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**Datasheet for the decision  
of 15 December 2021**

**Case Number:** T 1991/18 - 3.3.03

**Application Number:** 08787044.0

**Publication Number:** 2178946

**IPC:** C08G65/40

**Language of the proceedings:** EN

**Title of invention:**

IMPROVED POLY(ARYLEETHERKETONE)S AND PROCESS FOR MAKING THEM

**Patent Proprietor:**

Solvay Specialty Polymers USA, LLC.

**Opponent:**

Evonik Operations GmbH

**Relevant legal provisions:**

EPC Art. 56

**Keyword:**

Inventive step - obvious alternative



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Case Number: T 1991/18 - 3.3.03

**D E C I S I O N**  
**of Technical Board of Appeal 3.3.03**  
**of 15 December 2021**

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**Decision under appeal:** Interlocutory decision of the Opposition  
Division of the European Patent Office posted on  
21 June 2018 concerning maintenance of the  
European Patent No. 2178946 in amended form.

**Composition of the Board:**

**Chairman**            D. Semino  
**Members:**            M. Barrère  
                              A. Bacchin

## Summary of Facts and Submissions

I. The appeals of the patent proprietor and opponent 2 lie from the interlocutory decision of the opposition division posted on 21 June 2018 and concerning maintenance of European Patent number 2 178 946 in amended form on the basis of the claims of auxiliary request 1 filed during the oral proceedings on 9 May 2018 and an adapted description.

II. Claim 1 as granted read as follows:

"Process for the manufacture of a poly(aryletherketone) (P) comprising the steps of :

a. forming a reaction medium by adding to a reactor :

i. at least one  $\alpha,\omega$  difluorinated monomer (M1) and at least one  $\alpha,\omega$  dihydroxylated monomer (M2), and/or

ii. at least one  $\alpha$  monofluorinated,  $\omega$  monohydroxylated monomer (M3); then

b. adding to the reactor at least one salt (S1) of an alkali metal (A) other than lithium ; then

c. making the monomers and the salt (S1) react together, until the obtention of:

- a poly(aryletherketone) (P\*) comprising recurring units derived from the monomers and having an intrinsic viscosity  $\eta_{int}^*$  superior to

0.7, as measured in methane sulfonic at 30°C according to ASTM D2857, and

- as a by-product, a fluoride salt (S2) of the alkali metal (A), said salt (S2) having a solubility of more than  $5 \cdot 10^{-7}$  mol/kg in acetone at 25°C,

wherein, at the completion of step c, the ratio

$$[n_{M1} + (n_{M3}/2)] / [n_{M2} + (n_{M3}/2)]$$

is from 0.97 to 1.03,

where  $n_{M1}$ ,  $n_{M2}$  and  $n_{M3}$  are respectively the total number of moles of (M1), (M2) and (M3) which have been added to the reactor until the completion of step c ; then

d. forming the poly(aryletherketone) (P) from the poly(aryletherketone) (P\*), said poly(aryletherketone) (P) having an intrinsic viscosity  $\eta_{int}$  comprised between  $0.8 \cdot \eta_{int}^*$  and  $1.2 \cdot \eta_{int}^*$ , by adding successively to the reactor:

1. at least one  $\alpha, \omega$  difluorinated compound (M1'), different from or identical to (M1); then
2. at least one salt (S3) able to react with the fluoride salt (S2) to form a fluoride salt (S4), said salt (S3) having a solubility of more than  $5 \cdot 10^{-7}$  mol/kg in acetone at 25°C and said fluoride salt (S4) having a solubility of at most  $5 \cdot 10^{-7}$  mol/kg in acetone at 25°C ; then

3. at least one  $\alpha,\omega$  difluorinated compound (M1"), different from or identical to (M1) and (M1');

wherein, at the beginning of the addition of the salt (S3), the ratio

$$[n_{M1} + n_{M1'} + (n_{M3}/2)] / [n_{M2} + (n_{M3}/2)]$$

is above 1.03,

where  $n_{M1'}$  is the total number of moles of (M1') which has been added to the reactor after the completion of step c and until the beginning of the addition of the salt (S3) and

wherein, at the completion of step d, the ratio

$$[n_{M1} + n_{M1'} + n_{M1''} + (n_{M3}/2)] / [n_{M2} + (n_{M3}/2)]$$

is above 1.08,

where  $n_{M1''}$  is the total number of moles of (M1'') which has been added to the reactor after the beginning of the addition of the salt (S3) and until the completion of step d ; then

e. recovering the poly(aryletherketone) (P) from the reaction medium."

The granted set of claims included in addition dependent process claims 2 to 9 and product claims 10 to 14.

Auxiliary request 1 included only granted process claims 1 to 9.

For the sake of clarity, the first, second and third ratios of claim 1 are referred to as Q, Q' and Q" respectively in the following.

III. The following documents were *inter alia* cited in the opposition division's decision:

E10: EP 0 211 693 A1

E14: US 4,820,790

E21: Letter of the patentee to the examining division dated 12 August 2013

E28: Declaration by Chantal Louis and additional experimental evidence dated 6 March 2018

IV. In that decision the opposition division held, among others, that:

- The subject-matter of granted claim 1 was novel over the disclosure of E10.
- The subject-matter of granted claims 10 and 11 was not novel over E14.
- E10 was the closest prior art for the subject-matter of claim 1 of auxiliary request 1 (which was identical to claim 1 as granted). Claim 1 differed from example 9 of E10 in at least two respects. The first difference was that the amount of the first portion of difluoro end-capping agent was sufficient to obtain a molar ratio Q' greater than 1.03 and that the salt S3 was added once the ratio was above this value. The second difference was the addition of a second portion of difluoro end-capping agent such that the molar ratio Q" was greater than 1.08. The objective problem to be solved was the provision of a process for the

production of a Poly(Aryl Ether Ketone) (PAEK) with a high fluoride end-capping, high molecular weight and good whiteness. No documents on file suggested the stepwise addition of difluoro end-capping agent and salt S3 with the specified molar ratios. Therefore the subject-matter of claim 1 of auxiliary request 1 involved an inventive step over E10 as the closest prior art.

V. With the statement setting out the grounds of appeal the patent proprietor (appellant 1) requested that the contested decision be set aside and that the patent be maintained as granted. In the alternative, maintenance of the patent in amended form on the basis of one of auxiliary requests 1-6 filed with that statement of grounds of appeal was requested.

Claim 1 of each of auxiliary requests 1-6 was identical to claim 1 as granted.

VI. With the statement setting out the grounds of appeal opponent 2 (appellant 2) requested that the contested decision be set aside and that the patent be revoked.

VII. With letter of 5 November 2021 appellant 1 further requested (as the lowest ranking request) that the patent be maintained on the basis of the claims found allowable by the opposition division (i.e. the dismissal of appellant 2's appeal).

VIII. With the explicit agreement of both parties, oral proceedings were held before the Board on 15 December 2021 by video conference.

IX. Appellant 1's arguments, insofar as relevant to the decision, may be summarised as follows:



**(a) Patent as granted**

**(i) Inventive step**

E10 was the closest prior art for the subject-matter of claim 1. Claim 1 differed from example 9 of E10 in that:

- 1) in step d., the difluorinated monomer was added in two steps (instead of one in example 9) with the addition of the salt S3 in-between and
- 2) the molar ratios  $Q'$  and  $Q''$  were at least 1.03 and 1.08 respectively (instead of 1.02 for the final molar ratio in example 9).

At the oral proceedings before the Board it was also contested that the condition of claim 1 concerning the viscosity of  $P^*$  was met.

It was credible, based on the experimental evidence provided in the opposed patent and in E28, that the above distinguishing features led to improved colouring resistance of the PAEK. None of the cited documents suggested to add the difluorinated monomer in two steps and to increase the ratios  $Q'$  and  $Q''$  in order to solve this problem. Although example 4 of E10 disclosed the two-step addition of a difluorinated monomer, it was not related to an improved colouring resistance. In fact, the general description of E10 taught away from using an excess of end-capping agent. Besides the PAEK end groups of E10 were not necessary fluorinated. The solution to the above problem was therefore not obvious starting from example 9 of E10 as starting point.

The same conclusion was obtained starting from example 4 of E10 which could be chosen as an alternative starting point.

Therefore the subject-matter of claim 1 involved an inventive step over E10 as the closest prior art.

X. Appellant 2's arguments, insofar as relevant to the decision, may be summarised as follows:

**(a) Patent as granted**

**(i) Inventive step**

E10 was the closest prior art for the subject-matter of claim 1. Claim 1 differed from example 9 of E10 in that:

1) in step d., the molar ratios  $Q'$  and  $Q''$  were at least 1.03 and 1.08 respectively (instead of 1,02 and 1.04 respectively in example 9).

The comparative examples on file were not suitable to show any improvement over example 9 of E10. The objective problem to be solved was therefore the provision of an alternative process. E10 taught that an excess of end-capping agent might be used. It was therefore obvious for the skilled person to increase the amount of fluorinated monomer as end-capping agent and thereby to obtain molar ratios as set out in claim 1.

Furthermore, if it were to be considered that the difluorinated end-capping agent was added in a single step in example 9 of E10, it would also be obvious for

the skilled person to add said agent in two steps as taught in example 4 of E10.

Finally the same arguments applied *mutatis mutandis* starting from example 4 of E10 as springboard to the subject-matter of claim 1.

For these reasons the subject-matter of claim 1 did not involve an inventive step over E10 as the closest prior art.

- XI. Appellant 1 requested that the decision under appeal be set aside and that the patent be maintained as granted, or in the alternative that the patent be maintained in amended form on the basis of one of auxiliary requests 1 to 6 filed with the statement of grounds of appeal. As a further alternative, appellant 1 requested that the appeal of appellant 2 be dismissed.
  
- XII. Appellant 2 requested that the decision under appeal be set aside and that the patent be revoked.

### **Reasons for the Decision**

- 1. Main request (patent as granted)
  - 1.1 Inventive step
    - 1.1.1 Closest prior art

The parties agreed that E10 constituted the closest prior art for claim 1 of the main request.

The Board sees no reason to deviate from that view.

### 1.1.2 Technical differences

It was not disputed between the parties that example 9 of E10 is an appropriate starting point within that document for the subject matter of claim 1 as granted.

The Board does not see any reason to depart from that view.

However, there was disagreement in view of the number and nature of distinguishing features between claim 1 and example 9 of E10. In particular, appellant 2 considered that example 9 disclosed a twofold addition of difluorinated monomer as end-capping agent.

The Board does not agree with appellant 2 on that point.

According to example 9 of E10, the polymerisation was conducted as in comparative example I and, after the reaction mass had become viscous at 320°C, magnesium chloride (corresponding to a salt S3) and 1,4-bis(4-fluorobenzoyl) benzene (corresponding to an  $\alpha,\omega$  difluorinated compound as end-capping agent) were added (see E10, column 28, lines 13-20). It is clear for the Board that the reference to a reaction temperature of 320°C implies that the process of comparative example I was followed until a temperature of 320°C was reached (see E10, column 27, lines 40) and then continued following the instructions of example 9. This reading of example 9 is also consistent with the purpose of this example, which is to show the effect of the invention as set out in claim 1 of E10 (i.e. effect of the addition of a salt S3; see E10, column 28, 31-35). In comparative example I, the end-capping agent is added without salt S3, while in example 9 the same

amount of end-capping agent is added with a salt S3. Therefore, contrary to appellant 2's view the Board considers that example 9 includes the steps of comparative example I until line 41 of column 27 and goes on with the steps described in column 28, lines 15-22.

Therefore example 9 of E10 discloses a process for the manufacture of a PAEK comprising the steps of :

- a. forming a reaction medium by adding to a reactor :
  - i. 4,4'-difluorobenzophenone corresponding to an  $\alpha,\omega$ -difluorinated monomer (M1) and hydroquinone corresponding to an  $\alpha,\omega$ -dihydroxylated monomer (M2) (see E10, column 27, lines 30-34); then
- b. adding to the reactor sodium carbonate corresponding to a salt (S1) according to claim 1 (see E10, column 27, lines 34-35); then
- c. making the monomers and the salt (S1) react together, until the obtention of:

- a viscous intermediate PAEK (see, E10, column 28, lines 15-16),

- as a by-product, sodium fluoride corresponding to a salt (S2) according to claim 1 (see opposed patent, paragraph [0054]),

wherein, at the completion of step c, the calculated molar ratio Q is 1,00 (see E10, column 27, lines 33-34); then

d. forming the final PAEK from said intermediate PAEK by adding to the reactor:

1,4-bis(4-fluorobenzoyl) benzene corresponding to  $\alpha,\omega$ -difluorinated compound (M1') according to claim 1 and

magnesium chloride correspond to a salt (S3) according to claim 1 wherein at the completion of step d

the calculated molar ratio  $Q''$  is 1.02;

then

e. recovering the final PAEK from the reaction medium.

While during the whole of the appeal proceedings it was never disputed that the conditions concerning the viscosities in claim 1 ( $P^*$  having an intrinsic viscosity  $\eta_{int}^*$  superior to 0,7 and P having an intrinsic viscosity comprised between  $0.8 \eta_{int}^*$  and  $1.2 \eta_{int}^*$ ) are implicitly met by the process of example 9, appellant 1 towards the end of the discussion at the oral proceedings contested that the first condition was met, albeit without providing any explanation for that. The Board considering the disclosure in E10 (in particular in view of the high reaction temperature of  $320^\circ\text{C}$ , the 1:1 molar ratio between the difluorinated monomer and the diol monomer and the information on the reduced viscosities in column 28, lines 25-27), the agreement of the parties till the latest stage of the proceedings and the lack of a explanation to the contrary for the disputed first condition, has no reason to consider that the first condition is not met,

which would be to the detriment of appellant 2 at a point in time in which they could not provide any evidence for that. As to the second condition, it is considered to be met with the agreement of the parties.

Therefore, claim 1 differs from example 9 in that :

i) in step d., the difluorinated monomer was added in two steps (instead of one in example 9) with the addition of the salt S3 in-between and

ii) the molar ratios  $Q'$  and  $Q''$  were at least 1.03 and 1.08 respectively (instead of 1.02 for the final molar ratio in example 9).

#### 1.1.3 Objective technical problem

According to appellant 1 comparative examples were provided which showed that a PAEK obtained by the process of claim 1 was characterised by an improved resistance to colouring compared to the process of example 9 of E10.

The Board does not agree with this interpretation of the comparative examples for the following reasons:

It should be noted that the examples 1-3 as well as the comparative examples 1 and 2 of the opposed patent are not comparable. This is mainly because the reaction time and temperatures are different for each example. For instance, while the polymerisation is carried out at 320°C in examples 1-3 of the opposed patent, the polymerisation temperature is reduced to 200-310°C in comparative examples 1-2 (see opposed patent, paragraphs [0122] and [0153]).

Thus, none of the comparative examples of the patent is suitable to provide a direct comparison with the disclosure of E10 and hence to show an effect specifically associated with the distinguishing features.

Likewise the comparative examples submitted by appellant 1 in E21 and E28 do not allow a proper comparison with example 9 of E10. While E21 relates to example 4 of E9 (instead of example 9), E28 discloses a modified version of example 9 wherein the molar excess of difluorinated end-capping agent is 30% instead of 2%.

In the absence of suitable comparative examples, the Board concludes that no effect has been plausibly shown in comparison with Example 9 of E10.

Consequently the objective problem to be solved over E10 is to provide an alternative process.

#### 1.1.4 Obviousness

Appellant 1 takes the view that E10 does not teach or instruct the skilled person to increase the molar ratios  $Q'$  and/or  $Q''$ . Instead E10 would discourage to use large amounts of end-capping agent (see E10, column 4, lines 20-22). Thus there would be no reason for the skilled person to add the difluorinated end capping agent in two steps (distinguishing feature i)) and at the same time increase the molar excess of said agent (distinguishing feature ii)).

The Board cannot follow this line of arguments. With regard to the distinguishing features i) and ii), it is emphasised that no technical effect can be associated



with it. It may therefore be assessed whether each of the distinguishing features is obvious in view of E10.

As to the addition of the difluorinated end-capping agent in two steps with the addition of a salt S3 in-between, it is pointed out that this procedure is known from example 4 of E10 (see column 22, line 48 to column 23, line 5). Distinguishing feature i) is therefore obvious in view of the teaching of E10 when simply aiming at providing an alternative process.

With regard to the increase of the molar excess of difluorinated end-capping agent (distinguishing feature ii)) the following is noted. While it is true that E10 teaches that an excess of end-capping agent may lead to a decrease of the molecular weight (see E10, column 4, lines 20-22), this document also provides a guidance to select suitable amounts of end-capping agent. In particular E10 indicates that the amount is at least a stoichiometric equivalent or an excess based on the number of phenate end group present and that effective amounts of end-capping agent may be chosen from 0.001 to 0.5 moles per mole of the charged phenol groups (see E10, column 8, lines 28-38). In example 9 the effective amount of difluorinated end-capping agent is 0.02 mole per mole of hydroquinone as phenolic monomer. An increase to 0.5 mole of end-capping agent (as suggested in column 8, lines 32-38 of E10) would however lead to a calculated ratio  $Q''$  of 1,5 which is well above the minimum value of 1.08 as set out in granted claim 1. It is therefore obvious in view of the teaching of E10 to increase the amount of difluorinated end-capping agent such as to obtain a molar ratio  $Q'$  of at least 1.03 and a final molar ratio  $Q''$  of at least 1.08 when simply aiming at providing an alternative process.

For these reasons it is obvious for the skilled person wishing to provide an alternative to the method of example 9 of E10:

to add the end-capping agent in two equal steps with the addition of the salt S3 in-between as suggested by example 4 of E10 and

to increase the amount of end-capping agent in each of the said steps so as to obtain a first molar ratio Q' of at least 1,03 and a second molar ratio Q" of at least 1,08 (as taught in column 8, lines 28-38 of E10).

The subject-matter of granted claim 1 therefore lacks an inventive step over E10.

2. The same conclusion applies to claim 1 of auxiliary requests 1-6 because said claims are identical to granted claim 1. For the same reason, in view of appellant 1's request to dismiss the appeal of appellant 2, this conclusion equally applies to claim 1 as maintained by the opposition division. Hence, these requests do not meet the requirement of Article 56 EPC, either.
3. In the absence of any valid request which conforms with the requirements of Article 56 EPC, the patent cannot be maintained.
4. There is therefore no need to deal with any further issue.

## **Order**

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

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated