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Aktenzeichen / Case Number / N^o du recours : T 131/87 - 3.3.2

Anmeldenummer / Filing No / N^o de la demande : 81 104 045.0

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Bezeichnung der Erfindung: Electrical device containing a halogenated
Title of invention: dielectric fluid
Titre de l'invention :

Klassifikation / Classification / Classement : H01B 3/18

ENTSCHEIDUNG / DECISION
vom / of / du 7 September 1989

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Occidental Electrochemicals Corporation

Einsprechender / Opponent / Opposant :

Wacker-Chemie GmbH

Stichwort / Headword / Référence : Dielectric fluid/OCCIDENTAL ELECTROCHEMICALS

EPÜ / EPC / CBE Art. 56 EPC

Schlagwort / Keyword / Mot clé : "Inventive step (yes) - broadness of the claims
as initially granted not justified"

Leitsatz / Headnote / Sommaire

Europäisches
Patentamt

Beschwerdekammern

European Patent
Office

Boards of Appeal

Office européen
des brevets

Chambres de recours



Case Number : T 131 /87 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal
of 7 September 1989

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Decision under appeal : **Decision of Opposition Division of the European
Patent Office dated 21 January 1989 rejecting
the opposition filed against European patent
No. 81 104 045.0 pursuant to Article 102(2) EPC.**

Composition of the Board :

Chairman : P. Lançon
Members : A.J. Nuss
 R. Schulte

Summary of Facts and Submissions

- I. European patent No. 0 041 220 was granted on 22 May 1985 with five claims in response to the European patent application No. 81 104 045.0, filed on 26 May 1981.

The Claims of the patent as granted read as follows:

- "1. Dielectric fluid composition containing halogenated unsaturated hydrocarbons characterized in a composition which is resistant to decomposition at elevated temperatures and the presence of oxygen comprising perchloroethylene containing less than about 0.005 percent of chlorinated ethanes, and an effectively stabilizing amount of an antioxidant.
5. An apparatus comprising an electrical device and a dielectric fluid composition according to any one of the Claims 1, 2 or 3."

- II. The Appellant (Opponent) filed notice of opposition against the European patent on 21 February 1986, requesting revocation of the patent on the ground that its object lacked inventive step in view of the following documents:

- (1) US-A-3 976 705
- (2) CH-A-412 856
- (3) DE-A-2 449 667
- (4) DE-A-1 618 950
- (5) DE-A-2 811 779
- (6) DE-A-2 627 989
- (7) US-A-2 019 338
- (8) IEEE Publication No. 79CH1510-7 by R.H. Hollister, C.L. Moore and P. Voytik.

- III. In a decision dated 21 January 1987, the Opposition Division rejected the opposition and maintained the patent unamended. The Opposition Division took inter alia, the view that the invention as claimed involved an inventive step since these eight documents did not render obvious the claimed subject-matter and that, in addition, the existence of an inventive step was further supported by the generally improved stability as shown by the acidity levels given in Tables I and II of the description.
- IV. The Appellant lodged an appeal against this decision in a letter filed on 20 March 1987, setting out in detail the grounds of appeal and paid the appeal fee at the same time.
- V. The parties having exchanged written submissions, oral proceedings took place on 7 September 1989 at the request of both parties, which had been informed previously by the Board in a communication dated 7 July 1989 that in view of the results provided in Table II of Example 2 of the patent in suit, only a combination of perchloroethylene containing less than about 0.005% by weight of total chlorinated ethanes with a specific stabiliser combination, viz. a mixture of N-methyl pyrrole and p-tertiary amylphenol, seemed to lead to a dielectric fluid having improved resistance to decomposition at high temperatures in the presence of oxygen.

At the end of the present appeal proceedings, the differing positions of the parties appeared to be essentially as described hereinafter.

- (i) After having dropped at the beginning of the oral proceedings the novelty objection raised initially in its letter of appeal, the Appellant finally considered that the invention as claimed did not involve an inventive step

since all relevant features were already known from the cited documents. The principal arguments put forward by the Appellant were the following:

- The use of stabilised perchloroethylene as transformer liquid was known from document (8).

- Document (1) disclosed perchloroethylene having the purity required in the patent in suit, i.e. containing less than 50ppm of chlorinated ethanes. Since it was however known from document (7) that chlorinated saturated hydrocarbons were less stable than unsaturated ones when used as a dielectric material, the suitability of the perchloroethylene described in document (1) as dielectric fluid was self-evident and made superfluous any specific mention in respect of the desirability to use highly pure perchloroethylene for such use.

- Documents (2) to (6) disclosed the stabilisation of chlorinated hydrocarbons such as perchloroethylene by addition of various antioxidants, whereby documents (3) and (6) were considered as the most relevant in view of the fact that both rendered obvious not only the specific combination of N-methyl pyrrole and p-tertiary amylphenol indicated in the patent in suit, but also the superior stabilising effect when combining stabilisers. Moreover, like in the patent in suit the comparisons in document (3) showed that N-methyl morpholine was not a powerful stabiliser for perchloroethylene.

- A maximum acidity level of 690ppm as shown in Table II of the patent in suit clearly indicated that the use of perchloroethylene with the claimed purity of less than 50ppm of chlorinated ethanes did not allow to avoid its decomposition, since the remaining impurities alone would only lead to a maximum acidity of 46.6ppm.

- (ii) The Respondent (Proprietor of the patent) argued that the claimed composition surprisingly provided an extremely stable perchloroethylene which will not decompose for many years. This had by no means been obvious to the man skilled in the art, even if he had known the references cited because they belonged to three different categories. Documents (1) to (6) were concerned with the stabilisation of chlorinated hydrocarbons for quite different purposes, whereas only documents (7) and (8) were directed to use of perchloroethylene as a dielectric fluid. Furthermore, document (1) only described how to prevent catalytic decomposition of perchloroethylene due to the presence of aluminium chloride at the stage of production. It was also known that such catalytic decomposition occurred in the presence of iron. It could therefore not be gathered from these references that it was just the amount of saturated chlorinated hydrocarbons which caused the instability of perchloroethylene and that the elimination of this impurity would lead to very stable solutions.

The Appellant's acidity calculations were considered by the Respondent as support for the presence of an inventive step all the more, as neither document (7) nor document (8) suggested that impurifications in chlorinated olefins could cause catalytic decomposition of saturated chlorinated compounds. The Respondent further argued that document (8) even mentioned that transformer oil, i.e. a mixture of perchloroethylene and oil had improved properties in comparison to perchloroethylene alone and that the products described in documents (7) and (8) lacked sufficient stability to be used as dielectric fluid in practice.

- VI. The Appellant requested that the decision under appeal be set aside and that the patent be revoked.

The Respondent requested that the patent be maintained as granted or according to the auxiliary request submitted during oral proceedings on the basis of amended claims and description.

Claim 1 of the auxiliary request reads as follows:

"1. Dielectric fluid composition containing halogenated unsaturated hydrocarbons characterized in a composition which is resistant to decomposition at elevated temperatures and the presence of oxygen comprising perchloroethylene containing less than about 0.005 percent of chlorinated ethanes, and an effectively stabilizing amount of antioxidant being a mixture of N-methyl pyrrole and p-tertiary amylphenol."

Dependent Claims 2 to 4 correspond to Claims 3 to 5 as granted.

Reasons for the Decision

1. The appeal complies with the requirements of Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. Claim 1 of the auxiliary request causes no objection on formal grounds, since it is the result of a combination of subject-matter disclosed in Claims 1 and 2 as granted, which are adequately supported by the claims and description as originally filed (see Claims 1, 5, 10 to 13, 15 and page 8, lines 1 to 3 of the application as filed) and, in addition, manifestly do not broaden the main claim as granted. This is not contested by the Appellant.

The remaining claims of the auxiliary request correspond to Claims 3 to 5 as granted, which had been adequately renumbered in consequence of said amendment.

The amended claims therefore comply with Articles 123(2) and (3) EPC.

3. The patent in suit is concerned with a dielectric fluid composition containing halogenated unsaturated hydrocarbons to be used in an apparatus comprising an electrical device such as a transformer or a power capacitor.

- 4.1 Document (8) is to be considered as closest state of the art. It relates to a project which was conducted to develop a substitute transformer as a replacement for the PCB transformer at a desirable cost level. As the project progressed, it became apparent that a lower cost cooling and dielectric liquid had to be developed to replace the relatively expensive fluorocarbon liquid (C₈F₁₆O) that was chosen for the first prototype unit. Thus, a search was begun for suitable lower cost, fire resistant chlorofluoro or chlorinated liquids knowing that perchloroethylene had already previously been mentioned as a promising liquid and that it is considered to be one of the most stable of chlorinated solvents.

Work with perchloroethylene has dealt with the thermal and chemical stability thereof, at temperatures up to 175°C, and its compatibility with other transformer materials such as transformer oil. Thermal aging tests have been conducted with stabilised as well as unstabilised perchloroethylene, of which a special grade had been tested. In particular, cooling tests on models have shown that perchloroethylene is more effective as a coolant than other liquids such as transformer oils (see page 239, left

column, fourth and last paragraph; page 239, right column, second paragraph to page 240, right column, first paragraph, and page 241, left column, first full paragraph after Table III).

4.2 As stated in the introductory part of the patent, it is most important that a satisfactory dielectric fluid must have excellent resistance to decomposition over long periods of time under severe operational conditions. In particular, the dielectric fluid must not decompose at elevated temperatures in the presence of oxygen to form electrically conductive or corrosive materials (see page 2, lines 11 to 14 and lines 41 to 47 of the description).

5. The technical problem vis-à-vis document (8) consisted thus in providing a dielectric fluid having improved resistance to decomposition at elevated temperatures in the presence of oxygen.

In order to solve this problem Claim 1 of the patent as granted proposes perchloroethylene containing less than about 0.005% of chlorinated ethanes, and an effectively stabilising amount of an antioxidant.

In view of the acidity levels given in Table II of the patent in suit for dielectric fluids including such containing commonly used stabilisers (see Example 2 on page 5 of the description), the problem appears to be solved by the composition presented in the patent in suit as the preferred dielectric fluid, viz. a combination of N-methyl pyrrole and p-tertiary amyphenol with perchloroethylene containing less than 0.005% chlorinated ethanes.

6. In the absence of any document which discloses all the features of the dielectric fluid composition according to the main as well as the auxiliary request, the claimed subject-matter is novel. This was no longer contested by the Appellant at the oral proceedings (see paragraph V(i) above).

7. It remains, therefore, to be examined whether the requirement for inventive step is met.

- 7.1 Document (8) reflects considerations made some seven months before the priority date of the patent in suit on an economic replacement for PCB transformers with emphasis on suitable cost reducing dielectric transformer liquids, whereby not only perchloroethylene, but also stabilised perchloroethylene had been considered as possible alternatives for which thermal aging tests had been made among others.

It is true that this document does not mention any specific stabiliser or stabiliser combination. In the opinion of the Board, however, the man skilled in the art would not have ignored the concrete suggestion to try stabilised perchloroethylene as a transformer liquid, because utilisation under severe operational conditions is expected to lead to degradation of the dielectric fluid due principally to the relatively high working temperatures (see paragraph 4.2 above), a phenomenon which is frequently encountered with perchloroethylene as may be seen from documents (3) and (6), which both review various stabilisers against oxidative degradation due, in particular, to heat (see document (3), page 1(3), first paragraph to page 2(4), third paragraph and claims; document (6), page 1(3), first paragraph to page 3(5), second paragraph).

The man skilled in the art had thus no reason to discard the suggestion made in document (8) for being too abstract.

- 7.2 The teachings from both document (2) and (6) show that stabilisation of unsaturated chlorinated hydrocarbons such as perchloroethylene by adding a single stabiliser or a combination of stabilising compounds may lead to more or less satisfying results and that in particular single compounds such as N-alkyl pyrroles do not really provide sufficient resistance to oxidative degradation. Stabilisers containing several stabilising compounds may require too much product or lead to incompatibilities (see document (6), page 2(4), paragraphs 1 to 4 and document (2), page 1, lines 22 to 29).

From document (3) it is in particular known that stabilisers which have an excellent stabilising action over long periods in combination with a specific chlorinated hydrocarbon, do not necessarily show the same efficiency with other chlorinated hydrocarbons and that the stabilising effect must therefore be of a particular specific nature. This document thus proposes to use as a specific stabiliser for perchloroethylene a mixture of N-alkyl morpholine and alkyl phenol, optionally in combination with N-methyl pyrrole (see page 2(4), paragraph 2 to page 3(5), paragraph 1).

Therefore, when trying to find a dielectric fluid with improved resistance to degradation, the man skilled in the art, i.e. the chemist in the present case, was aware that the efficiency of a stabiliser depends largely on the nature of the chlorinated hydrocarbon to which it is added and that, therefore, a satisfactory stabilising effect obtained in combination with perchloroethylene is not automatically transposable to other chlorinated hydro-

carbons. Consequently, the man skilled in the art would certainly be reluctant to use for dielectric uses a perchloroethylene grade containing significant amounts of other chlorinated hydrocarbons liable to impair the desired stabilising effect.

- 7.3 In the opinion of the Board, it is not unreasonable to expect from the man skilled in the art in that situation to do what is normally done in the field of chemistry when side reactions are to be expected from undesired impurities in a chemical agent, viz. to turn first of all towards higher purity grades, all the more in document (8) allusion is made to a "special grade" of perchloroethylene. However vague this information might appear to be when considered isolated, it does make sense in view of the preceding considerations since such information must be read with the eyes of the man skilled in the art, who is aware of not only what is disclosed in documents (3) and (6), but of course also of the existence of different grades of perchloroethylene mentioned in document (1), from which it is in particular known to produce a high purity grade perchloroethylene with a concentration of undesired contaminants, i.e. saturated chlorinated hydrocarbons such as unsymmetrical tetrachloroethane and 1,1,2-trichloroethane, below detectable limits, i.e. 50ppm (see in particular column 1, lines 17 to 24 and lines 43 to 64; column 2, lines 27 to 41).

The man skilled in the art would immediately have realised that in removing the saturated chlorinated hydrocarbons from perchloroethylene exactly those compounds had been eliminated in document (1) which are unsuitable for dielectric use because of their chemical instability, since this is known in the art for a long time as documented by document (7) published in 1935 (see page 1, left column, lines 18 to 33).

7.4 Although these considerations show that the man skilled in the art would have chosen as dielectric fluid perchloroethylene containing less than 50ppm of chlorinated saturated hydrocarbons, it is however clear from what is stated in paragraph 7.2 above that the resistance to degradation of perchloroethylene actually depends on the stabilising capacity of the stabiliser added and that neither a single stabiliser, nor a combination of several stabilising compounds may necessarily provide satisfying protection against thermally induced oxidative degradation. Moreover, the great number of stabilisers described in documents (2) to (6) provides the impression of a permanent need for improving the stability of all sorts of stabilisers for unsaturated chlorinated hydrocarbons, in particular, perchloroethylene. This is certainly an indication that really satisfying stabilisers had not been found in the past, although the intention had always been to find stabilisers superior to previous ones.

The testing methods for thermal stability used in the different documents of the state of the art including document (1), are all different from the one used in the patent in suit. Therefore, the stabilising capacity of the known stabilisers or stabiliser compositions in the simulated operating environment of an electrical transformer, as done in the patent in suit, cannot be derived from the prior art documents, with the consequence that the only true comparative tests available with commonly used stabilisers are those of Example 2 of the patent in suit. These uncontested results were made to display a dielectric fluid with improved resistance to decomposition at elevated temperatures in the presence of oxygen when compared to perchloroethylene containing commonly used stabilisers under similar conditions (see page 5, lines 2/3 and lines 63 and 64 of the patent in

suit). As explained in the patent in suit, this means that the dielectric fluid must not decompose over long periods (30 years) to form electrically conductive or corrosive materials, since in particular dehydrochlorination results in the formation of hydrogen chloride, with deleterious effects on the dielectric fluid and the electrical device used (see page 1, lines 11 to 14 and lines 56/57). As easily predictable from the foregoing, this goal cannot be expected to be reached by all stabilisers or combinations of stabilisers.

- 7.5 The results shown in Table II of Example 2 of the patent in suit indeed confirm that on the whole significant acidity levels are already reached after 5 days (corresponds roughly to 7 1/2 years in a transformer application - see Example 1 on page 3), at 175°C in the presence of 10% air in the headspace of the testing device and that only in one case with an acidity of merely 0.5ppm HCl an excellent resistance to decomposition is shown, viz. in the case of a specific stabiliser which is a mixture of N-methyl pyrrole and p-tertiary amyphenol (pentaphen), which manifestly may lead to similar good results even in the presence of extremes of 25 to 50% air (see Table I on page 4).

The requirement in the patent in suit for outstanding stability in respect to formation of hydrogen chloride is certainly not met in the case of commonly used stabilisers, such as methacrylonitrile, leading to acidity levels in the tested dielectric fluid up to 690ppm HCl by weight, after only 1/4 of the expected life time of the dielectric fluid. As Respondent's uncontested calculations show, merely at most 46.6ppm HCl is formed by decomposition of the remaining impurities, i.e. chlorinated ethanes, so that the rest of the acid must inevitably have been formed by decomposition of the perchloroethylene

itself. This is the case for 13 results out of a total of 23 indicated in Table II for 22 different stabilisers, which represents a proportion of 56.5%. In more than a third of all cases (34.8%) an acidity of 95ppm HCl is indicated, which is more than twice the theoretical level for the remaining impurities. There is thus no reason to believe that it is actually the absence of impurities in the dielectric fluid which leads to a perchloroethylene liable to be more effectively stabilised as hitherto over long periods of time against decomposition. On the contrary, the results in Table II confirm that the stability finally obtained depends above all on the efficiency of the individual stabiliser.

- 7.6 The superior performance of the N-methyl pyrrole/pentaphen stabiliser combination over 22 usual stabilisers becomes clearly apparent when compared to methacrylonitrile which leads to an acidity which is 1380-fold more important after only a 1/4 of the expected lifetime of the stabiliser composition.

It is certainly true that the man skilled in the art would have considered both N-methyl pyrrole and pentaphen (i.e. p-tertiary amyphenol) to be stabilisers in the broadest possible sense of such a definition in view of what is said in the most relevant documents (3) and (6). However, none of these documents suggests neither a combined stabiliser limited to these two compounds alone, nor that any significant improvement could be expected from such a combination:

- Document (3) discloses a stabiliser for perchloroethylene obtained by combining N-alkyl morpholine (e.g. N-methyl morpholine) with an alkylphenol (branched alkyl with 3 to 5 carbon atoms in p-position is preferred), and which in addition may contain N-methyl pyrrole for

further improving the stabilising effect, whereby especially good results were predicted for p-tert.butylphenol and N-methyl (or N-ethyl) morpholines (see page 2(4), second to last paragraph to page 3(5), second paragraph). There is thus no reason to believe that without one of the essential components of the stabilising composition, viz. N-alkyl morpholine, outstanding stability could be expected nevertheless.

- Document (6) concerns the stabilisation of trichloro- and/or perchloroethylene by a composition containing at least a nitroalkane in combination with at least a pyrrole derivate and at least a phenolic compound. This stabiliser is however said to be especially suitable for trichloroethylene (see Claim 1 and page 4(6), second to last paragraph). The preceding remark thus necessarily also applies here.

8. It follows from all this, that unexpected stability has only been established in the case of high purity perchloroethylene stabilised with a mixture of N-methyl pyrrole and p-tertiary amyphenol. Since altogether 22 reference stabilisers could not confer the required stability to perchloroethylene when used as a dielectric fluid, it is not credible that any other stabiliser not included in that list could well solve the technical problem as stated above. Under these circumstances, there is manifestly no basis for considering that the improvement obtained in the case of the specific combination of N-methyl pyrrole and p-tertiary amyphenol is readily transposable to the whole class of stabilisers. The Board is therefore not prepared to accept that the problem to provide a dielectric fluid with improved resistance to decomposition may be solved in a general way by combining perchloroethylene containing less than 0.005% of chlorinated ethanes with any stabiliser.

Consequently, an inventive step cannot be recognised for a dielectric fluid composition as defined in Claim 1 of the patent as granted, but only for the composition according to claim 1 of the auxiliary request in which the anti-oxidant stabiliser is limited to a mixture of N-methyl pyrrole and p-tertiary amyphenol.

9. For both requests, the above reasons against or in favour of an inventive step based on an unexpected improvement equally apply to the other (independent) claims concerning the use of the claimed dielectric fluid in electrical devices (transformers and power capacitors) as well as to an apparatus comprising an electrical device and a dielectric fluid composition as claimed.

The allowability of the dependent claim(s) depend(s) on the fate of the independent claim(s).

Therefore, only the claims of the auxiliary request are allowable.

For the rest, there are no objections to the description which has been brought into accordance with the amended scope of protection in connection with Respondent's auxiliary request.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.
2. The main request is refused.

The case is remitted to the first instance with the order to maintain the patent in amended form on the basis of the following documents:

- Claims 1 to 4 of Respondent's auxiliary request;
- description pages 1, 4 and 6, as granted and amended,
description pages 2, 3 and 5 (auxiliary request).

The Registrar:

The Chairman:

M. Beer

P. Lançon