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Veröffentlichung im Amtsblatt	<input checked="" type="checkbox"/> Ja/Nein
Publication in the Official Journal	<input checked="" type="checkbox"/> Yes/No
Publication au Journal Officiel	<input checked="" type="checkbox"/> Oui/Non

Aktenzeichen / Case Number / N^o du recours : T 137/83
Anmeldenummer / Filing No / N^o de la demande : 79 101 648.8
Veröffentlichungs-Nr. / Publication No / N^o de la publication : 58 27

Bezeichnung der Erfindung: Method of removing contaminants from water
Title of invention:
Titre de l'invention :

Klassifikation / Classification / Classement : C 02 C5/00

ENTSCHEIDUNG / DECISION

vom / of / du 15 April 1986

Anmelder / Applicant / Demandeur : THE DOW CHEMICAL COMPANY

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence : Contaminants/DOW

EPÜ / EPC / CBE
Article 56 EPC
"Inventive step"
Disincentive to adopt the claimed technique.

Leitsatz / Headnote / Sommaire

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Case Number : T 137 /83

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 15 April 1986

Appellant : The Dow Chemical Company
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Decision under appeal : Decision of Examining Division 026 of the
European Patent Office dated 25.11.82*
refusing European patent application
No 79 101 648.8 pursuant
to Article 97(1) EPC
* posted 14.03.83

Composition of the Board :

Chairman : K. Jahn
Member : P. Lançon
Member : P. Ford

Summary of Facts and Submissions

- I. European patent application 79 101 648.8 filed on 30 May 1979 and published on 12 December 1979 with publication number 0 005 827 claiming the priority of the prior application of 30 May 1978 (US 910 175) was refused by the decision of the Examining Division 026 of the European Patent Office dated 25 November 1982.

The decision was based on Claim 1 handed over in the oral proceedings on 25 November 1982 and Claims 2 to 10 filed on 29 December 1981, as well as on auxiliary Claims 1 to 8 also handed over in the oral proceedings on 25 November 1982.

Main Claim 1 read as follows:

A process for removing at least one organic material having a relative volatility with respect to water of at least one from water contaminated therewith said process comprising passing a current of vapor, sufficient to vaporize at least a portion of said organic material, through a moving stream of said contaminated water and then separating vapor containing the organic materials from the water to produce water having a reduced concentration of said organic material, characterised in that the organic material is selected from the group consisting of chlorinated phenols, phtalate ester, phosphate esters, phosphorothioate esters, chlorinated benzenes, chlorinated biphenyls, polynuclear armatics, nitrobenzene, alkyl nitrobenzenes, chloroalkenes, chlorocycloalkenes and chloroethers and in that it has a normal boiling point in excess of 200°C.

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Subsidiary Claim 1 comprises the additional feature of pH adjustment of the contaminated water.

II. The application was refused on the grounds that

(i) main Claim 1 did not involve an inventive step (Article 56 EPC);

(ii) the auxiliary Claim 1 could not be accepted for reasons of lack of clarity (Article 84 EPC).

According to the Examining Division, main Claim 1 is unpatentable over the teaching of the article:

"Air Stripping of Organics from Wastewater : A Compendium" by L.J. Thibodeaux. Proceedings of the National Conference on complete water re-use : Water's interface with energy, air and solids, A I Ch E, N.Y. 1975, pages 358 to 378 (hereafter "Thibodeaux's article").

The Thibodeaux article teaches that relative volatilization rate and not boiling point is the factor to be considered for the purpose of the process of removing contaminants from water. While some of the passages of the citation might be interpreted as suggesting a relationship between boiling point and percentage stripping, it does not establish a clear prejudice against attempting vapour

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stripping of high boiling compounds having sufficient relative volatility. Bearing this in mind, the skilled man would not have had to exercise any inventive ingenuity in attempting to remove organic material having a relative volatilization rate with respect to water of at least one from waste water by vapour stripping.

Regarding auxiliary Claim 1, the Examining Division concluded that the additional feature left the reader in doubt about the effective restriction.

III. On 16 May 1983, the appellants lodged an appeal against the decision of 25 November 1982 with the payment of the appropriate fee and submitted a Statement of Grounds on 20 July 1983.

IV. In their Statement of Grounds, the appellants argued that

- (1) the formulation of the problem of removal of the organic contaminants mentioned and having a boiling point above 200°C was novel;
- (2) the prior art ("Thibodeaux") would lead the person skilled in the art away from the claimed process.

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With particular reference to table 1, page 362, the appellants contended that, while it was possible to try to use the process described in "Thibodeaux" in consideration of the theoretical teaching, the concrete results would lead the man skilled in the art away from using this solution for contaminants having boiling points above 200°C.

(3) the comparison of data from "Thibodeaux" (table 1, page 362 and table 6, page 371) and of those of the application (table III) (represented on a graph) demonstrates surprising results.

- V. In an answer to observations of the Board, the appellants alleged the existence of a prejudice based on a report of the UNITED STATES ENVIRONMENTAL PROTECTION AGENCY of 11 July 1978 (hereafter "USEPA report") and submitted that it should be considered as an evidence of the opinion of experts in the art at the time of the invention.

With reference to the "Thibodeaux" instructions relating to the handling of samples at a pH of 3 or lower, they alleged that this was not used to avoid dissociation.

They also put forward a new argument of a commercial success allegedly due to high efficiency and low operating costs.

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VI. Oral proceedings were arranged at the request of the appellants on 15 April 1986.

During the oral proceedings, the appellants developed the arguments presented in their last letter.

They submitted that the fact that the man skilled in the art had to overcome an important technical prejudice would result from calculation of the relative volatility itself; on the basis of examples they alleged that calculations according to Thibodeaux would lead to a relative volatility close to 1 whereas calculations according to the application would lead to higher figures in the case of contaminants as defined in the application.

They concluded that, accordingly, air stripping was generally considered as a non-feasible process for such contaminants, for which the carbon bed adsorption method was generally used (see CHEMICAL ABSTRACTS, Vo. 61, No. 13, 21 December 1964, abstract No. 15834c: Contamination of drinking water with PCP (pentachlorophenol). Analysis and removal. K. UEDE, cited in the search report (hereafter: "K. Uede abstract")).

Invited to comment on the pH adjustment, the applicants admitted that, although it is important for the process of the invention, it was common general knowledge.

They further stressed the efficiency and the low operating costs of the claimed process which is allegedly not only used by the appellants but also by a number of licensees, and they put forward the technical advantages and surprising efficiency as the cause of the commercial success.

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VII. Finally, the appellants requested that the decision under appeal be set aside and that a patent be granted on the basis of Claims 1 to 7, submitted during the oral proceedings. Independent Claim 1 reads as follows:

Process for removing organic material from water contaminated therewith comprising passing a current of vapor, sufficient to vaporize at least a portion of said organic material, through a moving stream of said contaminated water and then separating vapor containing the organic material from the water to produce water having a reduced concentration of said organic material, said process being characterized in that :

- (a) the organic material is a chlorinated phenol, a phthalate ester, a phosphate ester, a phosphorothioester, a chlorinated benzene, a chlorinated biphenyl, a polynuclear aromatic compound, a chloroalkene, a chlorocycloalkene, a chloroether;
- (b) the organic material has a normal boiling point in excess of 200°C;
- (c) the organic material has a relative volatility with respect to water of at least about 4, said relative volatility being determined according to the following equation:

$$c_w = \frac{y_c/y_w}{x_c/x_w}$$

wherein y_c and y_w are the mole fractions of contaminant (y_c) and water (y_w) in vapor phase at equilibrium, the ratio being obtained at the vapor azeotrope,

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x_c and x_w are the mole fractions of contaminant (x_c) and water (x_w) in liquid phase at equilibrium, the ratio x_c/x_w being based on the solubility of the contaminant in water;

and

- (d) the vapor is passed countercurrently through the said moving stream of contaminated water and
- (e) the pH of the said contaminated water is adjusted to a level sufficient to prevent substantial dissociation of said organic material (more than 10 per cent) if it is capable of dissociation.

Reasons for the decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. There is no formal objection to the current versions of the claims since they are adequately supported by the original disclosure.

Claim 1 combines original Claims 1, 2 and 6 and incorporates features of the description (page 3, lines 23-29; page 4, line 30 to page 5, line 3; page 5, line 29 to page 6, line 7; page 6, lines 16-33; page 7, lines 9-23 and page 8, lines 13-20).

Claims 2 to 7 correspond to or are based on original claims 5, 3, 4, 8, 9 and 10 respectively.

The Board considers that the claims are clear (Article 84 EPC). Indeed the feature of adjusting the pH

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to prevent dissociation (impliedly where necessary) is not in contradiction with the fact that some compounds covered by Claim 1 do not dissociate.

3. The application relates essentially to a process for removing organic material from water contaminated therewith, the organic material being a chlorinated phenol, a phthalate ester, a phosphate ester, a phosphorothioester, a chlorinated benzene, a chlorinated biphenyl, a polynuclear aromatic compound, a chloroalkene, a chlorocycloalkene, a chloroether having a normal boiling point in excess of 200°C.
4. The application has been rejected by the first instance on the grounds that the subject-matter according to Claim 1 did not involve an inventive step with regard to Thibodeaux's article taken alone. The other document quoted in the Reasons for the Decision was relied upon for a different purpose.

After consideration of the true teaching of the State of the Art and for the reasons given later in this decision, the Board is of the opinion that the closest document of the relevant state of the art is the "K. Uede abstract".

5. There is stated that, in view of the widespread application of PCP as a weed killing agent, it is probable that it contaminates drinking water. The problem with which the authors of the article were concerned was the one of finding a method for removing this contaminant. Experiments had shown that activated carbon was most effective for removing it.

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6. Starting from this closest prior art document, the technical problem underlying the present invention was to provide an efficient and economically attractive alternative method to remove pentachlorophenol as well as other organic contaminants in water such as those produced, for instance, in the pesticides industry (description page 5, lines 3-22) with a boiling point in excess of 200°C.
7. The problem is essentially solved by the Appellants by a process comprising vapor stripping of the contaminated water under the conditions indicated in more detail in the cited Claim 1.

In view of the result of the 30 examples given in the description relating to PCP, other chlorinated phenols, diphenyloxid, trichlorobenzene and the further 15 compounds listed in Tables II and III of the application, the Board is satisfied that the problem is effectively solved.

8. As the solution claimed by the appellants is quite different from the solution of the closest prior art "K. Uede abstract", there is no objection to the novelty of the subject-matter of Claim 1. It has to be added that the first instance did not question the novelty when considering the rejected Claim 1, even in consideration of "Thibodeaux's article". The Board is of the same opinion and the reasons therefore can be derived from the next paragraphs.
9. The Board in agreement with the Examining Division considers that "Thibodeaux's article" is a very relevant document of the state of the art.

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9.1 In this document a literature survey of common organic pollutant constituents is given comprising chlorinated solvents, chlorinated hydrocarbons, nitrobenzene, nitro compounds, phenol wastes, phenolic compounds, phenol derivatives, dichloroethyl ether among others (see pages 360 to 362).

Table I (page 362) contains a brief summary of relative volatilities and Henry's Law constant for common industrial gases and liquids and also quotes the normal boiling points. The accompanying comment to the table states firstly, that "many of the common liquid species are more volatile than water and will therefore desorb readily as indicated by a relative volatility greater than one". Secondly, it adds that "although some high molecular organics may have low pure component vapor pressures and high boiling points compared to water, they nevertheless exhibit large relative volatilities due to large activity coefficients in water".

Reference is made to activity coefficients of 10^3 to 10^7 for n-esters, n-ethers, n-chlorides, n-alkylbenzenes among others (see page 362). According to the article, experiments have been made on waste water samples from industry. The pH of the samples was adjusted to 3,0 or lower (see page 368, column 2).

Table 6 contains the percentage removal of each of 11 species (see page 371). Finally, Thibodeaux repeats that air stripping is another unit process capable of removing even organics with relatively low vapour pressure. He adds that, in fact there are probably many industrial treatment systems in which organics are actually being air stripped from the waste water, although their removal is erroneously attributed to biological oxidation (see page 374). When read in context with the introductory statement

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that air stripping had been demonstrated to be a feasible technique for removing a portion of the organics from waste water (page 358, sentence bridging columns 1 and 2), at a first glance, it would appear that Thibodeaux's article gives to its reader all the instructions necessary to start experimentation in every water contamination situation.

- 9.2 Nevertheless, the Board is of the opinion that a careful examination of this article leads to a quite different teaching to the man skilled in the art.

Even if Thibodeaux's article gives a general literature survey of organic pollutant constituents, no removal of the contaminants defined in the claim is reported.

One reference is cited reporting 99.9% efficiencies in removing phenolic type compounds, but it is added that it was the result of bio-oxidation and that the reference did not mention the role of air stripping (page 358, column 2, last 4 lines).

In Table I (page 362), the compounds 10 to 20 comprise, according to the author, a representative cross section of industrial organic chemicals in waste water. It must be admitted that all these considered pollutants have a normal boiling point clearly lower than 200°C, phenol being the last, with the highest boiling point (181.4°C). Consequently, it must be concluded that air stripping has not been examined for contaminants having boiling points in excess of 200°C. However, some relation is clearly disclosed between the normal boiling point, on the one hand, and Henry's Law constant and relative volatility of the organic compounds, on the other hand.

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From Table 6 (page 371) which gives the percentage removals by air stripping for compounds 10 to 20, the skilled man can appreciate a drop in the percentage removal down to 5.4 for the phenol, which is the quoted compound with the highest boiling point (181.4°C).

Taking these facts into consideration, the Board is not inclined to give much weight to the accompanying comments to the tables.

- 9.3 It must be stressed that, due to the undesirability (for instance the toxicity) of the contaminants, only an efficient process could be considered as an alternative to the known and proved process (99.9% for phenolic type compounds by biooxidation). Although it is true that the skilled man could have tried air stripping for all sorts of contaminants, it appears clearly that faced with the existing problem, he could not have expected satisfactory results in view of the teaching of "Thibodeaux's article" (5.4% for phenol, see Table 6, page 371).

Consequently, in view of the existence of other promising methods, the skilled man would have refrained from such an attempt. Nevertheless, the appellants have shown that, under the conditions of Claim 1 of the application, an efficient recovery of compounds boiling above 200°C is obtained. The percentage recoveries lie between 46 and 98 for 15 compounds listed in Table II of the application. For PCP, 99% removal was obtained (Example 1). This is a quite unexpected result in view of the fact that phenol, boiling above 100°C (lower than PCP) was removed by air stripping only to the extent of 5.4%.

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The Board sees in the specific data in "Thibodeaux's article" a strong disincentive to using the stripping method for removal of contaminants boiling above 200°C when other proven methods were available. Among the generally known purifying methods the quoted prior art specifically mentions biooxidation ("Thibodeaux's article") and carbon adsorption ("K. Uede abstract").

10. In this context, it is interesting to compare this conclusion with the independent conclusions reported in the "USEPA Report" submitted by the appellants with their letter received on 10 December 1985 (see page 5, III(2)1., and Appendix A) which supports the position of the Board. As this report was not available to the public before the priority date but only in the priority interval, this document is not part of the state of the art. Nevertheless, it can be considered as an illustration of the thought of experts in the art at the relevant date.
11. Having considered the facts, the Board comes to the conclusion that the solution of the existing problem was not obvious to a skilled person because of a clear disincentive in the teaching of Thibodeaux, particularly in relation with the necessary efficiency in removing the contaminants covered by Claim 1.

Thus the process claimed in Claim 1 must be considered as not only new but also inventive. In view of the fact that the technique of vapor stripping of the contaminants from the contaminated water was not obvious, the question can be left open whether or not other features specified in Claim 1 contribute to the inventive step of its subject-matter. The subject-matter of Claims 2 to 7, which are fully dependent on the main claim are supported by the patentability of the subject-matter of the main claim.

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12. The Board cannot therefore agree with the grounds and conclusions of the impugned decision of the first instance. However, the patent applied for cannot be granted at present as the description in the specification has not yet been brought into line with the claims as amended and does not indicate the useful background art (Rule 27 CBE).

Order

1. The decision under appeal is set aside.
2. The case is remitted to the Examining Division for further prosecution on the basis of Claims 1-7, submitted during oral proceedings.

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