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DECISION of 10 August 1995

T 0124/93 - 3.3.2 Case Number:

Application Number: 85301994.1

Publication Number: 0159150

IPC: C01F 5/24

Language of the proceedings: EN

Title of invention:

Magnesium hydrocarbyl carbonate supports

Patentee:

AMOCO CORPORATION

Opponent:

Shell Oil Company

Union Carbide Chemicals and Plastics Company Inc.

Headword:

Supported Catalyst/AMOCO

Relevant legal provisions:

EPC Art. 54, 56, 83, 107, 123(2), 123(3)

Keyword:

"Right to appeal of proprietor after maintenance with agreed version of auxiliary request - yes"

"Novelty, inventive step and sufficiency - yes (after amendment)"

Decisions cited:

T 0150/82, T 0248/85, T 0205/83, T 0595/90

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 0124/93 - 3.3.2

DECISION of the Technical Board of Appeal 3.3.2. of 10 August 1995

Appellant: (Opponent 01) Shell Oil Company One Shell Plaza

Houston (US) Texas

Representative:

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Other party: (Opponent 02) Union Carbide Chemicals and Plastics

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Appellant:

AMOCO CORPORATION

(Proprietor of the patent)

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Chicago

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Representative:

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Decision under appeal:

Interlocutory decision of the Opposition Division of the European Patent Office of 5 November 1992, issued 23 December 1992 and, with new date, on 22 January 1993, concerning maintenance of European patent No. 0 159 150 in amended form.

Composition of the Board:

Chairman:

F. Antony

Members:

G. J. Wassenaar C. Holtz

## Summary of Facts and Submissions

- European patent No. 0 159 150 was granted in response to European patent application No. 85 301 994.1, filed on 22 March 1985. The mention of the grant was published in European Patent Bulletin 89/37 of 13 September 1989.
- II. Notices of opposition were filed against the European patent by Opponents 01 and 02 on 5 June 1990 and 8 June 1990, respectively. Revocation of the patent was requested on the grounds of lack of novelty, lack of inventive step and insufficient disclosure (Articles 100(a) and (b) EPC).

Of the documents cited, the following are relevant for the present decision:

- (1a) US-A-4 244 838
- (2) US-A-4 277 370
  - (4) US-A-4 384 983
  - (5) US-A-4 318 963
  - (6) GB-A-2 101 610
  - (7) US-A-4 199 473.
- III. The Opposition Division in an interlocutory decision of 5 November 1992, issued on 23 December 1992, and reissued under date of 22 January 1993, held that the patent could be maintained in amended form on the basis of the granted process claims 9 to 32, in accordance with an auxiliary request of the Proprietor. The granted product claims 1 to 8 were rejected on the ground of lack of novelty.

It was considered that the product claims should be interpreted as product-by-process claims whereby the product was obtained by reaction of a magnesium

hydrocarbyl carbonate support with a transition metal component. If the latter was, e.g. titanium chloride the reaction product could be merely a mixture of magnesium, aluminium and titanium chlorides, which was considered indistinguishable from the products disclosed in (1a) and (2).

On the other hand, the specific combination of starting materials and sequence of reaction steps set out in the independent granted process claims 9 and 24 were not disclosed in any of the cited documents. These claims were, therefore, considered novel. With respect to inventive step it was held that the problem underlying the invention was to provide catalyst components or catalysts with fewer fines; that the granted process solved this problem by the new step of forming a magnesium hydrocarbyl carbonate as intermediate product, which is further reacted with the transition metal compound; and that the available documents provided the skilled man no hint for such a process step.

- IV. Appeals against this decision were lodged by both, the Proprietor and Opponent 01 on 28 January 1993 and 17 March 1993, respectively. Statements of Grounds were submitted on 28 May 1993 and 19 May 1993, respectively.
- V. In his Statement of Grounds and further submissions, the Proprietor refuted the arguments of the Opposition Division with respect to lack of novelty of granted claims 1 to 8 and referred to the evidence provided by his first test report filed with his observations dated 21 March 1991 (Tables C and 7), and his second test report and further observations dated 30 September 1992 (Table 3), showing that the claimed product had at least one new property over prior art products, in particular, a narrower particle size distribution. During oral proceedings, held on 10 August 1995 the Proprietor filed

a new set of twenty claims to replace all existing claims on file. Of these, the independent claims 1, 3 and 4 read as follows:

- "1. A supported titanium-containing olefin catalyst component comprising the product of
- (a) reacting a magnesium alcoholate with carbon dioxide to form magnesium hydrocarbyl carbonate,
- (b) forming a precipitate by contacting the magnesium hydrocarbyl carbonate in a liquid medium with a compound selected from Group II and Group III(A) metal alkyls and Ti(IV)halides, and
- (c) reacting the precipitate with a Ti(IV) halide.
- 3. A catalyst composition comprising an olefin catalyst component as claimed in claim 1 and a Group II or III(A) metal alkyl component.
- 4. A process for preparing a supported titanium-containing olefin catalyst component comprising the steps of

reacting a suspension comprising a magnesium alcoholate with carbon dioxide to form magnesium hydrocarbyl carbonate,

forming a precipitate by contacting the magnesium hydrocarbyl carbonate in a liquid medium with a compound selected from Group II and III(A) metal alkyls and titanium(IV)halides, and

reacting the precipitate with a
titanium(IV)halide."

VI. The appealing Opponent 01 essentially argued that not only the product claims but also the process claims lacked novelty over (1a) and (6). The subject matter of the latter would anyhow lack any inventive step over (1a) or (6) in view of the disclosure of magnesium hydrocarbyl carbonates in (5). It was further

argued that the Proprietor's experimental reports did not demonstrate any clear and reliable distinction of the products of the patent in suit over the products disclosed in (1a) and (2). Furthermore, the products as claimed could not be obtained by the processes disclosed in the specification so that the invention was insufficiently disclosed.

- VII. The non-appealing Opponent 02 first and foremost challenged the Proprietor's right to appeal because the latter allegedly had agreed to the maintenance of the patent in the amended form held allowable by the Opposition Division, a position also taken by Opponent 01. The novelty was also attacked on the basis of (7), and the inventive step on the basis of (7) in combination with (4). The insufficiency argument was similar to that of Opponent 01.
- VIII. The Proprietor requested that the decision under appeal be set aside and that the patent be maintained on the basis of claims 1 to 20 filed during oral proceedings.

Both Opponents requested that the appeal by the Patentee be rejected as inadmissible.

The appealing Opponent 01 further requested that the decision under appeal be set aside and the patent be revoked in its entirety.

The non-appealing Opponent 02 further requested that the appeal of the Proprietor be dismissed.

### Reasons for the Decision

- 1. Admissibility of the appeals
- 1.1 The Board observes that the Opposition Division, after having delivered its decision with a mailing date of 23 December 1992, re-delivered the same with an additional enclosure under the new mailing date of 22 January 1993. Well-meant as the said re-dating certainly was, the Board is compelled to note that, under Rule 89 EPC, the Opposition Division was not empowered to do such redating, and the terms for filing notices of appeal and written statements setting out the grounds of appeal, notwithstanding such unlawful redating, would have expired two and four months, respectively, counting from the original mailing date of 23 December 1992.

The statement of grounds of the Proprietor and both the notice of appeal and the statement of grounds submitted by the Opponent 01 were filed after the respective dates of expiration. However, the parties were misled by the above-referred re-dating, and following the principle of good faith governing the relationship between the EPO and the parties acting before it, their submissions received after expiration of the normal terms are considered to have been made in due time.

1.2 With regard to Proprietor's right to appeal, the Board cannot find any statement in the minutes of the oral proceedings before the Opposition Division from which it could be concluded that the Proprietor had dropped his main request, i.e. maintenance of the patent with product claims as granted. In fact, during oral proceedings before the Board, the parties confirmed that no such statement had been made.

Moreover, according to paragraph 13 of the decision under appeal, the Proprietor had established that the order of preference for his then requests was main request, auxiliary request 2 (AR2), AR 1 then AR 3, indicating that the main request was not withdrawn. Furthermore, the novelty of the product claims was extensively discussed in the decision under appeal and in its paragraph 26 it was concluded that claims 1 to 8 (and claims 1 and 5 of auxiliary request AR 2) were not allowable. The reference to claims 1 to 8 could only have related to the claims as granted, i.e. to the main request.

From all this the Board must conclude that the product claims as granted were not withdrawn, so that the Proprietor had a right to appeal the adverse decision to refuse granted claims 1 to 8.

All other requirements of Articles 106 to 108 and Rule 64 EPC have been met by both appeals, which are therefore admissible.

Allowability of amendments under Article 123 EPC

### 2.1 Article 123(2) EPC

New product claims 1 to 3 are essentially based on a combination of claims 1 to 5, 9 and 10 as originally filed, new process claim 4 on a combination of original claims 9, 10 and 13. The sub-claims 5 to 20, dependent upon claim 4, are all based on the original sub-claims dependent upon claims 9 or 24. The reference to "olefin catalyst" can be derived from page 3, lines 20 to 24 of the original description.

The new wording in claims 1 and 4 of "forming a precipitate by contacting the magnesium hydrocarbyl carbonate in a liquid medium with a compound selected

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from Group II and Group III(A) metal alkyls and Ti(IV)halides" is meant to embrace both, the forming of a precipitate from a solution and from a suspension, the first meaning being covered by original claims 12 and 13, the latter illustrated by Examples I to V, both meanings to be more clearly understood in the light of page 12, line 36 to page 14, line 21 of the original description, particularly page 13, lines 1 to 5.

Thus the new set of claims does not contain subject matter which would extend beyond the content of the application as filed, so that it satisfies the requirements of Article 123(2) EPC.

## 2.2 Article 123(3) EPC

The Board cannot share the opponents' view that the new claims would extend the protection conferred by the granted version since they no longer require the presence of a magnesium hydrocarbyl carbonate support in the composition.

In the Board's opinion, taking into account that for determining the extent of the protection conferred by a European patent the description shall be used to interpret the claims (Article 69 EPC), it was clear to the skilled reader that granted claim 1 covered a support comprising the reaction product of the magnesium hydrocarbyl carbonate with the transition metal (Ti) compound. This position is supported by the fact that the granted patent specification indicated several times that the composition as defined in granted claim 1 was obtained by reacting said carbonate with a transition metal compound (e.g. page 3, lines 3 to 6 and claim 9). From the disclosure on page 6, lines 46 to 47 that the atomic ratio of transition metal to magnesium in the catalyst component of the invention ranges from 0.5:1 to

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20:1 it was evident that the said carbonate would completely react with the transition metal compound so that no carbonate would be left over in the final product.

Thus, in spite of the literal wording of the granted product claims, which seemed to suggest the contrary, it was clear to the skilled reader, taking into account the whole context of the specification, that the presence of a magnesium hydrocarbyl carbonate in the support was not required by the claims as granted.

The additional features in the independent claims 1, 3 and 4 limit the scope of the claims compared with the independent claims 1, 5, 9 and 24 as granted so that the present set of claims also satisfies the requirements of Article 123(3) EPC.

# Novelty

The novelty of the present set of claims was attacked on the basis of documents (1a), (6) and (7).

3.1 Document(1a) discloses a process for preparing a supported titanium-containing olefin catalyst component, comprising the steps of reacting in a solvent a magnesium hydrocarbyl component, such as a magnesium alkyl, with at least one of water, carbon dioxide or an organic oxygen-containing compound, such as an alcohol, to form a first reaction product, which product is subsequently treated with a transition metal component comprising halogen, such as titanium tetrachloride, to form a second solid, hydrocarbon insoluble reaction product (claim 1 and Examples 1 and 10).

Document (1a) does not literally disclose the forming of a magnesium hydrocarbyl carbonate, but according to the opponents such a carbonate is inevitably formed if magnesium alkyl is treated with carbon dioxide in the presence of an alcohol. While the Board accepts the evidence that under those conditions such a carbonate is indeed formed, the said conditions are not in fact disclosed in (1a). Although (1a) embraces the possibility of jointly using an alcohol and carbon dioxide to form the "first reaction product", such a combination would require a selection lacking any specific disclosure. In each of the ten examples only one of water, carbon dioxide or alcohol is used, and there is no pointer for such a combination serving any useful purpose.

Moreover, claims 1 and 4 of the patent in suit do not only require a carbonate to be formed, but also that it is formed by reacting a magnesium alcoholate with carbon dioxide. The forming of a magnesium alcoholate is not disclosed in (1a). From the evidence before the Board it cannot be derived that magnesium alcoholate would be formed if magnesium alkyl were reacted with carbon dioxide in the presence of an alcohol.

3.2 Document (6) discloses a process for preparing a supported titanium-containing olefin catalyst component, comprising the steps of treating a supported magnesium composition, such as magnesium alkyl, with at least one reagent selected from carbon dioxide, oxygen and a hydroxyl compound such as an alcohol to form an intermediate reaction product, followed by reacting the said intermediate product with at least one carbonyl compound and with a transition metal compound such as titanium tetrachloride to form a final reaction product. Here again, although a combination of carbon dioxide and alcohol is not excluded, there is nowhere a specific

disclosure for such a combination. If any combining of reagents is at all suggested, it is possibly for the combination of carbon dioxide and oxygen to be used as a gas mixture (page 3, lines 24 to 27). Of the 14 examples describing the preparation of a catalyst, only one, viz. Example 10, uses oxygen in the first reaction step; in all the other examples an alcohol is used. A combination of an alcohol and of carbon dioxide in the first reaction step is not disclosed in (6), so that the first reaction step of claims 1 and 4 of the patent in suit is not anticipated by (6).

3.3 Document (7) discloses a process for preparing a supported titanium containing olefin catalyst component, comprising the steps of reacting an organo-magnesium component with an oxygen-containing compound, such as an alcohol, or with carbon dioxide or oxygen, to form an intermediate product which is then reacted with titanium tetrachloride.

Here again, there is no disclosure for the use of a combination of carbon dioxide and an alcohol and the forming of a carbonate. Thus (7) does not anticipate the first reaction step of claims 1 and 4 of the patent in suit.

- 3.4 None of the other citations discloses the first reaction step of the claimed process, hence process claim 4 and its dependent claims 5 to 20 are new over the available prior art.
- 3.5 The fact that a process for preparing a composition is new does, however, not necessarily mean that the prepared composition is new. According to established case law of the Boards of Appeal (T 150/82, OJ EPO 1984, 309; T 248/85, OJ EPO 1986, 261 and T 205/83, OJ EPO 1985, 363) product-by-process claims give protection for

the products as such, independent from the process by which they were made, and the said products, therefore, likewise have to fulfil the requirements of patentability such as novelty and inventive step independent from the novelty and inventive step of the process.

3.5.1 Considering the chemical reactions which take place, it is evident that the chemical compounds constituting the product of claim 1 may be identical to those of the products of (1a) or (2).

According to the Proprietor, however, the physical parameters of the product of claim 1 differ in several respects from those of the prior art products, notably concerning its particle size distribution. By way of support for this allegation, comparative examples were filed showing that the particle size distribution of the products obtained according to present Example III is much narrower than that obtained by the examples of (1a) and (2), in other words, that the particle size dispersion of the products of claim 1 is lower than of prior art products having the same chemical constitution.

3.5.2 The Opponent 01 has objected that those comparative examples were not reliable and did not convincingly show a lower particle dispersion for the claimed products. In particular it was indicated that the particle size distribution for product III B (Example III, Product B) as represented by Figure 4 of the Proprietor's first test report could not be compared with Figure 1 of Appendix III of his second test report representing the particle size distribution of Example 1 of (1a), because product III B had been three times treated with titanium chloride, whereas the product of (1a) had undergone only one such treatment.

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3.5.3 The Board accepts that the said comparative example alone does not indisputably show that the reduced particle dispersion is due to the formation of magnesium hydrocarbyl carbonate according to step (a) of present claim 1; there is, however, other evidence which strongly supports that position, as set out below: The particle size distribution of product III A (Example III, product A) as represented by Figure 3 of the Proprietor's first test report is very similar to that of product III B, although product III B is obtained from product III A by a further treatment with titanium chloride for a long time (12h) at elevated temperatures (110°C). On the other hand, products III A and B have also been compared with the corresponding products obtained without addition of carbon dioxide, i.e. without the forming of the intermediate magnesium hydrocarbyl carbonate, and revealing that without carbon dioxide the products had a particle size distribution which was much broader and rather resembled the distribution in the products obtained according to Example 1 of (2) and Examples 1 and 7 of (1a); compare Figures 3 and 4 of the Proprietor's first report with Figures 1, 2, 5 and 6 of the same report and sheets 1 to 11 of Appendix III of the Proprietor's second report. Although the reduced particle size dispersion was only proved for the product of Example III, the Board can see no reason why the products of the other Examples should not behave similarly, and the opponents have not provided any evidence that claim 1 would cover a substantial number of products without such reduced particle size dispersion. In the absence of such evidence the Board considers the reduced particle size dispersion to be an inherent property of the products of claim 1 establishing the novelty of its subject matter. The same applies to the product of claim 3, which comprises the product of claim 1.

- 4. Inventive step
- 4.1 Process Claims 4 to 20
- 4.1.1 The closest prior art is considered to be (1a) because it relates to the same type of catalyst and has more features in common with the present method of preparing the catalyst component than any other cited prior art document.

A general problem with supported catalysts for the polymerisation of olefins is the presence of catalyst fines. It is therefore desirable in preparing the catalyst to start with a catalyst component containing a low amount of such fines.

4.1.2 The technical problem underlying the invention can therefore be seen in providing a catalyst component with a reduced level of fines (see page 2, lines 47 to 48 of the description).

Although the specification does not provide clear numerical values for "fines", the expression is often used in the art and a "reduced level of fines" implies a particle size distribution in which the amount of particles with a size substantially lower than the average particle size is lower than in comparable catalyst components.

An indication for the amount of fines is the ratio of the particle sizes at a particle percentile of d10 and d50; the closer this ratio is to 1 the lesser fines will be present.

4.1.3 According to claim 4 the above-mentioned problem is solved by taking a magnesium alcoholate as a starting compound and, in a first step, reacting it with carbon dioxide to form magnesium hydrocarbyl carbonate. As

indicated above, the Proprietor's comparative examples reveal to the Board's satisfaction that the claimed method gives rise to a particle size distribution with a lower dispersion and a lower amount of fines than according to Examples 1 and 7 of (1a). The ratio of d10 to d50 for Examples 1 and 7 of (1a) is 0.14 and 0.16 respectively, whereas for present Examples III A and B this ratio is 0.74 (compare the Proprietor's first report Tables 3 and 4 and his second report Annex III, Sheets 1 and 2).

Thus the Board is satisfied that the said problem is solved by the process of claim 4.

4.1.4 It remains therefore to be decided if, for solving the above stated problem, it was obvious to start with a magnesium alcoholate, to convert it first into a magnesium hydrocarbyl carbonate by reacting it with carbon dioxide, and to react the said carbonate with titanium halide to obtain a supported catalyst component.

Contrary to the views expressed by the opponents, the references, to the use of at least one of water, carbon dioxide and an oxygen containing compound such as an alcohol (la), or of at least one of carbon dioxide, oxygen and a hydroxyl compound such as an alcohol (6), does not make the combination of carbon dioxide and an alcohol an obvious choice.

The purpose, in both said citations, for reacting the magnesium alkyl with "at least one" of the aforementioned compounds is to convert the magnesium alkyl in an oxygen containing compound before it is reacted with the transition metal halide. There is no indication in the entire prior art that any advantage can be achieved by combining two or three of the said

compounds, let alone that a better particle size distribution could be achieved by combining carbon dioxide with an alcohol. With respect to the particle size of the catalyst component (la) merely recommends to add an aluminium compound to the solution of the magnesium compound before the oxygen containing compound is added in order to avoid lumps in the catalyst (column 7, lines 47 to 63).

Moreover, a mere selection of alcohol and carbon dioxide would not automatically result in a process according claim 4. To obtain a magnesium alcoholate and to form a magnesium hydrocarbyl carbonate, the order of adding the reactants is important. There is no indication in (1a) or (6) of any specific order of adding the oxygen containing reagents if a combination of reagents were to be envisaged.

The use of an alcoholate as a starting material for a supported catalyst component is well known in the art, e.g. from (2). In none of the documents in which an alcoholate is so used is there any mention of a reaction with carbon dioxide or the formation of a carbonate.

Neither in (2), nor in any other citation is there any indication that the use of a carbonate as intermediate product could have any influence on the particle size distribution of the supported catalyst component.

While the formation of magnesium hydrocarbyl carbonate by reacting carbon dioxide with a suspension of a magnesium alcoholate has been disclosed in (5), this reaction merely serves to solubilise the magnesium compound for the purpose of treating paper. There is no connection whatever with particle size distribution or supported catalysts.

Document (7) discloses the preparation of a supported catalyst composition by reacting in a first step an organo-magnesium compound with an oxygen-containing compound such as an alcohol, or with carbon dioxide, or with oxygen to introduce a C-O-Mg bond into the organo-magnesium compound. The Board cannot follow Opponent's 02 allegation that (7) would also imply that the initially formed alcoholate could be further reacted with carbon dioxide to a magnesium hydrocarbyl carbonate.

Document (4), also relating to supported catalyst for the production of polyolefins, mentions both magnesium alkyl and magnesium alcoholate as support materials for supported catalysts. It also discloses, however, that the catalyst preparation is preferably conducted in an inert hydrocarbon in the substantial absence of water, oxygen, carbon dioxide and other extraneous catalyst poisons (column 6, lines 51 to 57). Since (4) discloses to avoid catalyst poisons such as carbon dioxide it can give no incentive to replace in (7) magnesium alkyl with magnesium alcoholate and to react the alcoholate with carbon dioxide to form magnesium hydrocarbyl carbonate.

Thus the available prior art does not give the skilled man any incentive that the above mentioned technical problem could be solved by the method as claimed in present claim 4.

It follows from the foregoing considerations that claim 4 is not only new, but also involves an inventive step in the meaning of Article 56 EPC. Since claims 5 to 20 are all sub-claims dependent upon claim 4, their patentability follows from that of claim 4 without the need for any separate considerations.

### 4.2 Product claims 1 to 3

As indicated above, the problem underlying the invention is to provide a catalyst or catalyst precursor with a reduced level of fines. As further indicated under point 3.5.3, the products of claims 1 and 3 differ from (la) in having a lower particle size dispersion. This is generally considered to be a favourable property for a powder. In the present case the skilled man would expect that a narrower particle size distribution of the catalyst would result in a more homogeneous polymer, i.e. polymer particles of about equal size and with about equal properties. The products of claims 1 and 3 might thus be called "obvious desiderata". For a "desideratum" to become obvious in the meaning of Article 56 EPC, there must however be obvious technical means available to the skilled man for its realisation (cf. T 595/90 OJ EPO 94, 695). Such means are not apparent here. Apart from (1a) recommending the addition of an aluminium compound to a solution of magnesium alkyl prior to adding an oxygen-containing compound to avoid catalysts containing lumps (cf. point 4.1.4 above), none of the cited documents provides any information how the particle size distribution of supported catalyst components could be influenced. There was therefore no obvious way for the skilled man how to reduce the level of fines and to obtain a catalyst component with a lower particle dispersion than that obtained according to (1a).

It follows from the foregoing considerations that the products of claims 1 and 3 are not only new, but also involve an inventive step in the meaning of Article 56 EPC. Since claim 2 is a sub-claim dependent upon claim 1, its patentability follows from that of claim 1.

5. Sufficiency of disclosure

The opponents had raised the ground of insufficiency with the argument that the claims as granted would require the presence of magnesium hydrocarbyl carbonate in the support of the catalyst component, whereas the description disclosed methods according to which the support did not comprise such. Since the present set of claims avoids this apparent contradiction, said opposition ground is now without substance.

### Order

### For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The requests that the appeal by the Patentee be rejected as inadmissible are refused.
- 3. The case is remitted to the first instance with the order to maintain the patent with claims 1 to 20 as submitted in the oral proceedings and a description to be adapted thereto.

The Registrar:

The Chairman:

P. Martorana

F. Antony