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**D E C I S I O N**  
of 14 September 1998

**Case Number:** T 0185/96 - 3.3.1

**Application Number:** 91200346.4

**Publication Number:** 0445859

**IPC:** C07C 69/54

**Language of the proceedings:** EN

**Title of invention:**

Process for obtaining sec-butyl acrylate

**Patentee:**

Compania Espanola de Petroleos S.A. -Cepsa

**Opponent:**

BASF Aktiengesellschaft

**Headword:**

Esterification/CEPSA

**Relevant legal provisions:**

EPC Art. 56, 123(2), (3)

EPC R. 57, 88

**Keyword:**

"Inventive step (no) - alleged unexpected effect not shown"

**Decisions cited:**

T 0123/85, T 0197/86, T 0752/93, T 0815/94

**Catchword:**

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Chambres de recours

Case Number: T 0185/96 - 3.3.1

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.1  
of 14 September 1998

**Appellant:** Compañia Espanola de Petroleos  
(Proprietor of the patent) S.A. -CEPSA  
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**Representative:** Ungría López, Javier et al  
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**Respondent:** BASF Aktiengesellschaft  
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**Decision under appeal:** Decision of the Opposition Division of the  
European Patent Office posted 20 December 1995  
revoking European patent No. 0 445 859 pursuant  
to Article 102(1) EPC.

**Composition of the Board:**

**Chairman:** P. Krasa  
**Members:** P. P. Bracke  
R. E. Teschemacher

## Summary of Facts and Submissions

I. This appeal is from the Opposition Division's decision revoking European patent No. 0 445 859, because the claimed process was considered not to be inventive vis-à-vis the teaching of document

(4) US-A-3 037 052,

submitted in the notice of opposition, which was based solely on lack of inventive step.

The decision was based on Claim 1 submitted during oral proceedings on 8 November 1995 and Claims 2 and 3 as granted. The only independent claim read:

"1. A process for obtaining sec-butyl acrylate, which comprises reacting, continuously or discontinuously, acrylic acid and 1-butene in the presence of an ionic exchange styrene divinylbenzene resin which has sulfonic acid groups as a catalyst, characterized in that

(a) said resin is macroporous and has a BET surface area between 20 and 100 m<sup>2</sup>/g, a maximum pore diameter of 1000 Å, a porosity between 20 and 80% and an ionic exchange capacity between 0.5 and 3 eq/l;

whereby the acrylic acid and the 1-butene are reacted

(b) at a temperature between 50 and 120°C (preferably between 80 and 110°C);

(c) at a pressure between 10 and 30 kg/cm<sup>2</sup>;

(d) for a reaction time between 0.5 and 4 h; and

(e) by using a molar ratio 1-butene/acrylic acid between 0.3 and 10 (preferably between 1 and 5)."

II. The Opposition Division considered that the claimed process differed from the one described in document (4), column 16, lines 12 to 34, only by the reaction time and by the nature of the macroporous ionic exchange resin but that these distinguishing features fell within the generic disclosure of document (4). Since an unexpected effect for the claimed process had not been shown by the Appellant (patent proprietor) the Opposition Division came to the conclusion that the selection of the resin and the reaction time and pressure was arbitrary and, consequently, that the claimed process was derivable from document (4) in an obvious manner.

III. The Appellant submitted with the letter dated 30 April 1996 (Grounds of Appeal) a new Claim 1 reading:

"A process for obtaining sec-butyl acrylate, which comprises reacting, continuously or discontinuously, acrylic acid and 1-butene in the presence of a macroporous ionic exchange resin having sulfonic acid groups, a maximum pore diameter of 1000 Å, and an ionic exchange capacity between 0.5 and 3 eq/l, as a catalyst, whereby the process is carried out

- at a temperature between 50 and 120°C (preferably between 80 and 110°C),
- and by using a molar ratio 1-butene/acrylic acid between 0.3 and 10 (preferably between 1 and 5)

characterized in that

- said resin has a BET surface area between 20 and 100 m<sup>2</sup>/g and a porosity between 20 and 80%

whereby the acrylic acid and the 1-butene are reacted

- at a pressure between 10 and 30 kg/ cm<sup>2</sup>,
- for a reaction time between 0.5 and 4 h."

As supporting evidence that the ionic exchange capacity range of the used resins should correctly be expressed in "eq/l" instead of "m eq/l" as written in the application as originally filed, the Appellant referred to document

- (7) Ion Exchange Catalysis and Matrix Effects, a publication of the Rohm & Haas Co, and

to document

- (Pat-1) Polymeric Reagents and Catalysts, ACS Symposium Series 308, American Chemical Society, Washington, by Warren T., pages 42 to 45, 1986.

Furthermore, he argued that the reaction time and pressure and the macroporous ionic exchange resin used in the claimed process were not suggested in document (4). As announced during the written procedure, he provided during the oral proceedings, held on 14 September 1998, an affidavit by Prof. Dr José Aguado Alonso and Prof. Dr Dolores Romero Diaz, which contained experimental data to show that the desired effect, i.e. an unexpectedly high conversion rate of the acrylic acid, was only obtained by selecting the reaction time and pressure and the resin according to the claimed process.

- IV. The Respondent (opponent) submitted that, by changing the parameter "m eq/l" to "eq/l" in Claim 1, subject-matter was added which extended beyond the content of

the application as filed and further that the appeal procedure was extended in an inadmissible way, since the new Claim 1 related to processes wherein any macroporous ionic exchange resin having sulfonic acid groups could be used, whereas the set of claims underlying the contested decision had been restricted to processes wherein only ionic exchange styrene divinylbenzene resins having sulfonic acid groups were used.

Moreover, he contested that the experimental data provided by the Appellant demonstrated that the desired effect was only obtained by selecting the reaction time and pressure and the resin according to the claimed process.

- V. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of Claim 1 as submitted with the letter dated 30 April 1996 and Claims 2 and 3 as granted.

The Respondent requested that the appeal be dismissed.

### **Reasons for the Decision**

1. The appeal is admissible.
2. *Formal requirements*
  - 2.1 The Respondent submitted that the modification in the new Claim 1 of "m eq/l" as contained in the application as originally filed to "eq/l" as the unit for the ionic exchange capacity of the resin could not be considered as the correction of an obvious error.

The relevant passage in the application as originally

filed reads:

"Said resin must be macroporous and have ... an ionic exchange capacity between 0.5 and 3 m eq/l. Commercially available products which combine the above mentioned characteristics and which may be used to carry out the reaction are Lewatit SPC118, Amberlist 15, Dowex MSC-1, Serdolit Red and others of similar nature" (page 5, lines 23 to 31 of the application as filed).

The Respondent agreed that from the discrepancy between the ionic exchange capacity range "between 0.5 and 3 m eq/l" mentioned in the application as filed and the ionic exchange capacities of the four commercially available products specified therein it was evident for a skilled person that an error had occurred. Nor did he contest that the ion exchange capacity of the commercially available products is of the magnitude of some few meq/g or meq/ml [see also Table 2 on the 8th page of document (7) for the Amberlyst range of products]. However, he submitted that it was not immediately evident that nothing else than an ionic exchange capacity between 0.5 and 3 eq/l had in fact been intended for the resins used in the claimed process since, for example, an error also could have occurred in the designation of the commercially available resins.

The Board cannot accept this argument for the following reason:

It is established beyond any reasonable doubt at least for one of the four designated commercially available ion exchangers that it complies with the requirement that it "may be used to carry out the reaction" since it is employed in the examples (i.e. Lewatit SPC118; see in particular examples 1 and 4). Therefore, the

skilled person would have immediately established the respective ion exchange capacity, which is an intrinsic property of these resins, and would have found that the unit "m eq/l" was a clear misprint for "eq/l". The Respondent's speculation that "m eq/l" was correct and that all the designations of commercially available ion exchange resins were wrong is, in the Board's judgement, far-fetched and not supported by the circumstances of the case. This finding is corroborated by the Respondent's admission not to be in the possession of any document providing evidence that any macroporous resin having an ionic exchange capacity of between 0.5 and 3 m eq/l was known to have a catalytic activity suitable for the claimed process.

For these reasons the Board concludes that the amendment is admissible under Rule 88 EPC and that no objections are to be raised against the amended Claim 1 under Article 123 EPC.

- 2.2 Contrary to the Respondent's opinion, the Appellant was not prevented from filing a main claim which is broader than the main claim forming the basis for the decision under appeal. Whereas during opposition proceedings the broadening of the claims as granted is excluded under Article 123(3) EPC, there is no corresponding principle restricting the Appellant to the claims submitted in the first instance. The limitation of a claim is not to be considered as an irrevocable surrender of a part of the patent (T 123/85, OJ EPO 1989, 336; see also T 752/93 and T 815/94, cited in EPO Board of Appeal Case Law 1996, special ed. of the OJ EPO 1997, page 119). Rather the proprietor is entitled to make amendments which are appropriate to remedy any deficiencies which may affect the validity of the patent. In the present case, the limitation made in the previous main claim in the proceedings before the



Opposition Division might have given rise to discussions on the basis of such limitation in the application as filed (cf Article 123(2) EPC). For this reason, the Board considered the removal of the limitation as appropriate under Rule 57 EPC.

3. *Novelty*

No comments by the Board are due in respect of novelty since this was not an issue in the proceedings.

4. *Inventive step*

4.1 The Opposition Division and the parties have agreed that document (4) represents the closest state of the art. The Board also accepts this citation as the starting point for evaluating inventive step.

Document (4) teaches that carboxylic acid esters may be prepared in high yields by reacting olefins with carboxylic acids in the presence of macro-reticular ionic exchange resins containing sulfonic acid groups (column 1, lines 14 to 19, and column 2, lines 19 to 27). This document further teaches that the molar ratio of olefin to carboxylic acid may be from 10:1 to 1:10 and, preferably, from 1.5:1 to 1:1.5 (column 6, lines 27 to 37); that the reaction temperature required for satisfactory conversion depends on the specific olefins and acids employed and, to some extent, on the reaction time (column 6, lines 38 to 42); that the reaction time is a variable value depending on the specific reactants employed and the temperature used, the upper limit being determined by the relative rates of the esterification reaction and the rates of

reaction of unwanted reactions (column 6, lines 47 to 57); and that the pressure employed is not critical, but preferably such as to maintain the reactants in liquid form (column 7, lines 34 to 36).

The reaction of acrylic acid with 1-butene is described in column 16, lines 12 to 24, as yielding - at a reaction temperature of 80°C and using a 1:1 molar ratio of reactants - 71% of secondary butyl acrylate after a contact time of 10 minutes and 72% after a contact time of 20 minutes. The yield is quantitative when based on consumed acrylic acid and is about 80% when based on consumed 1-butene.

- 4.2 According to the patent in suit, page 2, lines 49 to 55, the conditions disclosed in document (4) for preparing secondary butyl acrylate from acrylic acid and 1-butene "do not permit high degrees of conversion of acrylic acid to be reached which, due to its boiling point close to that of the corresponding acrylate, along with the fact of having to carry out distillation at a reduced pressure due to the parallel process of polymerization, gives rise to a complex purification scheme and recycling of the non-converted acid or to substantial losses of the reagent that make the process economically unfeasible".

The patent in suit also teaches that, when the claimed process is carried out under suitable operating conditions, it is possible to simultaneously obtain a high conversion of acrylic acid and a high selectivity of secondary butyl acrylate, thus allowing easy separation of the components of the reaction mixture (page 3, lines 12 to 14 and 50 to 53), and that such

suitable operating conditions are provided by the claimed method, more especially by using a macroporous ionic exchange resin according to Claim 1 and by conducting the reaction during 0.5 and 4 hours at a pressure between 10 and 30 kg/cm<sup>2</sup>.

- 4.3 Therefore, the first point to be considered in assessing inventive step is whether it has been convincingly shown that by selecting the operating conditions according to the present Claim 1 such a high conversion of acrylic acid and such a high selectivity of secondary butyl acrylate may be obtained that the components of the reaction mixture can be easily separated.

In an attempt to show that the above mentioned result is effectively obtained with the claimed process, the Appellant filed an affidavit signed by Prof. Dr José Aguado Alonso and by Prof. Dr Dolores Romero Diaz, wherein four tests for preparing secondary butyl acrylate from acrylic acid and 1-butene were described. According to test 4, the only one conducted using a process according to Claim 1, a conversion of acrylic acid of 70.5% and a selectivity for 1-butene of 85.8% is obtained if the reaction is conducted during 40 minutes at 80°C and at 20 kg/cm<sup>2</sup> in a molar ratio 1-butene/acrylic acid of 1/1 and using Lewatit SPC118.

Since the reaction conditions in test 4 differ from those of the reaction described in column 16, lines 12 to 24, of document (4) only by the nature of the macroporous ionic exchange resin bearing sulfonic groups and by the reaction pressure and time, i.e. by the distinguishing features of the claimed process, both reactions may be validly compared in order to show an alleged effect. This is in accordance with the jurisprudence of the Boards of Appeal of the EPO (see T 197/86 OJ EPO 1989, 371).

Since according to the reactions described in document (4) and test 4 the conversion of acrylic acid is 71% and 70.5% respectively, this comparison shows that the conversion of acrylic acid in both reactions is similarly high.

Since test 4 is silent on the selectivity of the reaction for the desired secondary butyl acrylate no further conclusions can be drawn in respect to the yield of this ester.

- 4.4 Consequently, in view of the teaching of document (4), the problem underlying the invention can only be seen in providing a further process of reacting acrylic acid with 1-butene wherein a similar conversion of acrylic acid is achieved.
- 4.5 Therefore, it remains to be decided whether a skilled person would have expected that by reacting acrylic acid with 1-butene according to the reaction features of Claim 1 a similar conversion of acrylic acid as disclosed in document (4) could be obtained.

The Appellant argued that it was taught in document (4), column 7, lines 34 to 43, that the pressure was not critical as long as the reactants were in liquid form and, consequently, that a skilled person would not have expected that the pressure exerts any influence on the degree of conversion. Moreover, since in column 16, lines 12 to 24, of document (4) it is only said that the increase of 10 minutes to 20 minutes reaction time produces virtually no change in the conversion of carboxylic acid, he considered there was no suggestion that the reagents be reacted for 0.5 to 4 hours.

However, the Board cannot follow this argumentation, because the teaching of document (4) is to be taken as a whole and from the general teaching of this document a person skilled in the art would have clearly deduced that the temperature, the pressure, the time of reaction and the molar ratio of the reactants may be selected within wide ranges (see 4.1 above) in order to have a high conversion of the starting carboxylic acid. Consequently, by selecting a temperature, a pressure, a time and a molar ratio within the ranges taught in document (4) a skilled person would have expected a similarly high conversion of acrylic acid.

- 4.6 The Appellant also argued that, in comparison with the process described in document (4), the claimed process combines a lower pressure with a longer reaction time. Since reactions at higher pressure were known to be economically disadvantageous, he concluded that the claimed process has the advantage of being more economical.

However, the economy of a chemical reaction is not only influenced by the applied pressure, but also, for example, by the reaction time, which in the present case is increased, exerting an unfavourable influence on the economy of the reaction. Therefore, in the absence of any evidence of an overall economical advantage, the Board cannot accept this argument either. Moreover, even if one would accept a beneficial economical effect of the claimed process, which in fact cannot be done for the reasons given, this would not help the Appellant's case since document (4) discloses also, as already stated above, that the pressure has no particular influence on the reaction concerned and that, thus, there was no need to apply high pressure.

5. Therefore, the Board comes to the conclusion that a skilled person, looking for a process for obtaining secondary butyl acrylate from acrylic acid and 1-butene with a similarly high conversion of acrylic acid as in the reaction described in document (4), would have selected the reaction parameters of Claim 1 with a reasonable expectation of success.
  
6. Consequently, the subject-matter of Claim 1 does not involve an inventive step and does not meet the requirement of Articles 52(1) and 56 EPC.

### Order

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

The appeal is dismissed.

The Registrar:



E. Gorgmaier

The Chairman:



P. Krasa