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File No.: T 0946/92 - 3.3.1
Application No.: 84 301 880.5
Publication No.: 0 119 865
Classification: C07F 3/02
Title of invention: Hydrocarbon soluble dialkylmagnesium composition

D E C I S I O N
of 1 September 1993

Applicant:
Proprietor of the patent: Texas Alkyls Inc.
Opponent: Witco GmbH Gewerblicher Rechtsschutz

Headword: Dialkylmagnesiums/TEXAS ALKYLs
EPC: Art. 56
Keyword: "Inventive step (no); clear incentive in prior art"

Headnote
Catchwords



Case Number: T 0946/92 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 1 September 1993

Appellant: Witco GmbH
(Opponent) Gewerblicher Rechtsschutz
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D - 59180 Bergkamen (DE)

Representative:

Respondent: Texas Alkyls Inc.
(Proprietor of the patent) Westport
Connecticut 06881 (US)

Representative: Brekelmans, Paul Joahn Gérard
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office of 23 June 1992, posted on
13 August 1992, rejecting the opposition filed
against European patent No. 0 119 865 pursuant to
Article 102(2) EPC.

Composition of the Board:

Chairman: K.J.A. Jahn
Members: R.W. Andrews
J.A. Stephens-Ofner

Summary of Facts and Submissions

I. European patent No. 0 119 865 in respect of European patent application No. 84 301 880.5, which was filed on 20 March 1984, was granted on 2 November 1988 (cf. Bulletin 88/44) on the basis of eight claims. Independent Claims 1 and 6 read as follows:

"1. A hydrocarbon solution characterised in that it comprises:

(a) di-(n-butyl)magnesium;

(b) diethylmagnesium;

(c) at least one further dialkylmagnesium compound in which the alkyl groups are straight-chain containing from 5 to 15 carbon atoms; and

(d) a solvent selected from aliphatic, cycloaliphatic and aromatic hydrocarbons containing from 5 to 20 carbon atoms; components (a), (b) and (c) being present in respective molar amounts of: from 70 to 93 mole percent of di(n-butyl)magnesium; from 2 to 20 mole percent of diethylmagnesium; and from 5 to 25 mole percent of component (c).

6. A process for the production of a hydrocarbon solution as claimed in any of Claims 1 to 5 characterised in that it comprises:

(a) reacting, in the presence of a hydrocarbon solvent, magnesium metal with an alkyl halide in which the alkyl group is a straight-chain alkyl having from 5 to 15 carbon atoms;

- (b) either simultaneously with step (a) or subsequent thereto, reacting, in the presence of the solvent of step (a), further magnesium metal with an n-butyl halide;
- (c) either simultaneously with step (a) and/or step (b) or subsequent thereto, reacting with further magnesium metal, an ethyl halide forming a mixture of a hydrocarbon solution containing dialkylmagnesium compounds and undissolved solids; and
- (d) separating the hydrocarbon solution from the undissolved solids; all steps being conducted in a substantial absence of both moisture and oxygen."

II. A notice of opposition, which was filed on 13 July 1989, requested the revocation of the patent on that its subject-matter did not involve an inventive step. The opposition was supported by, *inter alia*, the following document:

(2) US-A-4 127 507.

III. By a decision delivered orally on 23 June 1992, with written reasons being issued on 13 August 1992, the Opposition Division rejected the opposition. The Opposition Division considered that, in the light of the closest prior art as represented by document (2), the technical problem underlying the disputed patent was to provide a hydrocarbon soluble dialkylmagnesium composition with a high magnesium content. The Opposition Division held that an increase in yield of soluble magnesium alkyl obtained by the addition of n-hexyl chloride was not to be expected. Therefore, the proposed solution to the said technical problem was not obvious.

IV. An appeal was lodged against the decision on 15 October 1992 with payment of the prescribed fee. In his Statement of Grounds of Appeal filed on 18 December 1992, the Appellant contended that the Opposition Division's conclusion that the passage on page 3, lines 23 to 25 of the disputed patent stating that it is known that alkylmagnesium compounds containing either branched-chain alkyl groups or straight-chain alkyl groups of five carbon atoms or more are effective as solubilising agent is internal state of the art was wrong since a similar passage occurred in column 3, lines 14 to 29 of US-A-4 207 207 (document (5)). Therefore, the Appellant argued that the alleged invention merely involved the application of this known solubilising effect of long chain dialkylmagnesium compounds to the butyl-ethylmagnesium system of document (2).

The Appellant further alleged that the increased solubilising effect can only be obtained in the presence of aluminium alkyl compounds and has sought to demonstrate this experimentally.

V. The Respondent argued that the passage referred to in document (5) gives no information whatsoever regarding the specific use of long chain dialkylmagnesium compounds or any reference to the solubilising effect on di-n-butylmagnesium in the ternary systems of the disputed patent.

In order to counter the Appellant's allegation regarding the necessary presence of alkylaluminium compounds the Respondent repeated Example 4 and obtained a 74.3% yield of soluble magnesiumalkyl.

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

The Respondent requests that the appeal be dismissed.

Neither party requested oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. The patent in suit relates to a solution comprising di-(n-butyl)magnesium, diethylmagnesium and a hydrocarbon solvent and a process for the preparation thereof. Document (2), which is considered to represent the closest state of the art, also discloses such solutions.

However, although a yield of hydrocarbon soluble dialkyl-magnesium of 70% could be obtained at a molar ratio of n-butyl chloride to ethyl chloride of 1:1 (cf. document (2), Examples 2 to 8), at molar ratios of n-butyl chloride to ethyl chloride of 2.7:1 and 8.1:1 the yields of hydrocarbon soluble dialkylmagnesium fell to 38% and 5% respectively (cf. Comparison Examples 3 and 5 of the disputed patent).

Therefore, the technical problem underlying the disputed patent is to provide a solution of di-(n-butyl)magnesium and diethylmagnesium in a hydrocarbon which contain, at yields of soluble dialkylmagnesium of approximately 70%, higher concentrations of di-(n-butyl)magnesium than the solution known from document (2).

According to the patent in suit this technical problem is essentially solved by adding 5 to 13 mole per cent of

at least one further dialkylmagnesium compound in which the alkyl groups are straight-chain containing from 5 to 15 carbon atoms to the mixture of di-(n-butyl)magnesium and diethylmagnesium.

In light of the results obtained in Examples 4, 6 and 7 of the disputed patent and the experimental data filed by the Respondent on 30 June 1993, the Board considers it plausible that the above-defined technical problem has been solved. In these experiments, yields of soluble dialkylmagnesium compounds ranging from 68 to 76% were obtained at molar ratios of n-butyl chloride to ethyl chloride of from 6.25:1 up to 46:1.

The Appellant maintained that the high yields of soluble dialkylmagnesium obtained in Examples 4, 6 and 7 were obtained by adding not only n-hexyl chloride but also tri-(n-octyl)aluminium to the compositions. This was based on Example 1 (Comparison Example) and the fact that Examples 3 to 7 were carried out using the same procedure described above. In Example 1, after the separation of the clear hydrocarbon phase, the remaining solids were solubilised by the addition of tri-(n-octyl)-aluminium.

The Board cannot accept this argument since it is clear from the wording of Example 1 that the tri-(n-octyl)aluminium was only added to the solids to enable an analysis to be carried out. The Board's position is further supported by the results of a repetition of Example 4 filed by the Respondent on 30 June 1993 from which it is abundantly clear that tri-(n-octyl)aluminium was not included in the composition.

The Appellant also supported his allegation that the technical problem as defined above had not been solved by the experimental results filed on 6 May 1991 and

18 December 1992. According to these experimental reports, two repetitions of Example 5 (comparative) and Example 6 gave yields of soluble dialkylmagnesium of 14% and 5% and 42% and 10% respectively. From the variations in the yields of soluble dialkylmagnesium obtained in all these experiments, it is clear that these yields are sensitive to changes in the conditions used in the preparation of the solutions. In these circumstances, the Board considers that a skilled person would be able to achieve the yields of soluble dialkylmagnesium of Examples 4, 6 and 7 of the disputed patent on the basis of the information provided by the patent in suit and his common general knowledge in a field of organometallic chemistry.

3. The only question to be decided in the present appeal is whether the subject-matter of the disputed patent involves an inventive step.

3.1 As mentioned above, document (2) discloses a solution comprising di-(n-butyl)magnesium and diethylmagnesium at a n-butyl to ethyl ratio of from about 0.25:1 to about 4:1 in a hydrocarbon solvent (cf. Claim 1). However, this document provides no indication of how the ratio of n-butyl to ethyl in the solution may be increased.

3.2 After commenting on the presumed reason for the general insolubility of straight-chain lower alkyl magnesium compounds, including those having up to 4 carbon atoms (cf. column 1, line 30 to 35 in combination with column 3, lines 3 to 13), document (5), which discloses a solution comprising di-(n-propyl)magnesium and dimethylmagnesium in a hydrocarbon solvent (cf. Claim 1), goes on to state that it is known that dialkylmagnesium compounds containing either straight-chain alkyl groups of five carbon atoms or more or

branched-chain alkyl groups of any length are effective as solubilising agents (cf. column 3, lines 14 to 29).

This statement in a document published in 1980 renders invalid the Opposition Division's argument that a similar statement on page 3, lines 17 to 29 of the disputed patent represented "internal" prior art which had not been published before the filing date of the patent in suit.

Although this statement is of a very general nature, nevertheless, in the Board's judgment, it would provide a sufficient incentive for the skilled person to investigate the solubilising effect of such longer chain alkylmagnesium compounds on mixtures of diethylmagnesium and di(n-butyl)magnesium with a view to solving the technical problem underlying the disputed patent.

If experiments with higher dialkyl magnesium compounds suggested themselves to a skilled person as an obvious way of arriving at a solution offering increased contents of di-(n-butyl)magnesium in solution in hydrocarbon solvents, the extent of that increase cannot be taken as an indication that such experiments - obvious as they were - involve an inventive step.

Therefore, the subject-matter of Claim 1 does not involve an inventive step.

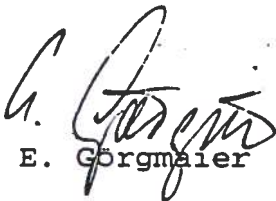
Since the Respondent's only request is that the appeal be dismissed, in the absence of a valid Claim 1 the patent must be revoked.

Order

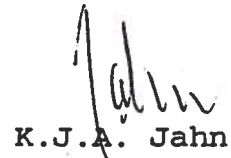
For these reasons, it is decided that:

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar:


E. Gorgmaier

The Chairman:


K.J.A. Jahn