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Aktenzeichen / Case Number / N° du recours : T 294/85

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Bezeichnung der Erfindung: Method of regenerating phosphoric acid
Title of invention: extraction solvent
Titre de l'invention :

Klassifikation / Classification / Classement : C 01 B 25/46

ENTSCHEIDUNG / DECISION
vom / of / du 8 January 1987

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet : FMC Corporation (Respondent)

Einsprechender / Opponent / Opposant : Hoechst AG (Appellant)

Stichwort / Headword / Référence :

EPO/EPC/CBE Article 56

Kennwort / Keyword / Mot clé : "Inventive step" (confirmed)
"Lack of feasibility as grounds for opposition -
late submission"

Leitsatz / Headnote / Sommaire



Case Number : T 294 /85

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 8 January 1987

Appellant :
(Opponent)

Hoechst AG
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Representative :

Respondent :
(Proprietor of the patent)

FMC Corporation
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Representative :

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Decision under appeal :

Interlocutory decision of the Opposition
Division of the European Patent Office
dated 01.10.85 concerning maintenance of
European Patent No. 0 040 487 in amended
form.

Composition of the Board :

Chairman : K. Jahn
Member : R. Andrews
Member : C. Payraudeau

Summary of Facts and submissions

- I. The mention of the grant of the patent No. 0 040 487 in respect of European patent application No. 81 301 974.2, filed on 5 May 1981 and claiming priority of 19 May 1980 from a prior application in the United States of America, was announced on 14 September 1983 (cf. Bulletin 83/37).
- II. On 15 February 1984 the Appellants filed an opposition citing the following documents
- (1) US-A-3 410 656
 - (2) DE-B-2 127 141
 - (3) US-A-3 366 448
 - (4) GB-A-1 117 427
 - (5) GB-A-1 091 931 and
 - (6) DE-C-884 358
- and requesting the revocation of the patent on the ground of lack of inventive step.
- III. By an interlocutory decision dated 1 October 1985 the Opposition Division maintained the patent in amended form. Claim 1 (as corrected) read as follows:
- "The process of washing a substantially water immiscible solvent with an aqueous solution to separate impurities from the solvent, the solvent being selected from alkyl phosphates, aryl phosphates and alkyl aryl phosphates, and optionally also including an immiscible organic diluent, which solvent has been used to extract phosphoric acid from an impure phosphoric acid solution and from which the phosphoric acid has been stripped, characterized by conducting the washing with an aqueous solution containing sufficient alkali metal orthophosphate to maintain the pH

of the solvent-aqueous mixture at from 9.5 to 12.5, the solution containing at least 2 weight percent orthophosphate measured as P_2O_5 , and having a mole ratio of alkali metal to phosphorus of from 2.5:1 to 3.5:1."

- IV. The decision to maintain the patent in amended form was based on the argument that the documents (4) and (5) do not in fact disclose the purification of amines and quaternary ammonium salts used for extracting phosphoric acid by treating them with alkali metal hydroxides, alkali metal carbonates or alkali metal phosphates at a pH of between 11 and 12. Furthermore it was not obvious for a person skilled in the art to transfer any teaching relating to the purification of amines and quaternary ammonium salts used for extracting phosphoric acid to the purification of organic phosphates used for the same purpose. Documents (1), (2) and (3), which relate to process for the solvent extraction of phosphoric acid from impure aqueous solutions of phosphoric acid, are not specifically concerned with the purification of the solvent after the phosphoric acid has been stripped from it. Document (6) discloses that the solvent used in this extraction process may be purified by distillation.
- V. An appeal was lodged by the Opponent against this decision on 25 November 1985 together with the statement of grounds and payment of the appropriate fee. The Appellant argued that it would be obvious to apply the method for regenerating the amine extractants disclosed in documents (4) and (5) to the organic phosphate extractants known from documents (1), (2) and (3), particularly since it is known from documents (1) and (3) to purify stripped organic phosphate extractants by a alkali wash. The Appellant also referred to (7) 'Phosphorus and its Compounds' by von Wazer, Vol. 1 (1966), page 481 to demonstrate that a

solution of, for example, trisodium phosphate has a pH of about 12.

- VI. The Respondent contended that the Appellant has apparently misunderstood the teaching of documents (4) and (5). His arguments are therefore based on a misconception both of the prior art and the problem and inventive solution.
- VII. At the oral proceedings held on 8 January 1987, the Appellant argued that whether fatty acid esters are present as impurities in the wet acid depends upon the source of the phosphate rock used for its production, and in any case, an alkali metal orthophosphate solution having a mole ratio of alkali metal to phosphorus of 3.5:1 would saponify any fatty acid esters present to give rise to soapy materials in the same manner as the caustic soda used in the prior art process. Furthermore, it is common general knowledge to coagulate the dispersed phase of a colloidal suspension by adding polyvalent anions and, in order to avoid further contamination of the present mixture, it would be obvious to add orthophosphate anions in the form of a solution of alkali metal orthophosphate to cause coagulation of the dispersed phase. The Appellant also cast doubt on whether in fact the claimed process permits the easy separation of the solvent, aqueous phase and solid from each other in view of method of separation described in Example 1 of the disputed patent.

The Respondent replied that until the oral proceedings the stated advantages of the claimed process had never been disputed and that such allegations should not be raised at this stage of the proceedings. Furthermore, the formation of a high density solid mixture which settles in the aqueous phase is only achieved by a combination of pH and the content of alkali metal orthophosphate.

VIII. The Appellant requests that the decision under appeal be set aside and that the patent be revoked. The Respondent requests that the appeal be dismissed and that the patent be maintained in amended form.

Reasons for the Decision

1. The appeal complies with the requirements of Articles 106 to 108 and Rule 64 EPC and is, therefore, allowable.
2. There are no formal objections to the present statement of claim since it does not extend the protection conferred (Article 123(3) EPC). The amended Claim 1 represents a combination of original Claims 1 and 4. Amended Claims 2 to 6 and 8 to 13 correspond to original Claims 2, 3, 5 to 7 and 9 to 14 respectively. Amended Claim 7 corresponds to original Claim 8 with the deletion of the features of original Claim 4 now incorporated in the amended Claim 1.
3. The disputed patent relates to a process for the regeneration of organic phosphate solvents which have been used to extract phosphoric acid from impure phosphoric acid and from which the phosphoric acid has been stripped. The closest state of the art is represented by document (3) which discloses the regeneration of the same organic phosphate extractants by washing with caustic soda. A disadvantage of this prior art process lay in the fact that the separation of the solvent from the neutralised impurities was rendered difficult due to the formation of emulsions (cf. US-A-3 767 769, col. 2, lines 60 to 65, mentioned in the discussion of the prior art in the present patent specification on page 3, line 13) or soapy materials which caused the fine solid sodium silicates to be suspended in the solvent phase. This entails severe problems in the separation of the organic phase from the aqueous one.

4. In the light of the closest prior art the technical problem underlying the patent-in-suit has to be seen in providing an improved process for regenerating organic phosphate extractants which have been used to extract phosphoric acid from an impure phosphoric acid solution and from which the phosphoric acid has been stripped with regard to the ease with which the extractant, aqueous solution and solids may be separated from each other.

This problem is solved according to the disputed patent by washing the organic phosphate solvent with an aqueous solution containing sufficient alkali metal orthophosphate to maintain the pH of the solvent-aqueous mixture at from 9.5 to 12.5, the solution containing at least 2 weight per cent of orthophosphate measured as P_2O_5 and having a mole ratio of alkali metal to phosphorus of from 2.5:1 to 3.5:1. This causes the coprecipitation of some of the orthophosphate values with the solid alkali metal compounds formed by the neutralisation of the impurities in the solvent with the formation of a high density solid mixture which settles in the aqueous solution and permits an easy separation of the solvent, the aqueous solution and the solids from each other.

In view of the results obtained in the Examples of the disputed patent the Board is satisfied that this technical problem has been plausibly solved.

It was only during the oral proceedings that the Appellant alleged that the measures adopted did not solve the technical problem. However, proper proceedings before the EPO require that the facts, evidence and arguments known to the Opponent which support the grounds for opposition to a disputed patent are brought forward at the earliest possible date, i.e. in principle within the nine-month

opposition period. To the fundamental grounds against patentability belong, as here, the allegation that the effect claimed according to the patent does not occur and therefore that the technical problem underlying the patent is not solved or only solved for a part of the claimed area. The submission of such grounds almost three years after the filing of the opposition in the last stage of the opposition appeal procedure, the oral proceedings, must be in fact regarded as an abuse of the procedure.

Moreover, in this connection the Opponent and present Appellant has neither submitted proof nor an incontrovertible line of argument that would render it plausible that the effect claimed and substantiated by the Patentee does not in fact occur or only occurs to a limited extent.

Thus the allegation that an alkali metal orthophosphate solution with a mole ratio of alkali metal to phosphorus of 3.5:1 would be sufficiently alkaline to saponify any fatty acid esters coming from the acidulated phosphate rock with the formation of the same soapy materials as in the prior art process must be dismissed in the light of the present Examples and in the absence of any proof or compelling arguments from the Appellant. The further allegation that the technical problem underlying the disputed patent would only arise with specific types of phosphate rocks also cannot be accepted in the light of the above-mentioned passage in US-A-3 767 769 indicating a more general technical problem and in the absence of any proof from the Appellant. The Appellant's opinion that the conditions specified in the Examples of the disputed patent for the separation of the solvent, aqueous solution and solids from each other cannot be regarded as representing an easy

separation of these materials is unsubstantiated and cannot be considered by the Board. The Board therefore comes to the conclusion that the measures adopted in the patent-in-suit do in fact solve its underlying technical problem.

5. Having examined the documents on file the Board has reached the conclusion that, since a process having all the features of Claim 1 is not disclosed in any of the cited documents, the subject-matter of the patent-in-suit is novel. As the Appellant has not raised the issue of novelty it is not necessary to consider the matter in detail.

6. Documents (4) and (5) both relate to processes for the extraction of phosphoric acid from aqueous solutions containing iron as an impurity by treating the solutions with an extractant medium which is immiscible with the aqueous phosphoric acid and which comprises a water-insoluble amine or quaternary ammonium base or water-insoluble salts thereof (hereinafter amine). After having been stripped of the phosphoric acid the amine extractant containing complexed iron may be regenerated by washing with an aqueous basic solution. Numerous basic solutions are mentioned one of which is an alkali metal orthophosphate solution (cf. (4), page 7, lines 106 to 118 in combination with page 5, line 87 to page 6, line 56, particularly page 6, line 23; and (5) page 6, lines 6 to 18 in combination with page 4, line 117 to page 5, line 53, particularly page 5, line 21). The pH of the aqueous phase in equilibrium with the amine phase is preferably 4 to 9.5 in order to obtain the best result in regenerating the amine extractant and to keep the complexed iron in solution (cf. (4), page 7, lines 118 to 124; and (5) page 6, lines 19 to 24). After removing the amine extractant phase the pH of the aqueous phase is increased to 11 to 12 to precipitate the iron as iron hydroxide (cf. (4) page 7, line 127 to page 8, line 2; and (5) page 6, lines 27 to

34). Thus the pH of 11 to 12, which overlaps with the range claimed in the patent-in-suit, is only disclosed in connection with the aqueous phase after the removal of the amine extractant phase. Thus these two prior art documents relate to a different problem, viz. the separation of iron from impure phosphoric acid solutions, use different extractants and different pH's during the extractant regeneration step. There is therefore no incentive to the skilled person to transfer any teaching which he may derive from documents (4) and (5) to solve the completely different problem underlying the disputed patent and, as defined, arising from the use of caustic soda to regenerate the organic phosphate extractants as disclosed in document (3).

7. Although it is generally known that the disperse phase of colloidal suspensions may be coagulated by the addition of polyvalent ions (Tyndall Effect), in view of the different and far more complex nature of the present mixture comprising emulsions or three phase systems with a large interfacial layer a person skilled in the art would not be in the position to predict that the addition of aqueous alkali metal orthophosphate under the conditions of pH as specified in the present Claim 1 would result in the formation of a high density solid mixture and thus solve the problem underlying the present invention. Moreover, the Board is not aware from its own knowledge that this common general knowledge put forward by the Appellant has been consolidated into a teaching relating to the separability of comparatively complex three phase mixtures by the addition of polyvalent anions. This judgement is supported by the fact that, notwithstanding this common general knowledge and the disadvantages associated with the regeneration of organic phosphate solvents by caustic soda washing according to document (3), the prior art, as represented by US-A-3 767 769, nevertheless indicated that

the problem underlying the disputed patent was solved by further washing the solvent with nitric acid after the caustic soda wash. This second washing step coagulated the suspended solids but the coagulated material settled to the solvent-aqueous liquid interface rather than in the aqueous phase as occurs with the present process.

8. Document (1) discloses that organic phosphate solvents used for extracting phosphoric acid from impure phosphoric acid solutions and from which the phosphoric acid has been stripped may be regenerated by treatment with activated charcoal, an alkali wash and/or distillation. Document (2) merely discloses the use of tributyl phosphate in this extraction process. Document (6) describes the purification of organic solvents used for extracting phosphoric acid by distillation. Document (7) serves to confirm the fact that a solution of trisodium phosphate has a pH of about 12. The disclosure of all these citations is far removed from the subject-matter of the disputed patent and is unsuitable therefore for calling into question the presence of an inventive step for the present subject-matter.

9. The Board therefore considers that the prior art and common general knowledge relied on by the Appellant did not provide any indication to the skilled person that the problem underlying the patent-in-suit would be solved by washing the organic phosphate extractant with an alkali metal orthophosphate solution under the conditions specified in the present Claim 1. The subject-matter of Claim 1 therefore involves an inventive step.

Claims 2 to 13 relate to particular embodiments of the process according to Claim 1 and their patentability is derived from that claim.

Order

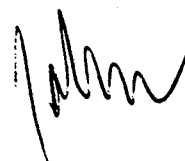
For this reason it is decided that:

1. The appeal is dismissed.
2. The case is remitted to the Opposition Division with the order to maintain the patent in the form as amended on the basis of the documents specified in the communication of 14 May 1985 subject to the additional correction of clerical errors (Claim 1 correction of the spelling of the word "phosphoric"; the insertion of the word "metal" between the words "alkali" and "to" in Claims 1 and 5 and in the insertion to line 40 on page 3).

The Registrar



The Chairman



CP
~~L.H.~~ R.W.A.