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Bezeichnung der Erfindung:
Title of invention:
Titre de l'invention : Stabilisation of 1,1,1-trichloroethane

Klassifikation / Classification / Classement : C07C 17/42

ENTSCHEIDUNG / DECISION

vom / of / du 1 December 1989

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet : Imperial Chemical Industries PLC

Einsprechender / Opponent / Opposant : Solvay & Cie

Stichwort / Headword / Référence : Stabilisation/ICI

EPÜ / EPC / CBE Article 56 EPC

Schlagwort / Keyword / Mot clé : "Inventive step (confirmed) - after amendment"

Leitsatz / Headnote / Sommaire

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Case Number : T 573 /88 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 1 December 1989

Appellant : (Proprietor of the patent)
Imperial Chemical Industries PLC
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Representative :
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Respondent : (Opponent)
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Representative :

Decision under appeal : Decision of Opposition Division of the European Patent Office dated 21 September 1988 revoking European patent No. 0 024 113 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
C.V. Payraudeau

Summary of Facts and Submissions

I. The mention of the grant of European patent No. 0 024 113 in respect of European patent application No. 80 302 376.1 filed on 15 July 1980 and claiming priority of 25 July 1979 and 25 April 1980 from two prior applications filed in the United Kingdom, was announced on 9 November 1983 (cf. Bulletin 83/45).

II. On 9 July 1984 a notice of opposition was filed requesting the revocation of the patent on the ground that its subject-matter did not involve an inventive step. The opposition was supported, inter alia, by the following documents:

(1) JP-B-67-25 641 (English translation)
(2) JP-B-70-26 483 (English translation)
(3) US-A-3 974 230
(4) GB-A-1 435 548.

The Patentee referred to US-A.3 549 715 (5). However, the Opposition Division refused to consider this document, since it was filed at a very late stage in the opposition proceedings.

III. By a decision dated 21 September 1988, the Opposition Division revoked the European patent. In the light of the combined teaching of documents (1) to (4) and common general knowledge, the Opposition Division considered it was obvious to replace the 3-methyl-1-butyn-3-ol (MBy) in the stabilising compositions disclosed in document (3) by t-butanol (TBA). Furthermore, since it was known that binary mixtures of (TBA) with a nitroalkane, such as nitromethane (NM) or nitroethane (NE), stabilise 1,1,1-trichloroethane, it was obvious to use mixtures of nitro-

alkanes with TBA, particularly as mixtures of nitroalkene with MBy or MBy and t-amyl alcohol (TAA) are disclosed in document (3) for the same purpose. In the Opposition Division's opinion the identification of specific amounts and relative proportions of the stabiliser components could not be regarded as involving an inventive step.

The Opposition Division also concluded that it had not been established that the claimed ternary compositions are more effective in stabilising 1,1,1-trichloroethane than the closest binary compositions of the prior art. Furthermore, the Opposition Division did not accept that it was surprising that the claimed ternary mixtures are as effective as the binary mixtures known in the art.

IV. A notice of appeal was lodged against this decision by a duly confirmed telex on 21 November 1988 and the prescribed fee paid in due time.

In his Statement of Grounds of Appeal filed on 23 January 1989, the Appellant argued that the Opposition Division had erred in concluding that there is nothing unexpected in the fact that ternary mixtures TBA, NM and NE or nitropropane (NP) are as effective as binary mixtures of TBA and NM (or NE) in the stabilisation of 1,1,1-trichloroethane, since, in the Appellant's view, it was common general knowledge that, even in admixture with TBA, the nitroalkanes, are not equally effective and they exhibit the same order of effectiveness as do the nitroalkanes used alone viz NM>NE>NP. Therefore, it must be expected in respect of mixtures with TBA that replacing a part of the NM by NE or NP would lead to a loss of stabiliser effectiveness. That this does not happen can only be regarded as surprising and the invention cannot be considered to be obvious.

The Appellant filed experimental evidence confirming the above-mentioned general knowledge.

V. In reply to a communication from the Rapporteur, the Appellant filed new claims of restricted scope on 31 October 1989. Independent Claims 1, 8, 10 and 11 of this amended statement of claim reads as follows:

"1. A solvent composition comprising 1,1,1-trichloroethane having a stabilising amount of a mixture of stabilising components dissolved therein, characterised in that the mixture of stabilising components comprises (i) tertiary butanol, (ii) nitromethane and (iii) 1- or 2-nitropropane or nitroethane in which the mole fractions per cent of the stabilising components are within the range encompassed by Contour M in Fig. 1 of the accompanying drawings or by Contour C in Fig. 2 of the accompanying drawings.

8. A mixture of stabilising components for the stabilisation of 1,1,1-trichloroethane, characterised in that the mixture comprises (i) tertiary butanol, (ii) nitromethane and (iii) 1- or 2-nitropropane or nitroethane wherein the mole fraction per cent of the stabilising components are within the ranges encompassed by Contour M in Fig. 1 of the drawings or by Contour C of Fig. 2 of the drawings.

10. A method of inhibiting decomposition of 1,1,1-trichloroethane by metals which comprises incorporating a mixture of stabilising components in the 1,1,1-trichloroethane, characterised in that the mixture of stabilising components is as claimed in Claim 8 or Claim 9.

11. A method of cleaning metal and other articles which comprises contacting the articles with a solvent composition or vapour thereof, characterised in that the

solvent composition is as claimed in any one of Claims 1 to 7."

VI. The Appellant requests that the decision under appeal be set aside and a patent maintained on the basis of Claims 1 to 11 filed on 31 October 1989.

The Opponent has neither replied to any of the official communications nor filed any requests in respect of the appeal.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.

2. There are no formal objections under Article 123 EPC to the present Claims 1 to 11 since they are supported by the original disclosure and do not extend the scope of protection conferred. Thus, current Claim 1 represents a combination of Claims 1, 4 and 5 as originally filed and granted, Claims 2 to 11 correspond to the originally filed and granted Claims 6 to 15 respectively.

3. The disputed patent relates to the stabilisation of 1,1,1-trichloroethane against corrosive attack by light metals, especially aluminium, occurring during the vapour degreasing of these metals. It is known in the art to stabilise 1,1,1-trichloroethane by adding a mixture of TBA and NM, and a commercially available stabilised 1,1,1-trichloroethane contains 70 mole fraction per cent of TBA and 30 mole fraction per cent of NM (cf. the undisputed statement of the Patentee in his letter filed on 16 October 1986 in the paragraph headed "Points 7, 8 and 9").

3.1 In the light of this prior art, the technical problem underlying the patent in suit may be seen in providing alternative solvent compositions comprising 1,1,1-trichloroethane having stabilising components dissolved therein which are approximately as effectively stabilised as this known composition.

According to the disputed patent this technical problem is essentially solved by compositions comprising 1,1,1-trichloroethane having dissolved therein stabilising amounts of (i) TBA, (ii) NM and (iii) NE or NP in which the mole fraction per cent of the stabilising components are within the ranges encompassed by Contours M and C in Figs. 1 and 2 of the drawings respectively.

3.2 According to the disputed patent the effectiveness of stabilisers for 1,1,1-trichloroethane may be determined by subjecting the stabilised compositions to an accelerated stability test. This test involves adding dry aluminium particles, which have been degreased by boiling trichloroethylene, pickled with nitric acid and washed with water, to the stabilised composition. The mixture is vigorously agitated and then allowed to stand. After filtering, the filtrate is analysed to determine the colour developed (in Hazen Units) on a Lovibond 1000 Nesslerizer, against a standard which is pure (unstabilised) 1,1,1-trichloroethane (cf. disputed patent page 4, lines 21 to 28).

3.3 From Fig. 2 filed by the Patentee on 18 September 1985, it can be deduced that if the known composition containing 1,1,1-trichloroethane, 70 mole fraction per cent TBA and 30 mole fraction per cent NM were to be subjected to this accelerated stability test, it would have a colour between 15 and 35 Hazen Units.

In the light of the results obtained for mixtures H I and J (cf. Table III of the disputed patent) and mixture F (cf. Table V of the disputed patent) the Board is satisfied that the above-defined technical problem is plausibly solved. Having regard to the nature of the test method, the Board considers that the values 20, 15, 65 and 10 Hazen Units for mixtures H, I, J of Table III and mixture F of Table V indicated that the present stabilisers are at least as effective as the known mixture of TBA and NM.

4. After examination of the cited documents, the Board has concluded that the claimed subject-matter is novel. Since novelty is not in dispute it is not necessary to consider this matter in detail.

5. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.

5.1 Document (3) discloses solvent compositions comprising 1,1,1-trichloroethane and specified amounts of MBy or mixtures of MBy and TAA and NM or mixtures of NM and NE. Additionally the compositions contain an alkylene oxide having 4 to 6 carbon atoms, including cyclohexene oxide, as an acid acceptor (cf. Claim 1).

From the data presented in Tables I, II and III, it is concluded in this document that mixtures of MBy and TAA are better stabilisers than equal concentrations of MBy or TAA alone; mixtures of MBy and NM or TAA and NM are more effective than MBy or TAA or NM alone at equal concentrations; and addition of NM to a mixture of MBy and TAA gives improved stabilisation (cf. column 6, lines 45 to 51).

However, with respect to stabilising compositions containing mixtures of NM and NE, this document only specifically discloses the two nitroalkanes in admixture with MBy (cf. last three compositions in Table III and five compositions in Table IV). Thus, there are no specific disclosures from which the skilled person could draw any conclusions regarding the stabilising ability of compositions containing NM, NE and TAA, the alcohol which is more closely structurally related to TBA than MBy. From the results obtained with stabiliser compositions containing MBy, NM and NE, particularly the failure of these compositions in the aluminium hot scratch test (cf. Table III, column 6) the skilled person would not be encouraged to consider using mixtures of nitroalkanes in place of NM.

Therefore the disclosure of this document, either alone or combined with that of the other cited documents, would not have provided the skilled person with any indication that the solution to the technical problem underlying the disputed patent lay in the use of mixtures of TBA, NM and NE or NP in the proportions defined by Contours M and C of Figs. 1 and 2 respectively.

5.2 Document (1) discloses that 1,1,1-trichloroethane is stabilised by the addition of TBA, an aliphatic nitro compound and a vic-epoxide (cf. Claim 1 on page 5 of the English translation). Suitable aliphalic nitro compounds and vic-epoxides are NM and NE, 1,2-epoxybutane and 2,3-epoxypentane (cf. Table I).

In the absence of any suggestion in this document of the use of mixtures of nitroalkanes it would not be of any assistance to the skilled person seeking a solution to the present technical problem.

5.3 Document (2) discloses that the stability of 1,1,1-trichloroethane is improved by the addition of a saturated monohydric tertiary alcohol, such as t-butyl alcohol or t-amyl alcohol, an aliphatic carboxylic ester of not more than 6 carbon atoms, for example ethyl or butyl acetate and a nitroalkane of not more than 3 carbon atoms, such as nitromethane or nitroethane (of Claim 1 and the Table on page 5 of the English translation). This document clearly teaches the use of ternary mixtures containing a single nitroalkane (cf. page 3, lines 11 to 16) and would not provide the skilled with any incentive to investigate the ability of mixtures of TBA, NM and NE or NP to stabilise 1,1,1-trichloroethane.

5.4 Document (4) discloses that 1,1,1-trichloroethane is stabilised against metal induced degradation by specified amounts of a stabilising agent containing a) a nitroalkane having 1 to 3 carbon atoms or a mixture of nitroalkanes; b) a monoepoxide or monochloro-monoepoxide each having 3 to 8 carbon atoms or a mixture of epoxides and c) a ternary mixture containing dioxane and two component mixtures selected from combinations of 1,1-dimethoxy-ethane, dimethoxymethane, methyl ethyl ketone, dioxene, dioxolane, trioxane, NM and TBA in the proportions within the shaded area of Figs. 1 to 20 (cf. Claim 1). According to Figs. 3, 5, 6, 8, 10, 16 and 19 TBA forms one component of these ternary mixtures. Thus, this document broadly envisages the stabilisation of 1,1,1-trichloroethane with a composition comprising TBA and a mixture of nitroalkanes containing 1 to 3 carbon atoms. However, the teaching of this document, particularly in view of the expressed preference for nitromethane (cf. page 2, line 24), would not suggest to the skilled person the combination of TBA, NM and NE or NP in the claimed proportions as a solution to the problem underlying the disputed patent.

5.5 Document (5) teaches that 1,1,1-trichloroethane is stabilised by incorporating therein an appropriate concentration of a nitroalkane, in particular NM (cf. column 1, lines 68 to 71 and column 4, lines 8 to 10). Claim 1 of this patent relates to composition comprising 1,1,1-trichloroethane containing, based on the weight of the 1,1,1-trichloroethane, about 0.5 to 10% of a nitroalkane having 1 to 3 carbon atoms and about 0.5 to 10% of a lower alkanol. However, this document is completely silent regarding the use of mixtures of nitroalkanes either alone or combined with a lower alkanol, to stabilise 1,1,1-trichloroethane.

Furthermore, from his common general knowledge (cf. also the experimental results reported in the Statement of Grounds of Appeal), the skilled person is aware that the effectiveness of NM, NE and NP when used alone to stabilise 1,1,1-trichloroethane decreases in the order NM>NE>NP. The above-mentioned experimental results also demonstrate that, in mixtures with TBA, the nitroalkanes are not equally effective and the order is the same as for nitroalkanes used alone, i.e. NM>NE>NP. Therefore, the skilled person would have expected that replacing part of the NM by NE or NP would lead to a loss of stabiliser effectiveness. The results for mixtures H, I and J in Table III and F in Table V show that this does not happen.

6. Therefore, in the Board's judgment the proposed solution to the above-defined technical problem is not obvious in the light of the cited prior art. The subject-matter of the present Claim 1 is patentable.

6.1 Dependent Claims 2 to 7, which relate to preferred embodiments of the compositions claimed in Claim 1, derive their patentability from this claim.

6.2 For the above reasons, Claims 8 and 9 and Claim 10, which, respectively, relate to mixtures comprising TBA, NM and NE or NP in the proportions defined in Claim 1 and the use thereof for inhibiting the decomposition of 1,1,1-trichloroethane and which are based on the same inventive concept, are also allowable.

6.3 Claim 11, which claims a method of cleaning metal and other articles by contacting them with the solvent compositions of Claims 1 to 7 or the vapours thereof, is allowable in view of the patentability of these claims.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent in amended form on the basis of Claims 1 to 11 filed on 31 October 1989 and a description and Figures which are in agreement with the amended statement of claim.

The Registrar: **The Chairman:**

M. Beer K. Jahn