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**Datasheet for the decision
of 21 June 2006**

Case Number: T 1162/01 - 3.3.05

Application Number: 96108969.5

Publication Number: 0747320

IPC: C01B 25/08

Language of the proceedings: EN

Title of invention:

Composition for producing phosphine-containing gas

Applicant:

Degesch de Chile Ltda

Opponent:

-

Headword:

Composition for producing phosphine/DEGESCH

Relevant legal provisions:

EPC Art. 84, 54, 56

Keyword:

"Clarity - yes"
"Novelty - yes"
"Inventive step - yes"

Decisions cited:

-

Catchword:

-



Case Number: T 1162/01 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 21 June 2006

Appellant:

Degesch de Chile Ltda
Camino Antiguo a Valparaiso no. 1321
Penafior Casilla: 2404 Correo Central
Santiago (CL)

Representative:

Fuchs Mehler Weiss & Fritzsche
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Decision under appeal:

Decision of the Examining Division of the
European Patent Office posted 17 May 2001
refusing European application No. 96108969.5
pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: M. Eberhard
Members: E. Waeckerlin
S. Hoffmann

Summary of Facts and Submissions

- I. The appeal was lodged against the decision of the examining division to reject the European patent application No. 96 108 969.5.

The decision was based on the set of amended claims 1 to 20 according to the main request and claim 1 according to the auxiliary request, both submitted with letter dated 4 April 2000.

- II. In the decision of the examining division the following reasons were given:

The claims according to both the main and auxiliary request did not meet the requirements of Article 123(2) EPC, because the term "dust-free" relating to the finely divided metal used as one of the starting materials and referred to in claims 6 and 10 did not have a basis in the application as originally filed. Furthermore no direct basis existed for claiming any use of the metal phosphide composition within the broad terms of claim 16.

It was stated in the decision that even in the case that the objections under Article 123(2) EPC could be overcome, the claimed invention would still not meet the requirements of Article 52(1) EPC. The decision referred to specific paragraphs of the examining division's communications and concluded that the process for the manufacture of a metal phosphide composition according to claims 10 to 15 of the main request did not involve an inventive step.

Having regard to the claims directed to the metal phosphide compositions according to the main request, the decision referred also to the communications issued by the examining division, stating *inter alia* that the claimed compositions lacked novelty in respect of GB-A-2 062 602 (D1) or GB-A-2 097 775 (D2). The composition of claim 1 according to the auxiliary request was considered to lack an inventive step.

- III. The appellant submitted together with the grounds of appeal two sets of amended claims as a main and an auxiliary request. In his view Article 123(2) EPC had been applied wrongly. Therefore he asked for the refund of the appeal fee. With regard to the merits of the case he presented extensive arguments relying, *inter alia*, on a declaration by an expert, Dr. H. Hahn, submitted during the examination procedure on 12 February 1999.
- IV. In a communication the board expressed various reservations under Article 123(2) EPC against claims 7, 10 and 17 of the main request and claims 8 and 12 of the auxiliary request. Furthermore objections regarding lack of clarity (Article 84 EPC), lack of novelty and lack of inventive step were raised against certain claims.
- V. In reply to the above communication by the board, the appellant filed three sets of amended claims as a main and two auxiliary requests.
- VI. During the oral proceedings, which were held on 21 June 2006, the appellant submitted an amended set of claims as a main request and withdrew the two auxiliary

requests, so that there remained a single (main) request. Furthermore he withdrew his earlier request directed to the refund of the appeal fee.

Independent claims 1, 9 and 14 of this request read as follows:

"1. A metal phosphide composition ready to be released into liquid water in a hydrolysis process or apparatus for the production of phosphine, comprising solid particles of metal phosphide selected from magnesium phosphide, aluminium phosphide and calcium phosphide and mixtures thereof, characterised in that the composition is in the form of free-flowing loose particles of said metal phosphide and that it is essentially free of metal phosphide dust and that the composition is essentially free of hydrophobic substances in the form of coatings or hydrophobising additives and essentially free of hydrolysis retarding agents and that more than 90% by weight of said particles have a size ranging from 0,1 to 2,5 mm and are hydrolysed in liquid water in less than 3 minutes."

"9. A process for the manufacture of a metal phosphide composition according to claim 1 ready to be used for preparing phosphine by release into liquid water, which phosphine is free of auto-igniting contaminants which comprises reacting between 300 and 600°C a metal, selected from aluminum, calcium and magnesium with yellow phosphorus in an inert gas atmosphere and in the presence of a catalyst, selected from the group consisting of chlorine, bromine, iodine, compounds of any of the foregoing with one of phosphorus, sulphur, hydrogen, zinc, ammonium and the aforesaid metals, and

of water, characterised in that the metal is reacted in a finely divided particulate form more than 90% by weight having a particle size of from 0,1 to 2,5 mm and that throughout the reaction batch and throughout the process, once reacting has commenced, the temperature is maintained within the range of 350°C to 550°C, so that metal phosphide particles produced have essentially the same particle size(s) as the finely divided metal and that the metal phosphide is withdrawn as a free-flowing material of loose particles being further essentially free of metal phosphide dust, essentially free of hydrophobising substance in the form of coating or hydrophobising additives and essentially free of hydrolysis retarding agents and is packaged in a gas-tight dispenser container for use in phosphine generation in such free-flowing condition, for the doping of semiconductors or for fumigation with hydrogen phosphide."

"14. Use of a metal phosphide composition of anyone of claims 1 to 8 for fumigating agricultural or forestry bulk commodities or for space fumigation or for the doping of semiconductors with hydrogen phosphide by exposing the metal phosphide into liquid water, where it hydrolyses in less than 3 minutes when wholly submerged."

VII. During the appeal procedure the appellant presented in particular the following arguments:

According to the process of the invention a free-flowing and dust-free product is obtained by reacting phosphorus with fine metal particles at a temperature which nowhere in the reaction batch and nowhere during

the process is outside the range of 350°C - 550°C, i e never increases above the upper limit of 550°C, neither locally nor temporarily. In contrast to that, no care is taken in the process according to D1 to ensure that the fine metal particles which are used as a starting material are free of dust. Therefore the temperature exceeds locally and temporarily the upper limit of 550°C, even if the process is generally conducted at lower overall temperatures. This causes baking and agglomeration of the particles, so that the resulting particulate product, while being pourable, is neither free-flowing, nor free of dust, nor does it have a defined particle size. In contrast to the products of D1 the metal phosphide compositions according to the invention are free of auto-igniting contaminants when they are hydrolyzed. This allows a safe and rapid hydrolysis. There is no need to coat the surface of the particles with hydrophobic substances as in the case of D1, and no fire retarding agents such as ammonium carbamate have to be added.

Due to the absence of hydrophobising substances and hydrolysis retarding agents the metal phosphide particles according to the invention are hydrolyzed in liquid water within a very short time, so that the hazardous generation of phosphine can be stopped at any time immediately. The free-flowing particulate material of the invention can be poured out of its container and released into water at any desired rate no matter how small the rate is. When the supply of particles to the liquid water is stopped, the released particles are hydrolyzed immediately, and the generation of phosphine is stopped at the same time.

The appellant referred to the expert's declaration filed on 12 February 1999 and pointed out that there had been a prejudice against the invention. Before the invention was made, nobody provided a commercial metal phosphide product for the production of phosphine which had not been phlegmatized, nor did a person skilled in the art even consider using such a product. The prejudice in the art against an unhydrophobised metal phosphide product had been so massive that no manufacturer would ever have allowed such a product to leave the factory in any significant quantity, because of the perceived safety hazards and product liability.

VIII. The appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of claims 1 to 16 as filed during the oral proceedings on 21 June 2006.

Reasons for the Decision

1. *Allowability of the amended claims under Article 123(2) EPC*

Claims 1 to 16 have the following basis in the application as originally filed:

Claim 1: Claim 1 together with claim 8 and page 11, lines 1-5 as originally filed.

Claim 2: Claim 3 as originally filed.

Claim 3: Page 9, line 27 to page 10, line 1 as originally filed.

Claim 4: Claim 4 as originally filed.

Claim 5: Claim 4 as originally filed.

Claim 6: Claim 5 as originally filed.

Claim 7: Claim 6 together with claim 11; page 16, lines 1-3 and lines 11-12 as originally filed.

Claim 8: Claim 9 as originally filed.

Claim 9: Claim 11 together with claims 1, 8 and 14; page 8, lines 12-15; page 10, lines 18-22; page 12, line 14; page 13, line 2 and page 16, line 5 as originally filed.

Claim 10: Claim 12 as originally filed.

Claim 11: Claim 13 as originally filed.

Claim 12: Page 9, line 27 to page 10, line 1 as originally filed.

Claim 13: Claim 4 together with page 16, lines 16-18 as originally filed.

Claim 14: Page 13, line 2; page 14, lines 13-14 and 18; page 11, lines 1-5 and page 12, line 14 as originally filed.

Claim 15: Page 14, lines 19-24 as originally filed.

Claim 16: Page 15, lines 3-4 as originally filed.

2. *Clarity - Article 84 EPC*

- 2.1 According to claims 1 and 9, respectively, the metal phosphide composition is "essentially free of metal phosphide dust", "essentially free of hydrophobic substances in the form of coatings or hydrophobising additives" and "essentially free of hydrolysis retarding agents". Moreover according to claims 7 and 9, respectively, the metal phosphide particles produced have "essentially the same particle size(s)" as the particulate metal. During the examination of the application the examining division has raised an objection under Article 84 EPC against the use of the terms "essentially free" and "essentially" in the

claims, arguing that the scope of said claims was unclear. In order to overcome the objection, the appellant deleted the word "essentially" from the claims, but inserted it again during the oral proceedings before the board in response to a board's question concerning the allowability under Article 123(2) EPC of this deletion. Thus, it has to be investigated whether the use of the term "essentially" in claims 1, 7 and 9 is in conformity with the requirement of clarity laid down in Article 84 EPC.

- 2.2 Having regard to the absence of hydrophobic substances and hydrolysis retarding agents, the appellant has explained at the oral proceedings that such contaminants are undesirable and should be avoided as far as possible. It may nevertheless happen that, inadvertently, traces of such components are present. The word "essentially" used in this context thus reflects the fact that these substances are not intentionally added but might be present in very small amounts as impurities.
- 2.3 Concerning the absence of metal phosphide dust, likewise the metal phosphide dust should be avoided. However, in practice it is very difficult to obtain a product having no dust at all, some dust particles of metal phosphide being always present. The word "essentially" also reflects this situation.
- 2.4 The appellant has further explained that the expression "essentially the same particle size(s)" illustrates the fact that although, ideally, the particle size(s) of the product, i e the metal phosphide, is identical to the particle size(s) of the particulate metal, it

cannot be avoided that minimal deviations occur as a result of the operating conditions during the preparation of the product. The word "essentially" reflects the presence of such minimal deviations.

2.5 The board is of the opinion that under these circumstances the use of the term "essentially" in claims 1, 7 and 9 reflects the technical facts of the case in a proper manner. Therefore, no objection of lack of clarity arises.

3. *Novelty - Article 52(1) and 54 EPC*

3.1 D1 discloses a process for preparing the phosphide of aluminium or magnesium comprising reacting the finely divided metal or an alloy of the two metals with yellow phosphorus at a temperature from 300 to 600°C in an inert gas atmosphere in the presence of a catalyst (see claim 1). The metal may be in finely powdered or grit form (see page 1, lines 72-75; claim 8). According to the examples 1, 3, 4, 6, 9 and 10, respectively, a "grey, gritty product" of magnesium phosphide or aluminium phosphide was obtained by the process. In other words the products had a particulate structure resembling grit. D1 does not give any further details, however, regarding the size(s) of the particles, nor does it disclose that the products have to be essentially free of metal phosphide dust. Therefore the metal phosphide composition of claim 1 differs from the aluminium phosphide or magnesium phosphide of D1 at least by the following two distinguishing features:

- (1) The composition is essentially free of metal phosphide dust; and

- (2) more than 90 % by weight of the particles have a size ranging from 0,1 to 2,5 mm.

- 3.2 D2, which contains a reference to D1 (see page 1, lines 9-20) also describes a process for preparing aluminium- or magnesium phosphide comprising the reaction of the finely divided metal with liquid yellow phosphorus at a temperature from 300 to 600°C in an inert gas atmosphere and in the presence of a catalyst (see page 1, lines 41-47; claim 1). According to example 3 a "gritty" aluminium-magnesium phosphide product was obtained (see page 3, lines 58-60), but as in the case of D1 no details regarding the size(s) of the particles are given, and again there is no disclosure of the feature according to which the product is essentially free of metal phosphide dust.
- 3.3 D3 (WO-A-91/19671) relates to various methods and apparatuses for generating phosphine by the reaction of a metal phosphide formulation, usually based on aluminium phosphide or magnesium phosphide (see page 17, lines 10-15; page 27, lines 5-7), with water (see claims 1, 17 and 18 for methods and claims 6, 19 and 20 for apparatuses). The metal phosphide formulation may be in particulate, tablet or pellet form (see claim 1; page 17, lines 10-16; page 25, line 17). The use of powder formulations is also mentioned, although not preferred (see page 27, lines 8-16). No further information regarding the particle size(s) or other aspects of the morphology of the metal phosphide formulations are given in D3. In particular there is no disclosure that more than 90 % by weight of the particles have a size from 0,1 to 2,5 mm, nor is there an indication that the formulation has to be

essentially free of metal phosphide dust. According to D3 additives to prevent the auto-ignition of the phosphine are normally included in the phosphide formulations (see page 17, lines 18-22).

- 3.4 D5 (EP-A-0 318 040) discloses *inter alia* a cartridge filled with a "a porous mass of metal phosphide", e.g. "a bed of aluminium phosphide or magnesium phosphide powder or granulate" (see page 7, lines 32-34). If an aluminium powder is used, typically technical aluminium phosphide having an AlP content of 85 %, the balance being mainly inert aluminium oxide, the particle size is preferably "mostly in the range 2,5 mm to 0,05 mm" (see page 7, lines 34-36). This is illustrated by example 1, where an aluminium phosphide composition having an AlP content of 85 % is "ground to a particle size such that all of it passes a 2 mm screen. A typical particle size distribution is such that 85 to 95 %, e.g. 92 % passes 1 mm; between 8 and 20 [%], e.g. 12 % is from 0,5 to 1,0 mm; between 20 and 35 %, e.g. 27 %, is from 0,25 to 0,5 mm; between 25 and 40 %, e.g. 33 %, is from 0,1 to 0,25 mm and the balance is finer" (see page 8, lines 14-18). It is however not directly and unambiguously derivable from D5 that the metal phosphide has to be essentially free of metal phosphide dust.
- 3.5 None of the other prior documents discloses a metal phosphide composition which exhibits the two distinguishing features, namely (1) that the composition is essentially free of metal phosphide dust, and (2) that more than 90 % by weight of the particles have a size ranging from 0,1 to 2,5 mm. The metal phosphide composition according to claim 1 is therefore

novel. The same applies to the process for the manufacture of the metal phosphide composition (see independent claim 9) and the use of the composition (see independent claim 14), respectively, since both claim 9 and claim 14 contain a reference to the metal phosphide composition of claim 1.

4. *Inventive step - Article 52(1) and 56 EPC*

4.1 With regard to the product claim 1, D1 can be regarded as the closest prior art. It relates to the preparation of magnesium phosphide or aluminium phosphide in particulate form. D1 does not expressly mention the addition of hydrophobic substances in the form of coatings or hydrophobising additives, or of hydrolysis retarding agents to these products. However, as has been credibly stated by the appellant in writing and at the oral proceedings, the products of D1 were treated with a hydrophobic substance before their use in the production of phosphine by hydrolysis. This is confirmed by the declaration of Dr. H. Hahn submitted on 12 February 1999, according to which all metal phosphide products commercially available in the state of the art and also the metal phosphides manufactured as described in D1 and D2 were in a form compounded with substantial amounts of additives designed to retard and inhibit hydrolysis reactions of the phosphides and to prevent autoignition (see declaration, page 7, paragraph 2.8; pages 13-14, paragraph 2.12.4). Similar information can be inferred from page 7 of the application as filed in connection with D2.

4.2 Starting from the closest prior art D1, the technical problem underlying the present invention can be seen in

providing a metal phosphide composition suitable for the production of phosphine-containing gas by hydrolysis, which delivers a phosphine gas having no or no appreciable tendency to autoignite (see patent application, page 6, third paragraph).

4.3 It is proposed to solve the technical problem set out above by providing a composition of magnesium phosphide, aluminium phosphide or calcium phosphide, or mixtures thereof, which exhibits in particular the following features in combination:

(1) the composition is essentially free of metal phosphide dust;

(2) more than 90 % by weight of the metal phosphide particles have a size ranging from 0,1 to 2,5 mm; and

(3) the composition is essentially free of hydrophobic substances in the form of coatings or hydrophobising additives, and it is also essentially free of hydrolysis retarding agents.

(see claim 1).

4.4 According to the description the claimed metal phosphide composition permits the safe production of phosphine gas by exposure to liquid water. The phosphine formed is free of autoigniting contaminants, and the gas mixture formed has no tendency to autoignite (see description, page 8, second and third paragraphs; page 9, second to fourth paragraphs; page 18, example, first paragraph, lines 13-15). As the appellant has explained at the oral proceedings, there is no need to phlegmatise the product after its

formation, e.g. by impregnation and coating with a hydrophobic substance, before any further handling or before storage prior to use (see, in this context, description, page 7, second paragraph; page 9, second paragraph). These results were confirmed at the oral proceedings by the expert, Dr. H. Hahn, who is also the author of the declaration submitted on 12 February 1999.

- 4.5 In view of the findings set out above the board is satisfied that the technical problem has been credibly solved by the product according to claim 1.
- 4.6 It remains to be investigated whether the claimed solution was obvious to a skilled person.

At the oral proceedings both the appellant and the expert, Dr. H. Hahn, explained that the presence of metal phosphide dust was partly responsible for the self-ignition and pointed out that D1 does not disclose that the absence of metal phosphide dust is a decisive factor in the inhibition of self-ignition. The board sees no reason not to accept this explanation. The board can also accept the statement made at the oral proceedings by the appellant and confirmed by Dr. Hahn, according to which the skilled person, when reading D1, would conclude that the technical teaching of this document relates to the manufacture of aluminium phosphide or magnesium phosphide as such, but not to the preparation of metal phosphide compositions which can be used directly, i.e. without further treatment, for the generation of phosphine by hydrolysis. In order to produce a composition ready to be released into liquid water in a hydrolysis process for the production of phosphine, the metal phosphines obtained by the

process of D1 were impregnated with a hydrophobic substance, normally 3,5 % by weight of paraffin wax, immediately after having been withdrawn from the reactor and while still being hot. These additives were accepted as a necessary and unavoidable evil for safety reasons (see declaration by Dr. H. Hahn submitted on 12.02.1999, pages 13 to 14, paragraph 2.12.2). The board can accept the appellant's argument according to which it was standard common practice world-wide to treat metal phosphides with hydrophobic substances in order to produce metal phosphide compositions, thereby protecting the products against spontaneous and uncontrollable hydrolysis involving the risk of autoignition. In fact it would appear that even after the date of filing of the present application it was common ground among the skilled persons that the presence of hydrophobic substances was imperative. Thus, "Ullmann's Encyclopedia of Industrial Chemistry" contains the following statement in chapter 4, "Phosphides": *"The products [i e metal phosphides] are protected against uncontrollably rapid hydrolysis, such as could occur by accidental contact with water, by coating their surfaces with hydrophobic substances such as paraffin or metal stearates. By mixing the material with ammonium carbamate, which produces ammonia and carbon dioxide on decomposition, the possible spontaneous ignition of the PH₃ produced is prevented."* (See D4 = Ullmann's Encyclopedia of Industrial Chemistry, Sixth Edition, 1998 Electronic Release, Weinheim, 1998 or the corresponding statement in the printed edition of Ullmann's Encyclopedia of Industrial Chemistry, Sixth edition, Weinheim, 2003, vol. 26, page 205, left column, second paragraph.)

4.7 D1 contains no information suggesting on the one hand that the presence of metal phosphide dust might be one reason for the autoignition of the phosphine-containing gas, nor that the technical problem stated above might be solved without the addition of hydrophobic substances and hydrolysis retarding agents.

To arrive at the claimed solution the skilled person had to go against the usual practice of coating the metal phosphide with hydrophobic substances. According to page 9, paragraphs 3 and 4 of the description it is conceivable that contaminants which on hydrolysis give rise to autoignitable phosphorus compounds may be formed during the impregnation by some unknown reaction between the hot metal phosphide and the hydrophobic substance. This is in no way suggested in D1 or in the other cited documents, let alone that the usual coating with hydrophobic substances could be omitted while still avoiding the tendency to auto-ignite. Therefore, neither D1 nor the other cited documents, taken alone or in combination with D1, pointed towards the claimed solution.

4.8 Like D1, D2 is concerned with a process for the production of aluminium or magnesium phosphide (see page 1, lines 6-8; claim 1). It provides a teaching which corresponds to the teaching of D1 as set out above.

4.9 D3 addresses *inter alia* the problem of providing an improved method and apparatus for generating phosphine in a controlled way (see page 6, lines 8-10). This problem is solved by reacting a metal phosphide with liquid water, whereby the phosphine generation

technique involves either the enclosure of the phosphide formulation in a specific housing, which is closed by a membrane that is permeable to water and phosphine (see page 7, lines 1-6 and 25-28), or the periodic addition of a small quantity of the phosphide formulation to an excess of water (see page 8, lines 8-10). The metal phosphide formulations used in the method of generating phosphine are in particulate form or in the form of tablets or pellets (see page 11, line 21; page 12, line 17; page 13, lines 8-9; page 17, lines 15-16; page 25, line 17; page 27, lines 4-19; page 30, lines 20-21). Document D3 does not provide any details regarding the size(s) or the size distribution of the metal phosphide particles. On the other hand the document discloses that, normally, additives will be included in the phosphide formulation to prevent the auto-ignition of the phosphine produced by the reaction of the metal phosphide with water (see page 17, lines 19-22).

- 4.10 D5 relates *inter alia* to the problem of generating phosphine from metal phosphides while preventing the formation of an ignitable mixture of phosphine gas (see page 3, lines 28-29). According to D5, effective control over the phosphine gas concentration in the generator space is achieved by virtue of the moisture content of the flow being maintained at a given level (see page 3, lines 37-38). D5 also discloses an apparatus for carrying out the phosphine generating process, as well as a replaceable cartridge filled with "a porous mass of metal phosphide", for example a bed of aluminium phosphide or magnesium phosphide in powder or granulate form (see page 7, lines 32-34). In example 1 details regarding the size distribution of

the aluminium phosphide particles are given (see page 8, lines 15-18), but it is neither disclosed nor suggested that the composition should be essentially free of metal phosphide dust in order to avoid the tendency to auto-ignite. It is stated in example 1 of D5 that a portion of the particles (the balance to 100 %) is finer than 0,1 mm (see page 8, line 18), which means that the formulation contains a non-negligible amount of metal phosphide dust.

- 4.11 D8 (DE-A-1 122 762) is concerned with the problem of protecting metal phosphide against moisture in order to prevent the untimely generation of phosphine (see column 1, lines 7-11; 32-36). The proposed solution to this problem is to cover the individual particles or small groups of particles with a compact coating of a solid hydrophobic organic substance, to add a thermally decomposable substance, and to bring the material into the form of compacts (see column 2, lines 37-44; claim 1). No details regarding the morphology of the particles of metal phosphide, in particular the particle size(s) or the size distribution, are given in D8, and the presence of a hydrophobic organic substance is compulsory, which goes in a direction opposite to the claimed one.
- 4.12 Accordingly the metal phosphide composition of claim 1 cannot be considered as being obvious to a person skilled in the art having regard to the cited prior art. Therefore the claimed composition involves an inventive step as required by Articles 52(1) and 56 EPC.
- 4.13 The same applies to claims 2 to 8, since these claims are dependent on claim 1.

4.14 Both the independent process claim 9 and the use claim 14, respectively, contain a reference to claim 1. Thus, novelty and inventive step of the subject-matter of these claims are imparted by claim 1.

4.15 Claims 10 to 13 depend on claim 9, and claims 15 to 16 depend on claim 14. The patentability of the subject-matter of these claims is carried by the independent claims to which they refer.

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 grant a patent on the basis of claims 1 to 16 submitted during the oral proceedings and a description with drawings to be adapted.

The Registrar:

The Chairman:

C. Vodz

M. Eberhard