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D E C I S I O N
of 19 October 1994

Case Number: T 0627/91 - 3.3.2

Application Number: 85301543.6

Publication Number: 0155800

IPC: C06B 47/14

Language of the proceedings: EN

Title of invention:
Emulsion explosives composition

Patentee:
IMPERIAL CHEMICAL INDUSTRIES PLC

Opponent:
(01) The Lubrizol Corporation
(02) Nitro Nobel AB
(03) Dynamit Nobel Aktiengesellschaft
(04) Westpreng GmbH Sprengstoffe + Sprengtechnik

Headword:
Emulsion explosives/ICI

Relevant legal provisions:
EPC Art. 54, 56, 83, 84, 123

Keyword:
"Added subject-matter (no) - after amendment"
"Clarity (yes)"
"Sufficiency of disclosure (yes)"
"Novelty - main request (no) - first auxiliary request (yes)"
"Inventive step (yes) - commercial application of a discovered property"

Decisions cited:
G 0001/92, T 0390/88

Catchword:
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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0627/91 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 19 October 1994

Appellant: (Proprietor of the patent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office dated 24 June 1991 revoking European patent No. 0 155 800 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: P. A. M. Lançon
Members: I. A. Holliday
C. Holtz

Summary of Facts and Submissions

I. European patent No. 0 155 800 was granted on the basis of 20 claims contained in European patent application No. 85 301 543.8

II. Four oppositions were filed against the granted patent. Of the numerous citations referred to by the Opponents, only the following remains of relevance to the present decision.

(1) EP-A-0 018 085

Document (9) is the US equivalent of (1) based on the same Canadian priority documents.

In the course of the proceedings, several affidavits were filed of which the following remain relevant

(E1) J. Cooper, dated 2 July 1986
(E2) J. Cooper, dated 29 October 1991
(E4) C. G. Wade, dated 18 October 1989
(E8) D. J. Nicolarson, dated 16 October 1989

The Opponents also cited a prior use in relation to certain emulsion explosives sold before the first priority date by Atlas Powder Company.

III. The Opposition Division, whilst considering that the claimed subject-matter satisfied the sufficiency requirements of Article 83 EPC, held that it lacked novelty in the light of the disclosure of US-A-4 110 134 (15) and DE-A-3 329 064 (12).

IV. The Appellant lodged an appeal against the decision of the Opposition Division, submitting a new main request together with nine auxiliary requests.

Oral proceedings took place on 19 October 1994. Respondent O1 played no part in the appeal.

The summons to oral proceedings was accompanied by a communication pursuant to Article 11(2) of the rules of procedure of the Boards of Appeal which expressed the provisional opinion that the alleged prior use appeared to be well documented and accordingly the subject-matter of the requests then on file lacked novelty. On 8 September 1994, the Appellant submitted a new main request and five auxiliary requests.

V. The arguments of the Appellant, both at the oral proceedings and in the written procedure may be summarised as follows:

In the light of decision G 1/92 (OJ EPO 1993, 277), the Appellant accepted that the sales of the commercial explosives referred to in the affidavits E4 and E8 prejudiced the novelty of the claims then on file. Accordingly, Claim 1 of the main request filed on 8 September contained a disclaimer to the use of an emulsifier which is a condensate of poly(isobutenyl) succinic anhydride (hereinafter PIBSA) and N,N-diethylethanolamine. Claim 2, which related to a mixture of emulsifiers did not contain a disclaimer. The Appellant argued that although the prior use was relevant when considering novelty, it was not relevant to the question of inventive step.

The Appellant considered document (1) to be the closest state of the art since it was concerned with the same technical problem; decision T 423/89 of 10 June 1992

(not published in OJ EPO) was referred to. It was acknowledged that the explosive compositions known from (1) contained related condensates of PIBSA as emulsifiers and that they exhibited reasonable long term stability. The Appellant referred to Affidavit (E1) of Cooper which gave details of stability. The compositions of the patent in suit exhibited not only long term stability, manifested by a resistance to crystallisation but also resistance to shock, e.g. as encountered when transporting the compositions. Some samples prepared around the priority date of the patent in suit were still in I.C.I. magazines and remained usable explosives. Although the prior art did not record the conductivity of emulsion explosives, the Appellant denied that any prior art product, other than those of the acknowledged prior use, exhibited a conductivity below the value quoted in Claim 1 of the patent in suit. By measuring the conductivity, the Appellant has developed a convenient means to identify emulsions having good stability.

The Appellant defended the claims filed on 8 September 1994 against attacks under Article 123 and 84 EPC. However, in response to opinions expressed by the Board, references to a "pumpable" emulsion and to "storage life of at least 55 weeks at 10°C" were deleted and amended claims were placed on file at the Oral Proceedings.

VI. The Respondents' arguments both in the written procedure may be summarised essentially as follows:

The Respondents' objections under Article 123 EPC were at least partially met by amendments filed during the oral proceedings. In addition to those referred to above, concerning "pumpable" and storage life, one Respondent objected to the reference to "capable of being packaged into conventional 25 mm cylindrical

cartridges". It was also argued that the deletion of the reference to "in the absence of adjuvant" which had been a feature of the earlier main claims amounted to an extension of scope. The reference in Claim 13 to inclusion of such an adjuvant was also considered to be an unallowable extension. One Respondent argued that Claim 11 referring to a condensate of PIBSA with an ethanolamine restored subject-matter which had been disclaimed in Claim 1.

One Respondent objected to the form of the disclaimer itself, arguing that, it would have been possible to draft the main claim in a positive fashion.

In respect of Article 84 EPC, the Respondents argued that the terms "pumpable", "reduced tendency", "strongly lipophilic" and "high affinity" were of uncertain scope.

The Respondents made objections under Article 83 EPC, especially relating to the measurement of conductivity both during the proceedings before the Opposition Division and in written responses to the statement of appeal. This point was not discussed during the oral proceedings before the Board.

Respondent (O4) argued that the relation between the conductivity and the storage stability amounted merely to a discovery and as such was not patentable in terms of Article 52(2) EPC.

The Respondents maintained their argument that document (1) prejudiced the novelty of the claimed subject-matter. Similar emulsifiers based on PIBSA were used in compositions disclosed in (1) and would inevitably have conductivity values below the prescribed maximum in the claims of the patent in suit.

It was also argued that the prior use anticipated Claim 2 of the main request. The PIBSA condensates formed by reaction with N,N-diethylethanolamine were, having regard to the chemistry of their formation, inevitably mixtures of different species, e.g. monomers, dimers or trimers. Such a mixture would anticipate Claim 2 of the main request which did not contain the disclaimer of Claim 1.

The Respondents argued that both starting from the prior use or from document (1), the claimed subject- matter lacked inventive step. Respondent (O2) referred, just prior to the Oral Proceedings, to several US patents relating to PIBSA emulsifiers. These indicated that such emulsifiers were known some twenty years before the priority date of the patent in suit. The Respondent argued that it would have been obvious to substitute another PIBSA condensate for the condensate with N,N-diethylethanolamine known from the prior use and to arrive at the subject-matter of the patent in suit; such mixtures would inevitably satisfy the conductivity requirements of Claim 1. It was also argued that it would have been obvious to substitute emulsifiers known from (1) for the emulsifiers disclosed in the prior use

Starting from document (1), the Respondents argued that, even if novelty could be established, the problem underlying the patent in suit had already been solved. Particular reference was made to Examples 30 and 31 of (1) which employ (inter alia) PIBSA emulsifiers. It has been shown that the explosive compositions of these examples has good long term stability, a period of 12 months at 5°C being quoted. In the Respondent's view, it would have been obvious to substitute other PIBSA type emulsifiers for the "polyesters B" actually disclosed in (1).

VII. Claim 1 of the main request submitted at the oral proceedings on 19 October 1994 reads as follows:

"1. An emulsion explosive composition which is capable of being packaged into conventional 25 mm cylindrical cartridges and with a reduced tendency to crystallise during storage or transport,

consisting of an oxygen-supplying component forming a discontinuous phase, an organic medium forming a continuous phase and one or more emulsifiers,

characterised in that at least one emulsifier is strongly lipophilic (i.e. having a high affinity for the oily or organic medium) and is an electrical conductivity modifier consisting essentially of a hydrophilic moiety and a lipophilic moiety, and in which the lipophilic moiety comprises a chain structure incorporating a backbone sequence having at least 10 and not more than 500 linked atoms derived from a polymer of mono-olefin containing 3 to 6 carbon atoms linked to the hydrophilic moiety,

and said emulsifier-electrical conductivity modifier is present in an amount effective to provide an emulsion which exhibits an electrical conductivity, measured at a temperature of 60°C not exceeding 60,000 picomhos/metre,

excluding emulsion explosive compositions in which the emulsifier-electrical conductivity modifier is a condensate of poly(isobutenyl)succinic anhydride and N,N-diethylethanolamine."

Independent Claim 2, which does not contain a disclaimer, reads as follows:

"1. An emulsion explosive composition which is capable of being packaged into conventional 25 mm cylindrical cartridges and with a reduced tendency to crystallise during storage or transport,

consisting of an oxygen-supplying component forming a discontinuous phase, an organic medium forming a continuous phase and at least one emulsifier which is strongly lipophilic (i.e. having a high affinity for the oily or organic medium) and is an electrical conductivity modifier consisting essentially of a hydrophilic moiety and a lipophilic moiety, and in which the lipophilic moiety comprises a chain structure incorporating a backbone sequence having at least 10 and not more than 500 linked atoms derived from a polymer of mono-olefin containing 3 to 6 carbon atoms linked to the hydrophilic moiety,

and said emulsifier-electrical conductivity modifier is present in an amount effective to provide an emulsion which exhibits an electrical conductivity, measured at a temperature of 60°C not exceeding 60,000 picomhos/metre,

characterised in that said emulsifier-electrical conductivity modifier is one of a mixture of emulsifiers employed in said composition to promote the dispersion of the discontinuous phase in the continuous phase."

Independent Claims 14 and 15 relate to processes for preparing emulsion explosives based on Claims 1 and 2 respectively. Dependent Claims 3 to 13 and 16 to 17 relate to preferred embodiments of the compositions and processes respectively.

According to the first auxiliary request, Claim 2 is dependent on Claim 1 and Claim 15 on Claim 14. In other words the disclaimer applies also to Claims 2 and 15.

The second auxiliary request contains a modified disclaimer. Again Claims 2 and 15 are independent.

VIII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the

basis of Claims 1 to 17 (main request) or Claims 1 to 17 (auxiliary request 1) or Claims 1 to 17 (auxiliary request 2), all as filed at the oral proceedings.

The Respondents requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments*

2.1 In each of the worked examples of the patent in suit, the emulsion explosive prepared is packaged into cartridges of 25 mm diameter. The amendment "capable of being packaged into conventional 25 mm cylindrical cartridges" accordingly has adequate basis in the originally filed documents. The fact that cartridges of greater diameter are additionally employed in Example 4 does not alter this conclusion.

2.2 According to Wade (E4), the emulsion explosives sold before the first priority date contained, as emulsifier, a commercial product TN 0115 which was a reaction product of PIBSA and N,N-diethylethanolamine in a 1:2 molar ratio. The disclaimer which relates to all condensates of PIBSA and N,N-diethylethanolamine thus has a basis in the prior art which was not disputed by the other parties.

2.3 Claim 1 as granted related to an emulsion explosive composition which "in the absence of a supplementary adjuvant" exhibited a prescribed conductivity. According to the description (column 3, lines 20 to 27), adjuvants

such as waxes and microballoons are added in order to modify the explosive performance. Thus what was claimed in the granted claims was in effect a "pre-composition", i.e. an emulsion explosive less the above mentioned adjuvant. The present Claim 1 does not mention the word "adjuvant". However, what is now claimed amounts to the same thing; use of the expression "consisting of" would preclude the presence of an adjuvant.

2.4 The claims as granted did not contain a claim specifically referring to the presence of an adjuvant. However, there is basis in Claim 1 as granted, together with the description mentioned in point 2.3 above, for such a claim. In other words Claim 13 does not add subject-matter.

2.5 Dependant Claim 11 relating to a modifier which is a condensate of PIBSA and an ethanolamine (based on Claim 16 as granted) is not in conflict with the disclaimer but must be construed in the light thereof, i.e. a condensate of PIBSA with an ethanolamine **other than** N,N-diethylethanolamine.

2.6 The Board is also satisfied that a disclaimer is here the most convenient means of meeting the prior use novelty objection. To attempt to formulate a claim in a positive manner would have led to a very complicated claim which would in any event have had to exclude condensates of N,N-diethylethanolamine. The disclaimer corresponds to a disclosure of which the Appellant was apparently unaware at the time the application was filed.

2.7 The Board is satisfied that the other amendments to the claims have a basis as set out in Annex 2 of the Appellant's letter dated 8 September 1994. The claims presently on file (main and auxiliary requests) relate

to more restricted subject-matter than the granted claims. The requirements of Article 123(2) and (3) are accordingly satisfied.

3. *Clarity (Article 84 EPC)*

3.1 The main claims of the main and the two auxiliary requests contain the subjective expressions "reduced tendency", "strongly lipophilic" and "high affinity" which were not present in the claims as granted. Although the Board considers such terms to be undesirable, they are not such as to obscure the scope of the claims.

3.2 In full accordance with the description, the expression "reduced tendency" must of course be construed in relation to the prior art and the other expressions must signify a high lipophilic/hydrophilic balance.

3.3 The requirements of Article 84 are therefore regarded as satisfied.

4. *Sufficiency of disclosure (Article 83 EPC)*

The objections to the broad scope of the claims do not apply to the main and auxiliary requests currently on file since the chemical nature of the emulsifier is now specified. The Board is also convinced that the description from column 3, line 48 to column 4, line 5 gives adequate instruction to enable one skilled in the art to measure the conductivity and supports the view of the Opposition Division that the requirements of Article 83 EPC are satisfied.

5. *The main request*

5.1 Product Claim 1 of the main request is characterised by the presence as emulsifier of compounds having a lipophilic moiety comprising a chain structure incorporating a backbone sequence having at least 10 and not more than 500 linked atoms derived from a polymer of a mono-olefin containing 3 to 6 carbon atoms linked to the hydrophilic moiety. Such a definition would not provide a significant distinction from the Polyesters B known from document (1). According to page 5 of (1) (lines 1 to 9) the alkenylsuccinic anhydride is derived from a polymer of a mono-olefin having a chain of 40 to 500 carbon atoms.

5.1.1 The burden of proof lay with the Appellant to establish novelty over document (1). During the examination procedure, the Appellant submitted the Cooper affidavit (E1) which had already been filed in the prosecution of the corresponding US application. Dr Cooper prepared emulsion explosives derived from document (9). These corresponded to the use of polymeric emulsifiers 1, 4 and 5 known from (1). These compositions failed to detonate in 25 mm cartridges owing to premature crystallisation. The conductivity of specific compositions corresponding to Examples 15 and 16 of (1) were measured and found to be greater than the maximum specified in Claim 1 of the main request of the patent in suit. The second Cooper affidavit (E2) filed during the appeal verifies and amplifies these conclusions.

5.1.2 Respondent (3) relied on experiments filed during the opposition procedure which related to experimental compositions displaying conductivities as low as 3.0 ps/m. At the oral proceedings before the Board and previously in the written procedure, the Appellant objected to these experiments on several grounds.

Firstly, although allegedly based on compositions within the broad disclosure of (1), they were not based on Examples thereof. The compositions were prepared from refined materials which would not be used in commercial explosives and had viscosities well in excess of those normally applied. The Appellant also queried whether conductivity values as low as those quoted could be measured using the test set out in the patent in suit (cf. 4 above). The Appellant further argued that the different temperatures used to measure conductivity could lead to anomalous results. The Respondent was unable to dispel these doubts at the oral proceedings.

5.1.3 The Board is thus satisfied that, on balance, the Appellant has discharged his burden in establishing the novelty of Claim 1 of the main request over the disclosure of document (1).

5.1.4 The Appellant has not contested that the compositions according to the prior use of the Atlas explosives have conductivity values below the maximum prescribed by Claim 1. Novelty is, however, established by the disclaimer.

5.2 The disclaimer does not, however, apply to Claim 2 which relates to a mixture of emulsifiers at least one having the structure set out in 5.1 above.

5.2.1 The Board is convinced by the arguments of the Appellant at the oral proceedings that the condensates actually used in the compositions of the patent in suit, although relating to mixtures of different molecular species, are not polyesters in the sense of the normally accepted definition and thus differ from the Polyesters (B) of (1). Amendment to include the words "consisting essentially" in the definition of the emulsifier served to clarify the position. This, taken in conjunction with

Dr Cooper's affidavit (E1), is sufficient to establish the novelty of the subject-matter of Claim 2 over the disclosure of (1).

5.2.2 Different considerations, however, apply in respect of the prior use of the Atlas compositions. The Appellant admitted at the oral proceedings that the condensation of PIBSA with a diethylethanolamine is not a simple reaction, indicating that the reaction occurred in two stages, the first stage occurring very quickly (minutes) and the second stage more slowly (hours). The product must therefore be a mixture of different molecular species. It is also to be noted that even PIBSA itself is a mixture; a number average molecular weight of 1200 is quoted for the material used in Example 2 of the patent in suit with a distribution up to 3000. It is accordingly the Board's view that the condensates used by Atlas, although sold as a single emulsifier, must have consisted of a mixture of several different chemical entities and that the prior use thereof is sufficient to destroy the novelty of Claim 2 of the main request.

5.3 The main request must accordingly be refused.

6. *The first auxiliary request*

6.1 Claim 1 of the first auxiliary request is the same as Claim 1 of the main request. For the reasons set out in chapter 5 above, the subject-matter of Claim 1 is novel over both (1) and the Atlas prior use.

6.1.1 According to the first auxiliary request, Claim 2 is appendant to Claim 1; in other words the disclaimer also applies to Claim 2. This is sufficient to establish the novelty of its subject-matter over the Atlas prior use.

6.1.2 For the same reasons as those applying to Claim 1, the novelty of independent process Claim 14 can be established. Claim 14 relates to a process for producing an emulsion explosive in essentially the same terms as those of Claim 1. It also contains a disclaimer in the same terms as that in Claim 1. Dependent process Claim 15 relating to the use of a mixture of emulsifiers is novel for the same reasons as those applying to Claim 2 of the first auxiliary request.

6.2 In considering the problem underlying the patent in suit, the Respondents insisted that the Atlas prior use be regarded as the closest state of the art. On this basis, it was alleged that the problem was merely to develop an alternative emulsion explosive. The Board cannot accept this view. The problem underlying the patent in suit is not so simple as the solution provides not only a mere alternative but also a simple test which enables the skilled person to identify emulsion explosives of reliable storage stability.

6.2.1 The Board considers document (1) to be a more appropriate starting point since, like the patent in suit, it is concerned with the development of emulsion explosives which can be detonated in small diameter bore holes and which are stable over long periods of storage (page 3, lines 14 to 18). The emulsion explosives of (1) employ an aqueous solution of one or more oxygen-supplying salts which forms the discontinuous phase of a water-in-oil emulsion. The emulsifying agent employed is a mixture of a conventional water-in-oil emulsifier, e.g. a sorbitan fatty acid ester, and certain amphipathic graft, block or branch polymeric emulsiers. An example of such a polymeric emulsifier is the group of polyesters B defined on page 5 of (1) which are prepared by condensing PIBSA with a polyalkylene glycol. In the Cooper Affidavit (E1), the Appellant has

shown that compositions within the broad definition of (B) did not have the disired stability.

Starting from (1), the problem to be solved is to develop emulsion explosives which can also be detonated in small diameter (25 mm) bore holes and which have improved long term stability.

The problem is solved by selecting as emulsifier one having a lipophilic moiety derived from an olefin as set out in Claim 1 and which is present in the emulsion in amount such that the electrical conductivity, measured at 60°C, does not exceed 60,000 picomhos/metre. Having regard to the Examples of the patent in suit and to the Cooper affidavit (E1), the Board is satisfied that the problem has indeed been solved.

6.3 It remains to consider whether the claimed solution satisfies the requirements of Article 56 EPC in respect of inventive step.

6.3.1 The patent in suit differs from (1) insofar as it is not necessary to employ a conventional water-in-oil emulsifier in conjunction with the polymeric emulsifier. However, mixtures of emulsifiers are not precluded provided that a lipophilic emulsifier having the definition set out in Claim 1 of the patent in suit is present and that the amount of emulsifier used is such as to provide a conductivity below the minimum prescribed by Claim 1. Emulsifiers of the "Polyester B" type employed in (1) fall within the definition of the emulsifiers used according to the claim but condensates of PIBSA with hydroxyamines, such as ethanolamine and diethanolamine are preferred. The essential feature of the explosive compositions of the patent in suit is their low conductivity. Even though Examples 30 and 31 are stated to have an ageing period of at least 12

months at 5°C, document (1) makes no mention of conductivity. Compositions falling within the broad definition of (B) may or may not have conductivities as now claimed. However, Cooper (E1) has convincingly shown that the skilled person could not rely on document (1) to reliably obtain the desired conductivity. There is accordingly no hint in (1) of any relationship between the conductivity and storage stability of an emulsion explosive.

6.3.2 None of the other documents cited by the Respondent including documents (12) and (15) relied upon by the Opposition Division (cf. III above) make any reference to conductivity. There can thus be no hint of a relationship with the storage life.

6.3.3 In the light of the above, it is apparent that the subject-matter of Claim 1 of the first auxiliary request is in no way foreshadowed by the cited prior art. The Appellant has explained and put to technical use a relationship which enables one skilled in the art, by means of a relatively simple measurement, to predict the long term stability of an emulsion explosive. An inventive step can accordingly be recognised.

6.3.4 Corresponding arguments apply to independent Claim 14 of the first auxiliary request which relates to a process for producing such an emulsion explosive and which is expressed in essentially the same terms as Claim 1. The dependent Claims 2 to 13 and 15 to 17 derive their patentability from Claim 1 and 14 respectively.

6.3.5 It may well be that the compositions of the Atlas prior use have conductivity values below the maximum prescribed by the patent in suit; the Appellant made no attempt to deny this. However, there is no hint of the conductivity being measured before the priority date of

the patent in suit and even less of a connection between the conductivity and the storage stability. As was argued by the Appellant at the oral proceedings and as it is apparent from the Nicolarson affidavit (E8), customers of Atlas were unaware that the prior use explosive had long storage life as they continued to order relatively small quantities at regular intervals, e.g. on a more or less weekly basis according to the figures contained in (E8).

The said prior use relates to an emulsion explosive using as emulsifier a specific condensate of PIBSA and N,N-diethylethanolamine. It is clear, for example from the six US documents cited by the Respondent (O2) immediately prior to the oral proceedings that PIBSA based emulsifiers were generally known for use in water-in-oil emulsions many years before the first priority date of the patent in suit. The Appellant argued that such emulsifiers were not in fact on the commercial market at the priority date. Two standard books on emulsifiers were available at the oral proceedings to verify this point.

Having regard to the fact that the Atlas emulsion explosives were commercially accepted and performed in a satisfactory manner, there would seem to be no incentive for one skilled in the art to seek to modify them in order to solve the problem as defined above and to improve their long term stability, especially by employing one of the PIBSA emulsifiers used in the patent in suit which were not readily available on the market (cf. T 390/88 of 20 February 1990, Reasons, Point 8).

7. Respondent (O4) argued that the essential feature of the patent in suit establishing a relationship between the conductivity and the long term stability was merely a

discovery and thus precluded from patentability by Article 52(2)(a) EPC. In the judgement of the Board this is not the case. The Appellant may indeed have made a discovery but in addition it has been shown that the discovery can be applied in order to develop a simple test which can predict whether an emulsion explosive would have long term stability. The application of such a test provides a technical teaching and must be regarded as susceptible of industrial application and thus patentable in terms of Article 52(1) EPC.

10. Since the Board has decided that the patent can be maintained on the basis of the first auxiliary request, the second auxiliary request need not be considered.

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 on the basis of Claims 1 to 17 of auxiliary request 1 as submitted in the oral proceedings and a description to be adapted thereto.

The Registrar: The Chairman:

P. Martorana P. A. M. Lançon