BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

Internal distribution code:
(A) [ ] Publication in OJ
(B) [ ] To Chairmen and Members
(C) [X] To Chairmen
(D) [ ] No distribution

Datasheet for the decision of 10 July 2007

Case Number: T 0325/03 - 3.3.05

Application Number: 96109145.1

Publication Number: 0747319

IPC: C01B 25/06

Language of the proceedings: EN

Title of invention:
Process and apparatus for producing phosphine-containing gas

Patentee:
Degesch de Chile Ltda

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56, 82, 84, 123(2)

Keyword:
"Novelty and inventive step (yes; after amendment)

Decisions cited:
-

Catchword:
-

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0325/03 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 10 July 2007

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

Representative: Fuchs Mehler Weiss & Fritzsche
Patentanwälte
Postfach 46 60
D-65036 Wiesbaden (DE)

Decision under appeal: **Decision of the examining Division of the European Patent Office posted 15 October 2002 refusing European application No. 96109145.1 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: M. Eberhard
Members: H. Engl
S. Hoffmann

Summary of Facts and Submissions

I. This appeal lies against the decision of the examining division refusing European patent application 96109145.1.

The decision is based on the grounds that the claims in accordance with the main and the first auxiliary request then on file contravened Articles 82, 54 and 123(2) EPC; and that the claims in accordance with the second and third auxiliary requests contravened Article 123(2) EPC.

II. The following documents have been cited in the search report:

D1: GB A 2 062 602
D2: GB A 2 097 775
D3: WO-A-91/19671
D4: WO-A-93/25075
D5: EP-A-0 318 040

III. The applicant (henceforth: the appellant) lodged an appeal against the decision of the examining division and filed a new claim 2 dependent on claim 1. Arguments on novelty and inventive step were also submitted with the grounds of appeal.

IV. In the annex to the summons for oral proceedings the board raised a number of objections under Article 123(2) EPC against the claims of the main request. The independent apparatus claims 12 and 24 were objected to as lacking novelty and inventive step having regard to document D3.

V. In reply to the said summons the appellant submitted on 6 June 2007 new claims as a main and an auxiliary request and a second declaration by Dr Hahn.

VI. Oral proceedings took place on 10 July 2007. The appellant filed two sets of amended claims as a main and an auxiliary request in replacement of the previous ones.

VII. The independent claims in accordance with the said main request read as follows:

"1. A process for generating phosphine to form a mixture thereof with a diluent gas or gases, wherein a hydrolysable metal phosphide, selected from the group consisting of magnesium phosphide, aluminium phosphide and calcium phosphide is contacted with liquid water in a generating space, whereby the metal phosphide is hydrolysed to release phosphine which is withdrawn from the generating space and is diluted from the time of its generation to its reaching its locality of use with a diluent gas to a composition which is non-ignitable under the conditions of use, characterised in that the metal phosphide used is composed of loose metal phosphide particles essentially free of metal phosphide dust and hydrolysis retarding agent and essentially free of a hydrophobic substance in the form of coatings or hydrophobising additives and that it is conveyed in or through a carrier gas inert to phosphine and inert to the metal phosphide and is released in a free-flowing particulate form into the liquid water, said metal phosphide being maintained in such carrier gas before entering the water."

"12. A phosphine generator, suitable for carrying out the process as claimed in any one of the preceding claims, which comprises a phosphine generating chamber containing liquid water, having an inlet for admitting a hydrolysable metal phosphide composition into the water in the generating chamber, a gas outlet adapted to discharge the phosphine or a mixture of it with gas inert thereto from the generating chamber and feed means adapted for feeding the metal phosphide through the inlet at a controlled rate, characterised in that the feed means (1) adapted to feed the metal phosphide in a free-flowing particulate form, composed of loose metal phosphide particles."

In the said auxiliary request claims 1 - 11 directed to a process for generating phosphine are identical with those of the main request.

Independent apparatus claim 12 in accordance with the auxiliary request reads as follows:

"12. A phosphine generator, suitable for carrying out the process as claimed in any one of the preceding claims, which comprises a phosphine generating chamber containing liquid water, having an inlet for admitting a hydrolysable metal phosphide composition into the water in the generating chamber, a gas outlet adapted to discharge a mixture of phosphine with gas inert thereto from the generating chamber and feed means adapted for feeding the metal phosphide through the inlet at a controlled rate, characterised in that the feed means (1) includes a pneumatic entrainment device for the particulate metal phosphide composition and that the pneumatic entrainment device includes a receptacle (3)

for the particulate metal phosphide composition, a gas inlet (11) in its bottom region for a carrier gas leading to a venture device (10), followed by a powder forwarding duct (8, 13, 31) leading to the inlet for admitting the metal phosphide composition into the water, preferably including a powder feed rate regulator device."

VIII. The appellant's arguments may be summarized as follows:

D3 did not disclose an apparatus suitable for holding particulate metal phosphide in a free-flowing condition and feeding the same continuously at an accurately determined rate into the water. The free-flowing property of the particulate metal phosphide, resembling a flowing liquid, was a key element to the invention. The skilled person would also implicitly understand that in present claim 12 of the main request a continuous feed of the material was envisaged. In contrast, the closest prior art of D3 used tablets or pellets of metal phosphide which were batchwise, hence intermittently and discontinuously, added to the water. The claimed phosphine generator, in particular the feed means thereof, had to be specially adapted to exploit the free-flowing property and would thus be distinguished from the apparatus disclosed in D3.

Having regard to D1 and D2, the appellant relied on the first declaration by Dr Hahn, and argued that the phosphides produced in D1 and D2 were not free-flowing and not dust free. They had been treated with a hydrophobic agent and could not be conveyed by a carrier gas. The tablets or pellets used in D3 invariably contained a hydrophobic binder and therefore hydrolysed

quite slowly, making it impossible to stop phosphine evolution immediately after a shut off. In contrast, the highly reactive metal phosphide particles fed into the water in the claimed process and apparatus are hydrolysed in a very short time, due to their being essentially free of metal phosphide dust and hydrolysis retarding agent and essentially free of a hydrophobic substance in the form of coatings or hydrophobising additives. This fundamental difference involved an inventive step, because it departed drastically from the principles of safety of the prior art.

With respect to apparatus claim 12 of the auxiliary request, the appellant argued that the prior art did not disclose or even suggest a pneumatic entrainment device using a carrier gas to convey free-flowing metal phosphide particles into the generating chamber. Prior art devices, designed for metal phosphide pellets or tablets, or paste-like formulations, could not handle the free-flowing, particulate material used in the claimed invention.

IX. The appellant requested that the decision under appeal be set aside and a patent be granted on the basis of claims 1 - 27 of the main request or in the alternative claims 1 - 20 of the auxiliary request both filed during the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

2. Main request, apparatus claim 12: Article 84 EPC

The phosphine generator defined in said claim 12 comprises the following features:

- a phosphine generating chamber containing liquid water,
- the generating chamber having an inlet for admitting a hydrolysable phosphide composition into the water in the generating chamber,
- a gas outlet adapted to discharge the phosphine or a mixture of it with gas inert thereto from the generating chamber, and
- feed means adapted for feeding the metal phosphide through the inlet at a controlled rate, said feed means (1) being adapted to feed the metal phosphide in a free-flowing particulate form, composed of loose metal phosphide particles.

These features in combination are known from document D3. See description, page 22, line 14 - page 23, line 30, page 25, lines 13 to page 26, line 16, and in particular Figure 5, wherein reference numeral 23 depicts a hopper containing a particulate phosphide formulation in the form of tablets or pellets, said hopper together with disc member 25, aperture 32 of the disc and discharge point 33 of the hopper constituting a feed means for feeding the metal phosphide, at a controlled rate, through the inlet of a phosphine generating chamber 24 containing liquid water and having an inlet port 26, and a phosphine gas outlet at 36, the generating chamber further having a gas inlet 43 for nitrogen gas.

The appellant's argument that the apparatus disclosed in D3 would not be suited to hold particulate metal

phosphide in a free - flowing condition and to feed the same continuously at an accurately determined rate into the water is not convincing. Reference is made to page 27, lines 8 - 16 of D3, stating that even a powdered phosphide formulation could be used in hopper 33. This is, however, not preferred, because a constant supply of the phosphide formulation to the water chamber 24 turned out not to be possible over a period of 7 to 28 days. In the board's opinion, the passage confers the implicit teaching that for shorter periods of operation, the phosphide generator does work with powdered formulations. The board also observes that claims 1 and 12 do not define the size of the particles of the metal phosphide; according to the description, a preferred composition is composed of particles of which 90 % by weight are in the particle size range of from 0.1 to 2.5 mm. Therefore, particles having a dimension greater than 2.5 mm may also be used.

The board notes that the phosphine generator defined in claim 12 of the main request, is claimed to be "*suitable for carrying out the process as claimed in any one of the preceding claims*" (*i.e.*, process claims 1 - 11). It is stated in said process claims that the metal phosphide composed of loose particles *"is conveyed in or through a carrier gas inert to phosphine and inert to the metal phosphide"* and that *"the metal phosphide is maintained in such carrier gas before entering the water"* (see claim 1). Therefore, claim 12 has to recite the additional apparatus features which are essential to render the claimed apparatus suitable for carrying out the process.

Independent claim 12, however, fails to clearly define those essential elements for carrying out the process of claim 1, in particular the means or the device to be used for conveying the loose particles of metal phosphide into the generator and into the water by means of a carrier gas. Consequently, the claim does not meet the requirements of Article 84 EPC. See also Case Law of the Boards of Appeal, 6th Edition 2006, page 189, chapter II.B.1.1.3.

The claim is therefore not allowable and the main request must be rejected.

3. Auxiliary request

3.1. *Amendments*

Claim 1 of the auxiliary request is based on originally filed claim 1 and the description, page 7, paragraphs 3 and 4; page 8, last paragraph; the paragraph bridging pages 23 and 24 and Figure 1.

Claim 2 is based on original claim 3 and the description, page 2, second paragraph, and page 19, last paragraph.

Claim 4 is based on the description, page 9, second paragraph.

Claims 3 and 5 - 11 correspond to originally filed claims 2 and 4 - 10, respectively.

Claim 12 is based on originally filed claims 11 and 18 and on Figure 1. Claims 13 - 16, 18 - 20 correspond to original claims 19 - 22, 24 - 26. Claim 17 is based on the description, page 15, lines 8 - 15; page 16, line 21 - page 17, line 7; and page 17, lines 18, 19.

3.2. *Clarity*

The board accepts the originally filed wording *"essentially free of metal phosphide dust"* and *"essentially free of a hydrophobic substance"* in claim 1. The term *"essentially"* used in the context of the claim merely reflects the technical fact that unavoidable traces of dust, hydrolysis retarding agents and hydrophobic substances may be present, but should be avoided as far as possible. Therefore, no objection under Article 84 EPC arises.

3.3. The set of claims in accordance with the auxiliary request contains only one independent process and apparatus claim. The objections under Articles 82 and 84 EPC raised by the examining division under point 1 of the contested decision no longer apply to the amended claims.

4. *Novelty*

4.1. Documents D1 and D2 describe a process for preparing aluminium or magnesium phosphide, but do not disclose information about processes of generating phosphine gas from metal phosphides by hydrolysis. Furthermore, there is no disclosure of a product essentially free of metal phosphide dust.

Document D3 reveals an apparatus and a process for the generation of phosphine gas from metal phosphides. In accordance with one embodiment described on pages 12 and 13; on page 22, line 20 - page 23, line 30; page 25, line 13 - page 26, line 16; and in Figure 5; there is disclosed an apparatus comprising a gas generating vessel, the lower part of which (water chamber 24) contains liquid water; a hopper 23 for supplying metal phosphide in the form of tablets or pellets; a feed mechanism (disc member 25, aperture 32 and discharge point 33) for said pellets or tablets; a gas inlet 43 for a gas, preferably nitrogen, to stir the water; and a gas outlet 36 for the phosphine and the said gas. The pellets dispensing system is capable of supplying pellets or tablets at a predetermined rate. There is no indication in D3 that the metal phosphide formulation has to be essentially free of metal phosphide dust. Additives for preventing auto-ignition of the phosphine are normally included in the phosphide formulations according to D3 (see page 17, lines 10 - 22).

Document D4 deals with metal phosphide formulations and a method and apparatus for the controlled generation of phosphine. The particulate metal phosphide is formulated into an intimate mixture with a water immiscible compound which is inert to the phosphide, preferably a low melting point wax, petroleum jelly or a non-volatile oil. The mixture has the consistency, at the temperature of use, of an extrudable paste or thick slurry. This paste formulation is then forced by a plunger or piston action from a cartridge through a pipe and thus periodically introduced into a water bath in a reaction chamber (see page 13, lines 1 - 20; page 19, lines 9 - 30; claims 1, 2 and 10; Figure 5).

Document D5 discloses a process and an apparatus for fumigating an enclosed environment with a pesticidal gas mixture of phosphine gas and air. It describes *inter alia* a cartridge filled with a porous mass of metal phosphide, for instance a bed of aluminium phosphide or magnesium phosphide powder or granulate. Phosphine is generated. The metal phosphide is not hydrolyzed by introduction into liquid water, but by contact with a stream of moist air which is passed through said cartridge. The metal phosphide may be free of autoignition inhibiting substances, but it is not directly and unambiguously derivable from D5 that the metal phosphide has to be essentially free of metal phosphide dust. See abstract; page 3, lines 30 - 36; page 4, lines 8 - 10; page 7, lines 32 - 34; page 8, lines 10 - 20; claim 1; Figures 1 and 3.

4.2. The process of claim 1 in accordance with the auxiliary request differs from the processes disclosed in any of documents D1 to D5 at least in that the particulate metal phosphide is conveyed in or through a carrier gas inert to phosphine.

4.3. Since none of the cited documents discloses feed means including a pneumatic entrainment device for the particulate metal phosphide composition, the subject matter of apparatus claim 12 is also novel.

5. *Inventive step*

5.1. Starting from D3 as the closest prior art, the problem underlying the application may be seen in providing a process of generating phosphine gas from metal phosphide

particles and water wherein the production of phosphine can be stopped in a relatively short time and in which the phosphine gas has no tendency to autoignite (*cf*. description, page 5, second paragraph; page 6, first paragraph).

According to the description (page 8, second full paragraph; page 9, second paragraph), the highly reactive, free - flowing particulate metal phosphide essentially free of metal phosphide dust and hydrolysis retarding agent and essentially free of a hydrophobic substance in the form of coatings or additives reacts almost immediately with the water. Moreover, the claimed process employs a metal phosphide free of impurities which in hydrolysis liberate autoignitable phosphine homologues and derivates and is thus safe to use. The appellant has plausibly argued that the use of a carrier gas for conveying the particulate free - flowing metal phosphide facilitates the addition of small quantities at a controlled rate, thus making it possible to stop the phosphine generation very rapidly if necessary. It is therefore credible that the above defined technical problem has indeed been solved.

5.2. The claimed solution, as defined in claim 1, is considered to involve an inventive step, as the above discussed prior art does not provide a pointer towards the use of a carrier gas inert to the phosphine and to the metal phosphide for conveying the loose particles of metal phosphide into the generator and into the water, in order to solve the problem posed. Documents D1 and D2 do not suggest any particular means at all for feeding metal phosphide particles into water. Other feed means disclosed in the prior art are not pneumatic, but

mechanical: Rotating or sliding discs are disclosed in D3, a cartridge and piston in D4. However, document D4 does not employ free-flowing particulate metal phosphides, but a slurry- or paste-like formulation. D5 does not teach to hydrolyze the phosphide, contained in a cartridge, in liquid water, but by contact with a stream of moist air. Because of these differences, there is no reason why the skilled person should combine D3 with the teaching of document D4 or D5; and even a hypothetical combination of the said documents would still not lead to the claimed process.

The subject matter of apparatus claim 12, more specifically, a phosphine generator comprising a pneumatic entrainment device for the particulate metal phosphide composition, is also not suggested by the cited prior art.

The dependent claims define preferred embodiments of the claimed process and apparatus and derive their patentability from the independent claims to which they refer.

5.3. The subject matter of the claims in accordance with the auxiliary request thus involves an inventive step.

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 20 of the auxiliary request filed during oral proceedings and a description/drawings to be adapted thereto.

The Registrar: The Chairman:

C. Vodz M. Eberhard