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**D E C I S I O N**  
of 24 January 1996

**Case Number:** T 0062/93 - 3.3.3

**Application Number:** 86301790.1

**Publication Number:** 0194877

**IPC:** C08B 11/20

**Language of the proceedings:** EN

**Title of invention:**  
Purification of cellulose ethers

**Patentee:**  
THE DOW CHEMICAL COMPANY

**Opponent:**  
HOECHST Aktiengesellschaft

**Headword:**  
-

**Relevant legal provisions:**  
EPC Art. 56, 100(a) & (b)

**Keyword:**  
"New ground (Article 100(b) EPC) - excluded"  
"Inventive step - affirmed"

**Decisions cited:**  
G 0010/91

**Catchword:**  
-



Case Number: T 0062/93 - 3.3.3

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.3  
of 24 January 1996

**Appellant:**  
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**Representative:** -

**Respondent:**  
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**Decision under appeal:** Interlocutory decision of the Opposition Division  
of the European Patent Office dated 13 October  
1992 and issued in writing on 9 November 1992  
concerning maintenance of the European patent  
No. 0 194 877 in amended form.

**Composition of the Board:**

**Chairman:** C. Gérardin  
**Members:** R. Young  
J. A. Stephens-Ofner

## Summary of Facts and Submissions

- I. The mention of the grant of European patent No. 0 194 877, relating to the purification of cellulose ethers, in respect of European patent application No. 86 301 790.1, filed on 12 March 1986 and claiming a United States priority of 15 March 1985 (US 712287) was announced on 30 August 1989 (cf. Bulletin 89/35).
- II. Notice of Opposition was filed on 26 May 1990, on the ground of lack of inventive step. The opposition was supported by the documents:
- D1: Filtration & Separation; March/April 1979, pages 176 to 180;
  - D2: US-A-4 404 370; and
  - D3: Ullmanns Enzyklopädie der technischen Chemie, 4th Edition, Vol. 9, page 197.
- III. By an interlocutory decision which was given at the end of oral proceedings held on 13 October 1992 and issued in writing on 9 November 1992, the Opposition Division found that the patent could be maintained in amended form on the basis of a set of Claims 1 to 9 as filed during the hearing, Claim 1 of which reads as follows, and which differs from the version as granted by insertion of the underlined portions:

"A batchwise process for removing impurities from a cold water-soluble, thermally precipitating in hot water cellulose ether composition by contacting said composition with an aqueous alkali metal salt solution at a temperature above that at which the cellulose ether precipitates, characterised in that said process comprises

- (a) providing a functionally effective thickness of said cellulose ether composition in a filtration device,
- (b) contacting said cellulose ether composition with an aqueous alkali metal salt solution, in a manner such that said solution migrates through said thickness of the cellulose ether composition maintaining an undisturbed substantial thickness of the cellulose ether composition in the filtration device, and effecting a gradient impurity concentration through the thickness, and
- (c) repeating the said extraction step (b) at least once with the extracting solution of each extraction step except the last being the used batch of extracting solution from the corresponding immediately succeeding extraction step for a previously extracted batch whereby the extracting solution of the second and any subsequent extraction step contains a lesser concentration of alkali metal salt than the extracting solution of the immediately preceding extraction steps."

Claims 2 to 9 of the amended version are directed to elaborations of the process according to Claim 1.

According to the decision, whilst it was prima facie obvious to apply the counter-current washing process disclosed in the closest state of the art document, D2, to the purification of hot water precipitating cellulose ethers, by omitting alcohol from the washing solution, nevertheless, blocking of the system would have been expected due to a build up of low molecular weight cellulose ethers dissolved in the washing solutions, which could not leave the system. This did not occur, however, and the stabilisation of the concentration of the cellulose ethers with the consequent reduction of the loss of cellulose ether during the washing process was unexpected, so that an inventive step could be recognised.

- IV. On 15 January 1993, a Notice of Appeal against the above decision was filed, together with payment of the prescribed fee.

In the Statement of Grounds of Appeal, filed on 18 March 1993 and subsequent written submissions, the Appellant (Opponent) agreed with the finding of the decision under appeal concerning the omission of alcohol from the process of D2 and argued essentially as follows:

- (a) The evidence presented by the Respondent in support of the effect which had led to the recognition of an inventive step over D2 had been in the form of graphs which demonstrated that, if the salt concentration of the wash solution was reduced, the concentration of hydroxypropyl methylcellulose (HPMC) increased, so that, except for one low value of the HPMC concentration which lacked credibility, the relationship was a straight line. The maximum HPMC concentration was furthermore so low that the alleged problem of blockage of the system would not arise.

- (b) It was known from D2 that the loss of cellulose ether could be reduced in a countercurrent wash system. Such a loss was in any case excluded in the process of the patent in suit because the cellulose ether was insoluble in the high salt concentrations of the discarded liquor. Consequently, the effect of avoidance of cellulose ether loss was not surprising.
- (c) On the contrary, a review of the latter process as exemplified in the patent in suit showed that the product concentrations in the wash liquors did not approach a steady state value surprisingly and automatically as alleged by the Respondent, but rather were related to the amount of fresh water added to the system.
- (d) A number of details of the evidence provided by the Respondent were, moreover, not reflected in the Example of the patent in suit, so that there was a case of insufficiency of disclosure in the sense of Article 100(b) EPC.

Furthermore, the following document was cited for the first time:

D4: N. Sarkar, J. Appl. Pol. Sci., Vol. 24, (1979), pages 1073 to 1087.

V. The Respondent (Patentee) argued in essence as follows.

- (a) There were significant differences between the process according to Claim 1 and the process of D2 in addition to the omission of alcohol from the wash solution. In particular, Claim 1 required (i) the filter cake thickness to be increased to a

functionally effective thickness in which a gradient concentration existed, (ii) the filter cake to remain undisturbed during filtration, and (iii) the process to be conducted batchwise instead of continuously.

(b) The graphical evidence referred to showed that the HPMC concentration in the second and third wash solutions, after an initial, expected increase, actually decreased after about twenty batches to provide a steady state condition in which the cellulose ether content of the third wash filtrate was surprisingly below that of the second wash filtrate. This low concentration, which had been alleged by the Appellant to lack credibility was nevertheless the measured value.

(c) Even the analysis of the process by the Appellant, in spite of a number of unclarities, nevertheless also indicated an unpredictable final HPMC concentration, and this lay at the heart of the invention.

VI. In a communication accompanying a summons to oral proceedings, the Board drew attention to the ruling, in the opinion of the Enlarged Board of Appeal G 0010/91, concerning the ground of objection under Article 100(b) EPC, raised for the first time by the Appellant during the appeal proceedings.

VII. With a submission filed on 27 December 1995, the Respondent refused permission for this further ground to be considered.

VIII. Oral proceedings were held before the Board on 24 January 1996.

IX. The Appellant requested that the decision under appeal be set aside and the patent be revoked in its entirety.

The Respondent requested that the decision under appeal be confirmed, i.e. that the appeal be dismissed.

### Reasons for the Decision

1. The appeal is admissible.
2. *Admissibility of new ground of opposition*

Following the ruling in the opinion of the Enlarged Board of Appeal G 0010/91, and in the light of the refusal by the respondent to have the new ground of opposition (Article 100(b) EPC) admitted to the proceedings, this ground was completely excluded from the proceedings.

3. *Admissibility of new document*

The document D4, which is a learned article of a generally factual nature, is referred to in a declaration by the author filed during the opposition proceedings (Affidavit of Nikis Sarkar, filed on 1 February 1992, page 2) and was cited in support of a line of argument already on file with the Statement of Grounds of appeal. It has furthermore been commented on in detail by the Respondent (submission of 27 December 1995, paragraph 24).

Consequently, the Board sees no ground for excluding it from consideration. It is therefore introduced into the proceedings under Article 114(1) EPC.



4. Allowability of amendments

4.1 Claim 1 is based on Claims 1 and 6 as granted and, as regards the first two underlined insertions, on the description at column 1, lines 23 to 29, read in the light of column 4, lines 12 to 21 and column 6, lines 29 to 31 of the patent as granted. In this respect, the Board concurs with the view, expressed in the minutes of the oral proceedings held before the Opposition Division, that it is evident that the patent in suit relates to water soluble, thermally precipitating cellulose ethers which can be heated above their gel point.

The reference inserted in Claim 1 to an "undisturbed" thickness is based on column 4, lines 39 to 41 of the patent as granted.

The term "batch" is inherent in the phrase "batchwise process" in Claim 1 as granted.

4.2 Claims 2 to 5 remain unamended compared with the granted version, and Claims 6 to 9 correspond to Claims 7 to 10 as granted.

4.3 The amendments to the description either correspond to those made in the claims, or are adaptive to the amended claims.

The effect of the amendments is, furthermore, to restrict the scope of Claim 1.

Consequently, the amendments are allowable under Article 123 EPC. In any case, no objections were raised by the parties to the amendments during the appeal.

5. *Closest state of the art; the technical problem*

The patent in suit is concerned with the removal of impurities from cellulose ethers, for instance from methyl hydroxypropyl ethers of cellulose (HPMC), wherein, after etherification, the cellulose ether product is extracted in countercurrent on a filter with a purifying agent. Such a process is, however, known from D2, which is considered to represent the closest state of the art.

5.1 According to D2, a cellulose ether, preferably of the type which does not flocculate in hot water is purified by filtering off the suspension agent, e.g. isopropanol, extracting the reaction product with a purifying agent different from the suspension agent and drying it under suction, the product after filtering being extracted in countercurrent with the purifying agent at least once on the same continuously working filter (Claim 1; column 1, lines 54 to 64).

The purifying agents are preferably mixtures of water and methanol or ethanol containing 20 to 40 wt% water, optionally with the addition of some isopropanol (column 2, lines 40 to 50).

Suitable filters are those whose surface can be clearly divided into individual zones, e.g. plate, vacuum drum, and band filters (cf. column 3, lines 23 to 25).

According to the procedure of a typical example (Example 1), a suspension of 10 wt% of carboxymethyl cellulose in a mixture of 83 wt% of isopropanol and water is delivered from the etherification stage through a suction cell to a vacuum drum filter, the surface of the drum being divided into twenty zones,

from which the filtrates can be separately removed. After separation of the mother liquor and drying under suction, the product is subjected to countercurrent extraction in two zones with a mixture of 65% of methanol and 35% of water introduced through nozzles, the remaining zones serving for suction drying. The 20mm thick cake is stripped from the filter with a knife.

5.2 Compared with this state of the art, the technical problem underlying the patent in suit can be seen in the definition of an alternative application of a simplified process for removing impurities from a cellulose ether, with a capability of avoiding loss of cellulose ether product.

5.3 The solution proposed according to Claim 1 of the patent in suit is to apply the process to the purification of a hot water flocculating cellulose ether and to modify it in the following respects:

- (i) to omit alcohol from the purifying solution;
- (ii) to increase the thickness of the cake to a "functionally effective thickness" so as to establish a gradient impurity concentration;
- (iii) to avoid substantial agitation of the cake; and
- (iv) to apply a batchwise instead of a continuous process, using batches of aqueous alkali metal salt solution of decreasing salt concentration in successive extractions of the cake.

5.4 It can be seen from the results of the example in the patent in suit, that an efficient removal of impurities is enabled without the use of organic solvent, let alone two different organic solvents as

disclosed in D2, together with a very small loss of cellulose ether product, in respect of a hot-water flocculating cellulose ether (HPMC). It therefore represents a simplification.

Furthermore, the evidence presented in graphical form at the oral proceedings before the Opposition Division (copy filed with submission received on 15 October 1992) and also referred to in the written and oral submissions in the appeal indicates that the loss of cellulose ether product, especially of low molecular weight cellulose ether product, dissolved in the wash solutions falls, after an initial rise during the treatment of the first twenty or so batches and when the steady state has been reached, to a low, relatively constant value (submission filed on 27 December 1995).

5.5 Although the Appellant had expressed doubts on more than one occasion as to the credibility of this low value, citing technical results filed with the Statement of Grounds of Appeal and elaborated upon in the submission dated 28 April 1994 (sentence bridging pages 5 and 6), it was admitted in a later submission of the Appellant (filed on 15 January 1996) that these technical results had not been based on any experimental work but merely on information provided in the patent in suit. Furthermore, any doubts as to the existence of the effect relied upon by the Respondent were formally withdrawn by a corresponding declaration of the Representative of the Appellant at the oral proceedings before the Board.

5.6 The effect was furthermore confirmed by an uncontested submission of the Respondent, according to which an industrial application of the method according to the patent in suit had reduced washing losses of a named proprietary cellulose ether product from 15-25% (using cross-washing) to about 3% (submission filed on 27 December 1995, paragraph 17).

Consequently, it is credible that the claimed measures provide an effective solution of the stated problem.

6. *Novelty*

The novelty of the subject-matter claimed in the patent in suit has not been disputed. Nor does the Board see any reason to take a different view of the matter.

Consequently the subject-matter claimed in the patent in suit is held to be novel.

7. *Inventive step*

In order to determine the question of inventive step, it is necessary to consider whether the skilled person, starting from D2 would have expected an efficient alternative process with the potential to avoid cellulose ether product loss in the case of a hot water flocculating cellulose ether to result from effecting the combined measures and modifications defined in section 5.3, above to the countercurrent process.

7.1 Before the question of the modifications can be answered, however, it is necessary to establish first whether the skilled person would have considered the measure of applying the teaching of D2 to cellulose ethers which flocculate in hot water.

On the one hand, that part of the disclosure in D2 which relates to hot water flocculating cellulose ethers is merely an acknowledgment of prior art relating to conventional purification using hot water, and is not elaborated further. In particular, there is no suggestion that loss of cellulose ether product is still a problem (column 1, lines 22 to 30).

On the other hand, the disclosure of the countercurrent process is offered as the solution to a problem which arises primarily with another kind of cellulose ethers (those which do not flocculate in hot water). Furthermore, it is disjunctive to the acknowledgment, so that there is no suggestion that the countercurrent process would need to be used if the cellulose ether were of the hot water flocculating type.

Consequently, the disclosure of D2 is not concerned with the technical problem to which the patent in suit provides a solution. Hence, the skilled person would have no incentive to consider the disclosure of D2 for the relevant purpose.

7.2 Assuming, however, for the sake of argument, that the skilled person were, in spite of the above lack of incentive, to consider D2 as a possible starting point, and in spite of the absence of any reason to associate the countercurrent process with the solution of the stated problem, nevertheless to decide on this

line of attack, the question arises as to whether the modifications (i) to (iv) defined in section 5.3, above would themselves be obvious from the state of the art.

- 7.2.1 Taking modification (iv) first, there is no suggestion in D2 to use a batch process instead of a continuous process, since the latter is an essential requirement in D2 (Claim 1; column 1, lines 5 to 7; column 2, lines 19 to 21 and 51 to 55; column 3, line 1).

According to D1, which is a review of vacuum belt filters, one of the advantages of an application to carboxymethyl cellulose filtration is the use of a continuous process (page 178, right hand column, "Applications", A.).

Consequently, there would be no incentive to use a batch process.

The remaining documents D3 and D4, which are concerned with the properties of cellulose ethers do not relate to filtration processes and are therefore more remote.

Consequently, it would not be obvious to make the process of D2 a batch process.

- 7.2.2 As regards modification (i), there is no suggestion in D2 to omit alcohol from the washing solution. On the contrary, the presence of a water miscible compound such as methanol, ethanol or isopropanol is presented as essential (column 1, lines 24 to 30).

The disclosure of D1 is less detailed than that of D2 with regard to the composition of the wash liquor, but only alcohol containing mixtures are specified (page 178, right column, "Applications"; page 180, Figure 4).

Consequently, it would not be obvious to carry out modification (i) in D2.

7.2.3 The modifications (ii) and (iii) will be considered together, because they are explained in the patent in suit as being associated, in combination, with the phenomenon which enables loss of low molecular weight cellulose ether product to be avoided (column 4, lines 30 to 46; column 5, lines 40 to 56; column 8, lines 31 to 34). This is furthermore confirmed by the uncontested evidence of the Respondent based on the results of an industrial application of the process according to the patent in suit (section 5.6, above).

7.2.3.1 Whilst no numerical figure is placed, according to the patent in suit, on the critical thickness of the cake, it must nevertheless be sufficient to provide a "functionally effective thickness", i.e. to maintain a gradient impurity concentration, which in turn requires a sufficient amount of the thickness to be preserved after accounting for any agitation which occurs when the extraction solution is added to enable the extracting solution to migrate through the thickness without disturbing its uniformity. In the examples of the patent in suit, a cake thickness of the order of 30 cm is maintained, with extracting solution being added to the top of the cake so as to migrate through the cake without disturbing it (column 8, line 63 to column 9, line 9).



- 7.2.3.2 According to D2, in contrast, cake thicknesses of the order of only 2-3 cm are exemplified (Example 1 and Example 3, Table 1) and the extracting solution is introduced through nozzles. Such an application of extracting solution would presumably be in the form of jets.
- 7.2.3.3 The disclosure of D1 is less detailed but does not contradict D2 as to the cake thickness, and additionally indicates the possibility of an even more vigorous measure, namely steam injection (page 180, left column, second para. following Figure 4).
- 7.2.3.4 The argument of the Appellant, that any thickness of the cake must be a "functional thickness", and there was no reason to assume disturbance of the cake during washing according to D2 or D1, is unconvincing in view of the relevant disclosures of a relatively small thickness of filter cake and the use of nozzles and optional steam injection respectively, indicating a relatively vigorous application of extractant, which would be very unlikely to leave any part of the cake undisturbed, let alone establish a "functionally effective" undisturbed thickness as required by the solution of the technical problem.

Consequently, the filter cakes of D1 and D2 cannot be regarded as implicitly fulfilling the requirements of features (ii) and (iii) of the solution of the technical problem. On the contrary, the arrangements according to the teachings of D2 and D1 are precisely the opposite of what would be necessary for the achievement of such a solution.

Hence, it would not have been obvious to provide the modifications (ii) and (iii).

In summary, there was no incentive to provide any of the modifications (i) to (iv), let alone all of them in combination.

7.3 Finally, even if, in spite of the findings above, the skilled person had in some way been able to recognise the relevant technical problem from D2, had applied it to the countercurrent process and, further, had conceived the series of modifications (i) to (iv) above, nevertheless the question arises of what would have been expected to occur when such a process was applied in industrial practice, i.e. with a large number of cakes of raw cellulose ether product being treated in succession.

Under these conditions, each successive batch of raw cellulose ether filter cake will carry with it the proportion it contains of low molecular weight, soluble cellulose ether product into the wash system, from which the product cannot then escape, due to its insolubility in the high salt concentration of the only wash liquor which is discarded, as explained in the decision under appeal (Reasons, para. 10 a)), the Affidavit of Sarkar, filed on 1 February 1992, and the submissions of the Respondent, more particularly that filed on 27 December 1995.

Thus, there would be an expectation of an ever increasing concentration of such cellulose ether product in the non-discarded wash liquors of lower salt concentration, leading to a corresponding increase in their viscosity and, ultimately, to a blockage of the system.

7.3.1 The argument of the Appellant, that blocking would not have been expected because of the addition of replenishment water to the system and the low concentrations of cellulose ether product released is unconvincing because it is based on the technical results supplied with the Statement of Grounds of appeal. These results were admitted to have been calculated from information in the patent in suit (section 5.5, above), and were based on data relating to the treatment of only a single batch.

In particular, the results fail completely to take account of what would be expected to happen when the process is performed on an industrial scale (see above).

Thus the concentration of low molecular weight product in one particular wash liquor is irrelevant, and the addition of replenishment water merely implies an approximately constant total quantity of wash water in the system, which could not prevent the expected build up of low molecular weight cellulose ether product.

7.3.2 The further argument of the Appellant, that a build up of low molecular weight cellulose ether product would not have been expected from theoretical considerations based on D4, in which, according to Figure 2 on page 1078, the light transmission readings showed, if extrapolated to the temperature of the process exemplified in the patent in suit (120°C), that all the cellulose ether was precipitated even at very low concentrations, is not convincing for the following reasons:

7.3.2.1 The conclusion is contrary to the affidavit of Mr. Sarkar, according to which he believed, when first told of the concept of the invention by the inventor,

that the low-molecular weight cellulose ethers would build up in the process (page 2, third paragraph). Mr. Sarkar is also the author of D4, as is mentioned in the affidavit (page 2). Consequently, it would be inconsistent to assume that the absence of such build up would be obvious from the contents of D4.

In this connection, the argument of the Appellant at the oral proceedings, that weight should not be given to the statements of Mr. Sarkar because he was also an employee of the Respondent was a mere allegation unsupported by so much as a shred of evidence. The Board cannot entertain such unsupported allegations against a reputable expert such as Mr. Sarkar.

- 7.3.2.2 Notwithstanding the above, a closer examination of the text pertaining to Figure 2 in D4 shows that the decrease in light transmission merely illustrates the initial precipitation of the high molecular weight fractions, and does not reflect even the average molecular weight of the sample, but rather an unknown molecular weight somewhat higher than the average molecular weight.

Thus the figure gives no information about the precipitation behaviour of the relevant low molecular weight cellulose ethers at all, and would provide no reason for the skilled person to conclude that the expected build up of such cellulose ethers would, in practice, not occur.

- 7.4 To sum up, the skilled person, starting from D2 would have had no hint to the existence of the stated problem or any of the modifications constituting its solution according to the patent in suit, let alone all of them in combination. Even if the proposed solution had been obvious in itself, however, the

skilled person would have concluded that it would not be workable industrially because of a build up of relatively soluble low molecular weight cellulose ethers in the wash solutions, leading ultimately to a blockage.

That this does not occur is considered surprising.

8. Hence, the subject-matter of Claim 1 is not only novel but also involves an inventive step. By the same token, the subject-matter of Claims 2 to 9, which are directly or indirectly dependent on Claim 1, is also novel and involves an inventive step.

**Order**

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

The appeal is dismissed.

The Registrar:

  
E. Gorgmaier

The Chairman:

  
C. Gérardin

