Europäisches Patentamt
Beschwerdekammern

European Patent Office
Boards of Appeal

Office europeen des brevets
Chambres de recours

Veröffentlichung im Amtsblatt Ja/~~Nein~~
Publication in the Official Journal Yes/~~No~~
Publication au Journal Officiel Oui/~~Non~~

000215

Aktenzeichen:
Case Number: T 4/83
N° du recours :

ENTSCHEIDUNG / DECISION
vom / of / du 16 March 1983

Anmelder:
Applicant: Exxon Research and Engineering Company
Demandeur :

Stichwort:
Headword: Exxon/purification of sulphonic acids
Référence :

EPÜ / EPC / CBE Article 56,52(1)
"Inventive step : "test procedure not integral part of a process"

Leitsatz / Headnote / Sommaire

Where an invention concerns an improvement in a known purification process, a test described in that connection which serves to confirm the desired result of the purification process does not make obvious the incorporation of the known features of the said test as a final step in the purification process, if the relevant disclosure is confined to the test procedure and gives no pointer to the claimed solution of the problem.

Europäisches Patentamt
Beschwerdekammern

European Patent Office
Boards of Appeal

Office européen des brevets
Chambres de recours

Case Number : T 4/ 83

DECISION

of the Technical Board of Appeal 3.3.1

of 16 March 1983

Appellant: Exxon Research and Engineering Company
New Jersey, N.J.
United States of America

Representative: Bawden, Peter Charles
Esso Chemical Research Centre
P.O. Box 1
Abingdon
Oxfordshire OX13 6BB
England

Decision under appeal: Decision of the Examining Division 004 of 4 August 1982 to reject European Patent Application No. 78 300 810.5 in accordance with Article 97(1) EPC

Composition of the Board:

Chairman: D. Cadman

Member: K. Jahn

Member: L. Gotti Porcinari

SUMMARY OF FACTS AND SUBMISSIONS

I. European Patent Application No. 78 300 810.5 filed on 13 December 1978 and published on 11 July 1979 under publication No. 0 002 907, claiming the priority of the British prior application of 23 December 1977, was refused by decision of the European Patent Office dated 4 August 1982 on the basis of one claim with the following wording:

> "A process for the purification of sulphonic acids characterised by the following sequence of steps washing a crude alkaryl sulphonic acid with from 1% to 30% by weight based on the weight of the alkylate from which the sulphonic acid is derived of water, allowing the aqueous material to settle and removing the aqueous layer and then adding at least 1% by weight based on the weight of the alkylate from which the sulphonic acid is derived of an olefine and heating the sulphonic acid containing the olefine to a temperature in the range 100°C to 150°C for at least 15 minutes."

II. The stated ground for the refusal was that the subject-matter of that claim did not involve an inventive step. As the applicant admitted, washing and removal of an aqueous layer is well known in sulphonic acid purification. Furthermore, the feature of heating sulphonic acid with an olefine at 100 - 150°C for at least 15 minutes was known from FR-A-2 341 565. Therefore, it was obvious to combine the known steps in order to reduce the sulphonic acid to an acceptable level.

.../...

The applicant failed to demonstrate any unexpected advantage in his process, but simply referred to example 1. However, it was impossible to derive from this example that the combination of steps offered unexpected effects, since it did not distinguish the individual steps by their results.

It is true that the Examining Division accepted the applicant's argument that the heat treatment disclosed in the above citation was used in a test of the thermal stability of the olefine-stablised sulphonic acid, the purpose of that step being different from its purpose in the present application. However, it was considered not inconsistent with the present purpose and certainly did not lead away from it.

III. On 24 September 1982 the appellant lodged an appeal against the decision dated 4 August 1982 and on 29 November 1982 submitted a Statement of Grounds, the substance of which was as follows:

In order to demonstrate the extent of purificaton of sulphonic acids that is achieved in the different steps of the process as claimed, the appellant carried out three comparative tests. The results demonstrated that the process of the present invention was the only one to achieve the required content of sulphuric acid of less than 0.5 weight-%.

It is further the appellants' belief that the above data supports the relevance of their second argument that the heating used in the above citation, to test the thermal

stability of the sulphonic acid, is clearly very different from the process of the present invention and does not lead the reader to use a combination of olefine treatment, water washing and heat treatment now shown above to be essential for the purification of the sulphuric acid rich sulphonic acids with which the present invention is concerned.

On the Board's initiative the appellant filed a new set of claims and requested that the decision under appeal be set aside and that the patent sought should be granted. These claims read as follows:

"1. A process for the purification of sulphonic acids rich in sulphuric acid characterised by the following sequence of steps washing a crude alkaryl sulphonic acid with from 1% to 30% by weight based on the weight of the alkylate from which the sulphonic acid is derived of water, allowing the aqueous material to settle and removing the aqueous layer and then adding at least 1% by weight based on the weight of the alkylate from which the sulphonic acid is derived of an olefine and heating the sulphonic acid containing the olefine to a temperature in the range 100°C to 150°C for at least 15 minutes.

2. A process according to claim 1 in which the crude sulphonic acid contains more than 3 wt.% of sulphuric acid.

3. A process according to claim 1 or claim 2 in which the sulphonic acid has been prepared by oleum sulphonation.

.../...

4. A process according to any of the preceding claims in which the alkyl group contains from 20 to 30 carbon atoms.

5. A process according to any of the preceding claims in which from 2% to 10% by weight of the olefine is used.

6. A process according to any of the preceding claims in which the olefine has a molecular weight of from 294 to 336 and the sulphonic acid is a (C_{24} alkyl) benzene sulphonic acid.

7. A process according to any of the preceding claims in which heat treatment is carried out at a temperature between 120°C and 140°C for about 30 minutes".

.../...

REASONS FOR THE DECISION

1. The appeal is in accordance with Articles 106-108 and Rule 64 EPC; it is therefore admissible.

2. There can be no formal objection to the current version of the claims, since it is adequately supported by the specification as originally filed. Claim 1 is based on the original claim 1 in combination with page 2 paragraphs 2 and 4 and page 6 paragraph 4. Claims 2 to 7 correspond to claims 2, 3 and 5 to 8 as filed. Claims 3 and 6 are amended in the sense of page 2 last sentence and page 4 paragraph 3. There is no objection to re-instating the sub-claims at this procedural stage.

3. As indicated at the beginning of the present application, the applicant starts from DE-A-2 707 414 which is equivalent to the French citation and is concerned with the production of thermal- and colour-stable alkaryl sulphonic acids by adding thereto at least 1 weight-% of an olefine and, where appropriate, additionally the same amount of water (cf. page 1, paragraph 1, page 2, paragraph 3, page 3, lines 33-34 in combination with claims 11 and 12). The use of the olefine reduces at the same time the amount of sludge and sulphuric acid (cf. page 6, lines 8 to 14).

   Whilst this process has normally proved satisfactory, the applicant found the sulphuric acid content after the olefine treatment unacceptably high when the sulphonic acid due to be purified was particularly rich in sulphuric acid. From this it is evident that he was addressing himself to the problem of improving the old process in such a way that alkaryl sulphonic acid rich in sulphuric acid could be purified.

In order to solve this technical problem, the applicant proposes a process as set out in claim 1 comprising in a simplified form, the following sequence of steps:

(a) washing the crude sulphonic acid with water (including the removal of the aqueous layer)

(b) addition of olefine

(c) heat treatment

4. When examining for novelty, it should be taken into consideration that any information in a patent specification which conveys to the person skilled in the art a technical teaching belongs to the content of the disclosure irrespective of whether or not it falls within the scope of the claims or what purpose it serves. In applying this principle to the case in suit, besides the essential teaching of the citation, which consists of using an olefine in a process of purification of sulphonic acids, information concerning both the preliminary steps and the features of the final test for thermal stability must be considered as well.

Example 1, to which some other examples directly or indirectly refer, describes such a method of operation. Thereby a C_{24}-alkyl-benzene sulphonic acid, after being stripped of sulphur dioxide and sludge, is washed with aqueous hydrochloric acid (in order to remove the excess of sulphric acid) and subsequently treated with a C_{24} olefine. Samples of the resulting mixture were stored at 120°C for four days and their colours measured. Although

the feature of the olefine treatment and that of the colour test are identical with step (b) and (c) of the process as claimed, the washing medium used in the preliminary washing of the crude sulphonic acid is clearly different. For that reason the claimed process is novel.

5. It is therefore to be examined whether the subject-matter of claim 1 is obvious in relation to that prior art. This question was answered by the Examining Division in the affirmative on the basis of the cited prior art in combination with common general knowledge. As to common general knowledge, this was substantiated not by citing a document but by referring to the applicant's statement in his letter of 20 November 1980 where he admitted that it was well known to wash and remove an aqueous layer in sulphonic acid purification. The nature of the washing agent was not mentioned. That incomplete statement was not elucidated by a new claim, simultaneously filed, comprising the step of washing of the crude sulphonic acid with water and that of olefine treatment in its pre-characterising part. Since this combination was not known from the citation, this claim evidently offended against Rule 29(1)(a) which stipulates that only technical features which, in combination, are part of the prior art, shall form the pre-characterising part of a claim. The general principle that admitted facts need not be proved applies exclusively to cases where clear statements are made. That does not hold true in this case. In the case at issue it is simple to complete the applicant's statement, since the citation, which describes a purification process taking advantage of washing with aqueous hydrochloric acid, where appropriate, mentions in its introductory part that such washing is commonly used (cf. page 1, paragraphs 3 and 4). Consequently, the purification of sulphonic acids by washing

with water alone cannot be regarded as common general knowledge.

6. The only relevant state of art which remains for the assessment of inventive step is the above mentioned FR-A-2 341 565. This document teaches that the colour and thermal stability of alkaryl sulphonic acids can be improved by incorporating therein at least 1 weight-% of an olefine. Furthermore, it is mentioned that this olefine treatment reduces the amount of sludge and sulphuric acid to a extent whereby a preliminary removal of sludge (by decanting with solvent hydrocarbons) and of sulphuric acid by washing with aqueous hydrochloric acid even in the purification of alkaryl sulphonic acids with relatively long chains, is superfluous in certain cases (cf. page 6, lines 3 to 22). It would have been obvious for a skilled person faced with the problem of adapting the old process to the purification of sulphonic acids rich in sulphuric acid to introduce that, formerly optional, washing step as obligatory and perhaps to intensify the washing. However, it was not obvious to replace aqueous hydrochloric acid with water as washing agent, since neither the prior art offered an example therefor, nor was there any prospect of solving the problem in question, having in mind the common textbook knowledge that aromatic sulphonic acids are more soluble in water than in aqueous hydrochloric acid (cf. the until now uncited passage of Houben-Weyl, Methoden der Organischen Chemie, Vol. IX, 1955, 435).

7. Particularly suprising is the applicant's perception that the technical problem in question might be solved by a process comprising not only the step of washing and of olefine treatment, but additionally the heat treatment of the alkaryl sulphonic acid product as an essential final

step in the purification process, which step was formerly used in a test merely to confirm the desired result. Contrary to the examination for novelty, in examining inventive step on the basis of a single document, the purpose which a known technical feature serves can become crucial. As already said, the known process finishes with the olefine treatment. The test, which may subsequently be carried out, serves exclusively to verify, where appropriate, whether the desired colour and thermal stability of the sulphonic acids have been achieved, and gives no incentive to incorporate the features of the said test in the purification process as a final step, since it could not be expected that this sequence of steps would render possible the purificaion of sulphonic acids particularly rich in sulphuric acid. The teaching of the present application must, independently of whether it is expressed in the form of claim 1 or its sub-claims, be regarded as surprising, and hence involving an inventive step.

For these reasons it is decided that:

1. The decision of the Examining Division 004 of the European Patent Office dated 4 August 1982 is set aside.

2. The case is remitted to the first instance with the order to grant a European patent on the basis of the following documents:

Description pages	1 and 7	dated 2.4.81,	received on		3.4.81
" "	2 and 4	dated 1.3.83,	"	"	5.3.83
" "	3 and 5	dated 20.11.80,	"	"	1.12.80
" page	6	dated 3.10.80,	received on		7.10.81

Claim 1 dated 11.2.83, received on 15.2.83

Claims 2 to 7 dated 1.3.83, received on 5.3.83

The Registrar: The Chairman:

J. Rücker [signature] ... Cadman [signature]

.../...