO-1234ze differ by less than one degree Celsius.

The patent contains six examples which measure the relative volatility of HFC-245cb and (E)-HFO-1234ze in the presence of acetone, acetonitrile, dimethyl carbonate, ethanol, carbon tetrachloride and n-hexane respectively.

4. The respondent argued that the claimed method was sufficiently disclosed. There was no evidence that any solvent in claim 1 could not change the relative volatility of the compounds to be separated. Moreover, even if there were solvents in claim 1 which would not modify the relative volatility of HFC-245cb and (E)-HFO-1234ze, the patent disclosed suitable candidates in paragraphs [0033] to [0041]. Whether a solvent could be used in the claimed method could be checked as indicated in paragraphs [0025] to [0029] of the patent. A skilled person would thus find no difficulty in putting the claimed invention into practice.
5. However, the board concludes that the claimed invention is not sufficiently disclosed.

According to paragraph [0011] of the patent, "[i]n the industrial use of extractive distillation, the selection of the extraction solvent is most important."

Paragraph [0014] further discloses that the extractive distillation in claim 1 requires "a specific extraction solvent". By the patent's own disclosure, the solvent needs to be carefully chosen. It is thus not credible that every solvent in claim 1, which includes large classes of solvents such as hydrocarbons or esters, can be suitable for separating HFC-245cb and (E)-HFO-1234ze.

A skilled person could only identify the suitable solvents by trial and error, and this represents an undue burden in view of the large number of compounds covered by each of the general classes in claim 1.

The method in claim 1 of the main request is thus not sufficiently disclosed for it to be carried out, contrary to the requirements in Article 83 EPC. The main request is not allowable.

6. Claim 1 of auxiliary request 2 includes all the solvents in claim 1 of the main request and further requires them to bring the relative volatility of HFC-245cb and (E)-HFO-1234ze within set limits. The respondent did not dispute that the conclusion regarding sufficiency should be no different from that for the main request.
7. Claim 1 of auxiliary request 3 limits the solvents for the extractive distillation to forty-five chemicals. Six of them are tested in the examples of the patent.

There is no experimental evidence on file that any of the solvents in claim 1 would not be suitable for the claimed method. The solvents in claim 1 are those described as most preferred in paragraphs [0033] to [0041] of the patent, and are not very dissimilar from

those tested in the examples. For these reasons alone, the claimed invention is sufficiently disclosed.

In addition, even if not every solvent in claim 1 were suitable for the claimed method, as argued by the appellant, the number of candidates is limited and whether they modify the relative volatility of HFC-245cb and (E)-HFO-1234ze can be easily determined in the manner disclosed in the patent (see examples). Testing a limited number of compounds would not represent an undue burden for a skilled person.

The board thus concludes that the method in claim 1 of auxiliary request 3 is sufficiently disclosed for it to be carried out by a skilled person.

Inventive step

8. Claim 1 relates to a method for separating HFC-245cb and (E)-HFO-1234ze by extractive distillation with the aid of defined solvents.
9. Closest prior art
 - 9.1 The appellant regarded both D9 and D10 as suitable starting points, as concluded by the opposition division. The respondent considered only D10 to be suitable.
 - 9.2 D9 does not disclose the (E) isomer of HFO-1234ze, but HFO-1234ze in general. (Z)-HFO-1234ze has a boiling point sufficiently different from that of HFC-245cb to allow separation even by normal distillation.

In addition, HFC-245cb and HFO-1234ze are recovered from the top of the distillation column (page 12, lines

23 to 25) as azeotropic mixtures with HF. It is thus not apparent whether the process in D9 separates HFC-245cb and (E)-HFO-1234ze from each other, as specified in claim 1.

- 9.3 Document D10 discloses the purification of hydrofluoropropanes [0002] such as HFC-245cb [0017] from olefin impurities such as (E)-HFO-1234ze [0025] by reacting the latter with hydrogen and hydrogen fluoride (see claim 1).

Thus, D10 does not disclose the separation of HFC-245cb and (E)-HFO-1234ze, as required by claim 1, as the latter is consumed in the process.

10. Problem underlying the claimed invention

The appellant argued that, starting from either D9 or D10, the problem should be formulated as the provision of an alternative method for separating HFC-245cb and (E)-HFO-1234ze.

However, as explained above, neither D9 nor D10 discloses the separation of HFC-245cb and (E)-HFO-1234ze. The problem underlying the claimed invention is thus to provide a process for separating HFC-245cb and (E)-HFO-1234ze.

11. Solution

The claimed solution is the method in claim 1, characterised by an extractive distillation in the presence of specified solvents.

12. Success

Claim 1 provides the separation of HFC-245cb and (E)-HFO-1234ze. The problem is thus solved by the method in claim 1.

13. Obviousness of the claimed solution

The appellant argued that, like the claimed invention, D12 related to the separation of halogenated compounds with close boiling points and disclosed that extractive distillation using the solvents in claim 1 (Table 1 of D12) was suitable. A similar disclosure was to be found in D11. A skilled person would thus have consulted one of these documents and attempted extractive distillation with the solvents in claim 1 with a reasonable expectation of success.

However, even if a skilled person had consulted D11 or D12, they would not have considered the solvents in either to be certainly suitable for the separation in claim 1. By the appellant's own argument in the context of sufficiency of disclosure, which the board found convincing, not every imaginable solvent is suitable for separating the compounds in claim 1. Conversely, however, a skilled person could not have predicted which solvents could be suitable for the method in claim 1. Nor would a skilled person have expected solvents suitable for separating other compounds to be successful in the separation of HFC-245cb and (E)-HFO-1234ze as well.

14. The method in claim 1 of auxiliary request 3 is thus inventive (Article 56 EPC).

15. No other objections to auxiliary request 3 have been raised during the appeal, and none is apparent to the board. Auxiliary request 3 is thus allowable.

Order

For these reasons it is decided that:

The decision under appeal is set aside.

The case is remitted to the opposition division with the order to maintain the patent with the claims filed as auxiliary request 3 dated 4 August 2022, and a description to be adapted if necessary.

The Registrar:

The Chair:



C. Rodríguez Rodríguez

P. Gryczka

Decision electronically authenticated