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Datasheet for the decision of 5 February 2008

Case Number: T 0184/05 - 3.2.07

Application Number: 98105741.7

Publication Number: 0869203

IPC: C30B 29/12

Language of the proceedings: EN

Title of invention:
Calcuim Fluoride crystal, optical article and production method

Patentee:
CANON KABUSHIKI KAISHA

Opponent:
Schott Lithotec AG

Headword:
-

Relevant legal provisions:
EPC Art. 111(1), 123(2), 123(3), 54, 56
RPBA Art. 13

Relevant legal provisions (EPC 1973):
-

Keyword:
-

Decisions cited:
G 0004/88, T 0201/83, T 0191/93, T 0677/05

Headnote:
An impurity concentration value of a product obtained under specific process conditions cannot be taken in strict isolation from the examples unless it has been demonstrated that this value is not so closely associated via the applied process with specific (undisclosed) maximum values of all other impurities comprised in the product (see points 1.1.1 to 1.1.6 of the reasons)

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Case Number: T 0184/05 - 3.2.07

D E C I S I O N
of the Technical Board of Appeal 3.2.07
of 5 February 2008

Appellant: (Opponent)
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Decision under appeal:
Interlocutory decision of the Opposition Division of the European Patent Office posted 30 November 2004 concerning maintenance of European patent No. 0869203 in amended form.

Composition of the Board:

Chairman: H. Meinders
Members: H. Hahn
E. Lachacinski

Summary of Facts and Submissions

I. The joint opponents Schott Lithotec AG and Schott Glas lodged an appeal against the interlocutory decision of the Opposition Division to maintain European patent No. 0 869 203 in amended form.

II. An opposition had been filed against the patent as a whole under Article 100(a) EPC on the grounds of lack of novelty and inventive step, under Article 100(b) EPC on the ground of insufficient disclosure and under Article 100(c) EPC for extending beyond the content of the application as originally filed.

The Opposition Division held that claims 1 and 2 of the main request dated 25 October 2004 contravened Article 123(2) EPC while claim 3 contravened Rule 57a EPC. Claim 1 of the first auxiliary request dated 25 October 2004 was identical with that of the main request and thus likewise contravened Article 123(2) EPC. Process claim 2 of the second auxiliary request as filed at the oral proceedings before the Opposition Division on 16 November 2004 was considered to meet the requirements of Articles 100(b) EPC but not to meet the requirements of Article 56 EPC since it did not contain sufficient features for solving the technical problem of manufacturing the calcium fluoride crystal of claim 1. Claims 1-7 according to the third auxiliary request also filed at those oral proceedings were considered to meet all the requirements of the EPC, particularly of novelty and inventive step with respect to D1 (= US-A-2 498 186), D2 (= DD-PS-213 514), D4 (= US-A-2 550 173) and D6 (= Arkhangelskaya et al.: "Absorption and luminescence of Pb^{2+} ions in alkaline-

earth fluoride crystals", Opt. Spektrosk. (USSR) 47(4), Oct. 1979, 708-715).

III. Claim 1 as maintained by the Opposition Division reads as follows:

"1. A calcium fluoride crystal containing at least one kind of atom selected from the group consisting of Zn, Cd, Pb, Li, Bi and Na with a total content of 10 ppm or less, containing oxygen with a content of 50 ppm or less, and having an internal transmittance of 70% or more with respect to a 135 nm wavelength light, the internal transmittance being a value determined by subtracting the light absorbance (%) of a bulk from 100%, wherein the light absorbance is a value obtained based on a light absorption coefficient of a sample to be measured having a thickness of 10 mm, wherein said Zn is present in said calcium fluoride crystal and has a content of less than 0.5 ppm."

IV. With a communication dated 29 October 2007 and annexed to the summons to oral proceedings the Board presented its preliminary opinion based on claims 1-7 of the then single request of the respondent (patent proprietor) dated 11 November 2005, being identical with claims 1-7 of the third auxiliary request as maintained with the decision under appeal.

On the procedural aspects of the case, i.e. the request to register Schott AG in place of Schott Glas as opponent, the Board remarked that from the Commercial Register extracts HRB 8555 and HRA 2724 as filed by the appellant, it cannot be derived that the assets of Schott Glas have been entirely transferred to Schott AG.

Entry 3 of HRB 8555 mentions that the owing Carl Zeiss Stiftung retained certain assets in the transfer of Schott Glas to Schott AG, so that it cannot be concluded that all assets of Schott Glas have been transferred. Thus the requirements as established by G 4/88 (OJ EPO 1989, 480) did not appear to be met. Entry 70 of HRA 2724 contained the same information as entry 3 of HRB 8555. From case T 0677/05 (EP-99111917.3), treated by this Board and also involving Schott Glas as opponent, the Board had knowledge of further documents, annexed to the communication, indicating that Schott Glas no longer existed. It appeared therefore that the proceedings would have to be continued with the remaining opponent, Schott Lithotec AG only.

The Board considered that claim 1 appeared to contravene Article 123(2) EPC since the value "**<0.5 ppm Zn**" seemed to represent an intermediate generalization of embodiments originally disclosed only in a more specific form. Furthermore, the feature of claim 1 "containing at least one kind of atom selected from the group consisting of Zn, Cd, Pb, Li, Bi, and Na with a **total** content of 10 ppm or less, containing oxygen with a content of 50 ppm or less" had no explicit basis in the application as originally filed and seemed also not to be directly and unambiguously derivable therefrom. Therefore the request appeared to fail the requirements of Article 123(2) EPC.

The Board then stated that novelty would be dealt with for a formally allowable request. In this context the Board remarked that the product of claim 1 is not (and cannot be) considered restricted to a CaF_2 crystal

having been obtained by a specific process using a ZnF_2 scavenger, i.e. the crystal having the claimed Zn content of less than 0.5 ppm can be obtained by applying another purification process according to the state of the art using e.g. a PbF_2 scavenger (such as disclosed in D1 or D2 and most presumably also in D6), as it is evident that such a produced CaF_2 crystal will contain at least some Zn atoms as impurities, particularly when considering that Pb compounds always contain Zn (stemming from the minerals used for producing Pb and its compounds). This also holds when considering that the synthetic highly purified CaF_2 raw material used for further refining and then growing the CaF_2 crystal contains a very small amount of Zn as an impurity. The major part of said Zn impurity, however, will be removed during the purification steps in high vacuum so that the resulting CaF_2 crystal will have a Zn impurity content being well below said value of the precursor material, i.e. well below 1.0 ppm.

The Board further remarked that the issue of inventive step would be dealt with for a formally allowable request taking into consideration the problem-solution approach and that it seemed that D2 could be considered as the closest prior art.

In the opinion of the Board, the process of D2 includes the addition of a scavenger (PbF_2) in an amount of 0.2-2 wt.% with respect to CaF_2 in the crystal growth step under vacuum and comprises a subsequent tempering step in a PbF_2-containing atmosphere (such tempering is similarly carried out according to the examples of the contested patent but in an atmosphere containing ZnF_2). Said range of 0.2-2.0 wt.% PbF_2 according to D2

corresponds to 0.064-0.64 mol% which overlaps with the range of from 0.04-0.1 mol% for the first refining step according to claim 1. Thus it needed to be discussed whether or not the skilled person would modify the known processes in a specific manner in order to improve the known products and/or to use a specific different scavenger in order to better deal with environmental regulations.

The parties were given the opportunity to file observations to the communication which should be filed well in advance, i.e. at least one month, before the date of the oral proceedings in order to give sufficient time to the Board to prepare for the oral proceedings.

Finally, the parties were advised to take note of the amended Rules of Procedure of the Boards of Appeal, in force as of 1 May 2003 and particularly of Article 10b.

V. In its opposition the appellant also relied on an alleged prior use of the product as claimed in the patent in suit, which was not followed up by the Opposition Division, and reiterated this objection on appeal, filing further supporting evidence.

With letter dated 7 January 2008 the respondent filed a new main request and auxiliary requests 1 to 6 together with further arguments.

VI. Oral proceedings before the Board were held on 5 February 2008.

(a) The appellant requested that the decision under appeal be set aside and the patent be revoked.

(b) The respondent requested that the appeal be dismissed, auxiliarily that the patent be maintained on the basis of the main request filed at the oral proceedings or one of the auxiliary requests 1 to 5, filed with letter of 7 January 2008, or 5a, 6 filed at the oral proceedings.

VII. Claim 1 according to auxiliary request 1 reads as follows (amendments compared to claim 1 of the main request are in bold with deletions between brackets, emphasis added by the Board):

"1. A calcium fluoride crystal containing **atoms constituting a scavenger with a content of 10 ppm or less [at least one kind of atom selected from the group consisting of Zn, Cd, Pb, Li, Bi and Na with a total content of 10 ppm or less]**, containing oxygen with a content of 50 ppm or less, and having an internal transmittance of 70% or more with respect to a 135 nm wavelength light, the internal transmittance being a value determined by subtracting the light absorbance (%) of a bulk from 100%, wherein the light absorbance is a value obtained based on a light absorption coefficient of a sample to be measured having a thickness of 10 mm, wherein **[said]** Zn is present in said calcium fluoride crystal and has a content of less than 0.5 ppm."

VIII. Claim 1 according to auxiliary request 2 differs from that of auxiliary request 1 in that the additional feature "**, which constitutes zinc fluoride added as**

scavenger," was inserted between the terms "wherein Zn" and "is present in said calcium fluoride crystal …".

IX. Claim 1 according to auxiliary request 3 differs from that of auxiliary request 1 in that the additional feature "**, such as Zn, Cd, Pb, Li, Bi and Na,**" was inserted between the terms "constituting a scavenger" and "with a content of 10 ppm or less …".

X. Claim 1 according to auxiliary request 4 differs from that of the main request in that the feature "**total**" was deleted from the term "… with a total content of 10 ppm or less …".

XI. Independent claims 1 and 4 of auxiliary request 5 read as follows:

"1. A method of producing a calcium fluoride crystal comprising
conducting a refining step of adding zinc fluoride as a scavenger to a high purity synthetic calcium fluoride raw material and refining the raw material at least once, melting and gradually cooling the mixture and removing that part of the thus obtained crystal which is finally crystallized and
a crystal growth step of further adding the scavenger to the refined raw material and growing a crystal by using a crucible lowering method,
wherein an amount of zinc fluoride to be added in a first refining step is 0.04 to 0.1 mole % based on the raw material, and
wherein a total amount of the scavenger to be added in the subsequent refining steps and the crystal growth

step is 10% to 50% based on the amount of the scavenger to be added in the first refining step."

"4. A calcium fluoride crystal **obtainable by the method according to claim 1 or 2, so that** the calcium fluoride crystal contains Zn of less than 0.5 ppm and oxygen with a content of 50 ppm or less, and has an internal transmittance of 70% or more with respect to a 135 nm wavelength light, the internal transmittance being a value determined by subtracting the light absorbance (%) of a bulk from 100%, wherein the light absorbance is a value obtained based on a light absorption coefficient of a sample to be measured having a thickness of 10 mm."

XII. Independent claim 3 of auxiliary request 5a reads as follows (amendments compared to claim 1 of the main request are in bold with deletions between brackets, emphasis added by the Board):

"3. A calcium fluoride crystal **obtainable by conducting a refining step of adding zinc fluoride as a scavenger to a raw material of calcium fluoride and refining the raw material by heating to 1360°C to melt and gradually cool the mixture and removing the upper part of the thus obtained crystal which is finally crystallized by a thickness of 1 mm, said refining step being carried out at least once, and a crystal growth step of further adding zinc fluoride to the refined raw material maintaining the material at vacuum degree of $2x10^{-6}$ Torr and 1360°C for 11 hours and growing a crystal by using a crucible lowering method by lowering the crucible at a 2 mm/hour rate wherein an amount of zinc fluoride to be added in the first**

refining step is 0.04 to 0.1 mole % based on the raw material, and wherein a total amount of the scavenger to be added in the subsequent refining steps and crystal growth step is 10% to 50% based on the amount of the scavenger to be added in the first refining step, annealing step comprising placing the grown calcium fluoride crystal and zinc fluoride of 0.1 % by weight in an annealing furnace, exhausting the inside of the furnace and raising the crucible temperature from a room temperature to 900°C at a 100°C/hour rate, maintaining at 900°C for 20 hours and lowering the crucible temperature at a 6°C/hour rate so as to cool the crucible to a room temperature, and cutting and abrading the calcium fluoride crystal thus obtained, to form a 10 mm thick disc, contain**s** at least one kind of atom selected from the group consisting of **{at this point the claim mentions an intended insertion 2, which is, however, not listed; in a preceding version of auxiliary request 5a discussed in the oral proceedings this insertion reads: "scavenger-constituting metal elements including"}** Zn, Cd, Pb, Li, Bi and Na with a **[total]** content of 10 ppm or less, containing oxygen with a content of 50 ppm or less, and having an internal transmittance of 70% or more with respect to a 135 nm wavelength light, the internal transmittance being a value determined by subtracting the light absorbance (%) of a bulk from 100%, wherein the light absorbance is a value obtained based on a light absorption coefficient of a sample to be measured having a thickness of 10 mm, wherein said Zn is present in said calcium fluoride crystal and has a content of less than 0.5 ppm."

XIII. Claim 1 according to auxiliary request 6 reads as follows:

"1. A method of producing a calcium fluoride crystal comprising conducting a refining step of adding zinc fluoride as a scavenger to a raw material of calcium fluoride and refining the raw material by heating to 1360°C to melt and gradually cool the mixture and removing the upper part of the thus obtained crystal which is finally crystallized by a thickness of 1 mm, said refining step being carried out at least once, and a crystal growth step of further adding zinc fluoride to the refined raw material maintaining the material at vacuum degree of $2x10^{-6}$ Torr and 1360°C for 11 hours and growing a crystal by using a crucible lowering method by lowering the crucible at a 2 mm/hour rate wherein an amount of the scavenger to be added in the first refining step is 0.04 to 0.1 mole % based on the raw material, and wherein a total amount of the scavenger to be added in the subsequent refining steps and the crystal growth step is 10% to 50% based on the amount of the scavenger to be added in the first refining step, annealing step comprising placing the grown calcium fluoride crystal and zinc fluoride of 0.1 % by weight in an annealing furnace, exhausting the inside of the furnace and raising the crucible temperature from a room temperature to 900°C at a 100°C/hour rate, maintaining at 900°C for 20 hours and lowering the crucible temperature at a 6°C/hour rate so as to cool the crucible to a room temperature, and cutting and abrading the calcium fluoride crystal thus obtained, to form a 10 mm thick disc."

XIV. The appellant argued essentially as follows:

The upper value of "< 0.5 ppm Zn" of claim 1 of the main request is taken from the examples which were made under specific process conditions (vacuum degree, temperatures, duration, etc.) which influence said value. Decision T 191/93 (not published in OJ EPO) concerns the generalisation of a feature taken in isolation from drawings which was not allowable either and in the present case there exists a similar situation. It should also be considered that claim 1 relates to a product which may obtained by using PbF_2 as scavenger instead of ZnF_2, the Zn being present as unavoidable impurity. Hence the generalisation of this upper value contravenes Article 123(2) EPC. The feature "total content of 10 ppm or less" for Zn, Cd, Pb, Li, Bi and Na, all taken together, is not derivable in an unequivocal manner from the original description, e.g. page 9 does not specify whether the definition scavenger-constituting metal embraces one kind of metal or not. Consequently also this feature contravenes Article 123(2) EPC.

The same conclusions with respect to the feature "<0.5 ppm Zn" are valid for claim 1 of auxiliary requests 1 to 4 and claim 4 of auxiliary request 5. Hence the requirements of Article 123(2) EPC are not met by claim 1 of these auxiliary requests. Furthermore, the scope of claim 1 according to auxiliary requests 1 to 4 and of claim 4 of auxiliary request 5, due to the substitution of the feature "containing at least one kind of atom selected from the group consisting of Zn, Cd, Pb, Li, Bi and Na with a total content of 10 ppm or less" of claim 1 as granted with the feature

"containing atoms constituting a scavenger with a content of 10 ppm or less" (auxiliary requests 1 to 3 since the non-limiting insert "such as …" of auxiliary request 3 need not be considered), or the deletion of the feature "total" from claim 1 as granted according to claim 1 of auxiliary request 4 clearly extends the subject-matter claimed beyond the scope of claim 1 as granted, contrary to Article 123(3) EPC. The same conclusion is valid for claim 4 of auxiliary request 5 which no longer specifies the total impurity content of the six mentioned elements and thus similarly contravenes Article 123(3) EPC. The feature "high purity synthetic calcium fluoride raw material" of claim 1 of auxiliary request 5 renders this claim unclear.

The deletion of the term "total" from claim 3 of auxiliary request 5a implies extending the scope thereof compared to claim 1 as granted contrary to Article 123(3) EPC. Additionally claim 3 represents an "aliud".

The feature "said refining step being carried out at least once" as contained in the first, refining, step of claim 1 of auxiliary request 6 appears to be inconsistent with the plurality of refining steps mentioned furtheron in claim 1 and thus to contravene Article 84 EPC. Novelty is not contested but the method of claim 1 lacks an inventive step because such crystal growth processes are always carried out under vacuum. Any scavenger has to be sublimable and this applies also to its reaction products. The temperatures for such a process are also known and normally are slightly below the melting point of the crystal. The amounts of

the scavenger to be used in the refining step are likewise known, see e.g. D2, page 5, line 1 which specifies 0.2-2.0 wt% PbF_2 corresponding to about 0.06-0.6 mol%; or see D1 which specifies 1-2 wt% PbF_2 corresponding to about 0.3-0.6 mol% (see column 3, lines 12 to 14 and lines 18 to 25). The compulsory tempering step is also known to the person skilled in the art. According to D2 it is carried out with the addition of PbF_2 as scavenger (see page 5, lines 2 to 8). Since, compared to the closest state of the art D2, no improvement of the resulting CaF_2 crystal has been demonstrated the objective problem can only be the provision of an alternative process which takes account of environmental requirements by avoiding poisonous reactants (see patent in suit, page 7, paragraph [0082]). The solution to this problem (using a ZnF_2 scavenger) is considered to be obvious to the person skilled in the art and thus to not involve an inventive step as it only requires applying common general knowledge and ordinary skills.

XV. The respondent argued essentially as follows:

Claim 1 of the main request does not contravene Article 123(2) EPC because it is based on claims 1 and 2 in combination with page 8, lines 17 to 22 and page 24, Table 1, examples 3-8 of the application as originally filed. The objection raised in the Board's communication concerning an intermediate generalisation of the feature "<0.5 ppm Zn" taken from examples 3-8 is not understood although it is admitted that the raw material has a certain impurity content and that the contents of the other impurity elements are likewise reduced during the claimed CaF_2 crystal producing

process. However, with respect to the application of the decision T 201/83 (OJ EPO 1984, 481) as suggested by the Board in the oral proceedings it was remarked that the respondent cannot be obliged to also define the concentrations of the other elements as were present in the examples. This is particularly so, since only the contents of Zn, O, La, Y and the like have been measured but only the contents of Zn and O were specified in the Tables (see page 22, lines 13 to 15). Furthermore, it is not derivable from the application as originally filed that the internal transmittance is directly related to the impurity contents so that the concentrations of these impurities present in the examples are not so closely associated with the concentration of Zn and O present in these examples and now taken up in claim 1 in the sense of T 201/83 (*supra*) as argued by the Board. With respect to the Board's communication it was further argued that it would be clear to the person skilled in the art that only one kind of these metals Zn, Pb, Cd, Li, Bi or Na or a total content of these metals is meant when reading the quoted passages in the description as originally filed (i.e. page 8, lines 16 to 22; page 9, lines 13 to 16 and lines 23 to 26). The other interpretation as brought forward by the Board, resulting in the possibility of a total content of 60 ppm or less would not be plausible to the skilled person, in view of the desired high transmission values.

With respect to the product-by-process claim 4 of auxiliary request 5 which was objected to by the Board under Articles 123(2) and (3) EPC no further arguments are brought forward. The feature "high purity" of claim 1 of this auxiliary request has a clear meaning

for the skilled person (see also D6, right hand column, third paragraph in which the same designation is used).

Product claim 3 of auxiliary request 5a does not contravene Articles 123(2) and (3) EPC. Due to the incorporation of all process parameters taken from the examples the objection concerning an intermediate generalisation no longer applies. In respect of a prior version of this request, discussed in the oral proceedings, it was stated that due to the definition "scavenger-constituting metal elements including Zn …" it is clear that the content of the six scavenger elements is automatically 10 ppm or less. Claim 1 as granted was inconsistent with the description and this was the reason for opening up the definition concerning the metals falling under the term "scavenger", by referring to "including Zn …".

Claim 1 of auxiliary request 6 meets the requirements of Articles 123(2) and (3) EPC and is based on claim 2 as granted in combination with the process parameters taken from the examples (see page 21, line 2 to page 22, line 11). The claimed two-step process comprises a refining step and a crystal growth step and uses ZnF_2 as scavenger in both steps but with an amount in its crystal growth step different from the one in its refining step. The skilled person had no reason to select ZnF_2 since PbF_2 has been used until 1998, the filing year of the application. According to the claimed method a reduced amount of scavenger is added in the crystal growth step and it is evident from the comparative examples 1, 2, 9 and 10 that the good internal transmittance values can only be reached with the claimed ratios. D2 represents the closest state of

the art merely for being published in 1984 since all others are much older (D1: 1940; D6: 1979). The objective problem to be solved is to provide a process for producing a CaF_2 crystal having an improved, i.e. reduced deterioration ratio (see page 23 of the application as originally filed). D1 discloses a one-step process (see column 3, lines 18 to 28) and PbF_2 is soluble in CaF_2 (see column 2, lines 35 to 38; column 3, lines 12 to 14). The sequence of steps with differing scavenger amounts as defined in claim 1 improves the transmittance of the CaF_2 crystals. Comparative tests with respect to the crystals of D2 have not been made. Nevertheless, the method of claim 1 of auxiliary request 6 involves an inventive step.

Reasons for the Decision

1. *Amendments (Articles 123(2) and (3)EPC)*

Main request

1.1 Claim 1 of the main request is based on claims 2 and 3 as originally filed in combination with page 11, lines 21-26 and samples 3-8 of Tables 1 and 2 of the application as originally filed (corresponding to the published WO-A-95 20472), with the exception of the following features:

1.1.1 The feature "wherein said **Zn** is present … and **has a content of less than 0.5 ppm**" is taken from the examples 3-8 (see pages 24 and 27, Table 1 and 2). In this context, however, it has to be considered that according to the description the analysis of certain

elements, namely "of Zn, O, La, Y and the like" (see page 22, lines 10 to 15), as well as the transmittance measurements were carried out **after all process steps,** including a final tempering step in an atmosphere comprising 0.1 wt.% ZnF_2, had been carried out (see pages 21 to 26, examples 1 and 2).

1.1.2 Consequently, said value "**less than 0.5 ppm Zn**" is the result of **all** said **process steps** which involved specific heat treatments for specific time periods at specific temperatures and degrees of vacuum as well as the removal of a thickness of 1 mm of the upper part of the crystallized calcium fluoride after the first refining step. It is evident that the application of these process steps implies a reduction of other impurity elements by evaporation in vacuum, at least of the so-called scavenger-constituting elements, which are either comprised in the CaF_2 raw material or in the ZnF_2 scavenger in small amounts, as admitted by the respondent.

1.1.3 Neither the content of the impurities in the raw material nor the content of the ZnF_2 scavenger have been disclosed in the application as originally filed. These impurity contents will, however, vary depending upon the supplier of these materials.

Furthermore, as is self-evident, the extent of removal of e.g. the scavenger-constituting element Zn or of other impurity elements (including the other scavenger-type elements Cd, Pb, Li, Bi, and Na as specified in this claim) is strongly dependent upon the applied temperatures, the degrees of vacuum and the duration of the heat treatments.

Consequently, there exists a substantial degree of interdependence of the quantitative values for these elements, as is evidenced by the fact that the concentration thereof was measured for the specific examples ("… the contents of Zn, O, **La, Y and the like** were measured", see page 22, lines 10 to 16 of the original application).

Therefore, said value of "less than 0.5 ppm Zn" is closely associated via the actual process applied with specific maximum values for all other impurities comprised in the resulting final CaF_2 crystal which have an influence onto the internal transmittance of said crystal (see page 4, lines 1 to 4 of the application as originally filed). Such values, however, are not disclosed in the original application ("Results are **partially** shown in Table 1 and Fig. 1", page 21, line 15) or, if disclosed (for La and Y, see page 25, line 22 to page 26, line 1) are not present in claim 1.

1.1.4 Given this situation, the individual amount of Zn as given for examples 3-8, cannot be regarded in isolation as is now done via the amendment of claim 1. This may be done only in very exceptional cases and in this context reference is made to the existing longstanding Case Law, see in particular decision T 201/83 (*supra*, point 12 of the reasons, last sentence) "an amendment of a concentration range in a claim for a mixture, such as an alloy, is allowable on the basis of a particular value described in a specific example, provided the skilled man could have readily recognised this value as not so closely associated with the other features of the example as to determine the effect of that

embodiment of the invention as a whole in a unique manner and to a significant degree". Taking account of these considerations, which are not restricted to alloys, the conclusion can only be that because of the effects of the process parameters onto the constituents making up the claimed CaF_2 crystal and its properties, it is not possible to arbitrarily select an individual feature from the examples. To disregard the specific context would result in a new selection from the original range which was neither explicitly nor implicitly disclosed.

1.1.5 Therefore the incorporation of the feature "wherein said Zn is present in said calcium fluoride crystal and has a content of less than 0.5 ppm" taken as an isolated feature from examples 3 to 8 of Tables 1 and 2 into claim 1 according to the main request contravenes Article 123(2) EPC.

1.2 The feature of claim 1 "containing at least one kind of atom selected from the group consisting of Zn, Cd, Pb, Li, Bi, and Na with a **total** content of 10 ppm or less, containing oxygen with a content of 50 ppm or less" has no explicit basis in the application as originally filed. It is also not directly and unambiguously derivable therefrom for the following reasons:

1.2.1 Claims 1, 2 and 10 as originally filed only defined "containing at least one kind of atom selected from the group consisting of Zn, Cd, Pb, Li, Bi, and Na with a content of 10 ppm or less, and containing oxygen with a content of 50 ppm or less" with corresponding counterparts at page 5, lines 15-25 and page 7, lines 3-13, and page 28, lines 19-24" of the

application as originally filed. According to this definition each of the specified elements may be comprised in an amount of up to 10 ppm so that, in case that all six elements are present, a total content of these elements of up to 60 ppm is defined.

1.2.2 The list of metals specified in claims 1, 2 and 10 as originally filed is restricted to metals which constitute a scavenger. Their concentration definition is absolute and includes that amount of these elements comprised as impurities in the CaF_2 raw material which in combination with the elements and impurities of the non-removed scavenger has remained in the resulting CaF_2 crystal after its production process.

1.2.3 According to page 8, lines 17-22 of the application as originally filed "a fluoride crystal of the present invention has the content of metal atoms of 10 ppm or less, such as Zn, Cd, Pb, Li, Bi, and Na, i.e. metal atoms constituting a scavenger to be added in the refining step and the growth step of the fluoride crystal, and the content of oxygen atoms of 50 ppm or less". According to this passage a content of 10 ppm or less of metal atoms is defined which is **neither** restricted ("such as …") to the specified six elements **nor** is it clear from this passage that all of these metals should be present in said CaF_2 crystal since the term "metal atoms", i.e. the plural of a metal atom does not specify whether these atoms belong to only one kind of metal or not.

Said metal content is restricted to **metal atoms constituting a scavenger** which is to be added in the said process steps. Hence this definition "metal atoms

constituting …" is considered to include the impurities which are already present in the raw material for making the CaF_2 crystal as well as those comprised in the scavenger itself (compare point 2.1.2 above).

1.2.4 In this context the Board further considered that at page 9, line 5 to page 10, line 14 of the application as originally filed it is more clearly stated that according to the described production process, which includes the addition of specified amounts of a scavenger, that the fluoride crystals have "a scavenger-constituting metal element content of 10 ppm or less and an oxygen content of 50 ppm or less". In said passage it is further stated that "by repeatedly conducting the refining step, the impurity concentration of La, Y and the like can be further reduced" and that "La and Y contents can be 5 ppm or less, and 10 ppm or less, respectively". This passage thus implies that the amount of the added scavenger (the application only discloses the addition of one kind of scavenger (ZnF_2) and does not disclose the addition of a mixture of two or more kinds of scavenger) results in **a scavenger metal content of 10 ppm or less** in the fluoride crystal. This is interpreted to mean that if, for example, ZnF_2 would have been added as scavenger, then the Zn content in the CaF_2 would be limited to 10 ppm or less, but that the content of other metal elements contained as impurities in said fluoride crystal such as Cd, Pb, Li, Bi, and Na (and possibly also of La and Y), which have not been added, would not be restricted. This interpretation is in line with the aforementioned definition of claim 1 as originally filed according to which the content of a single metal is restricted to 10 ppm or less.

1.2.5 With respect to La and Y the application states in the context of the examples that "the contents of Zn, O, La, Y and the like were measured. Results are partially shown in Table 1 and Fig. 1" (see page 22, lines 10-15). Tables 1 and 2, however, only specify the contents of Zn and O and do not disclose any value of the two other mentioned elements. However, the application further discloses that "The La and Y contents can be reduced by increasing the number of the refining steps. By conducting the refining step one time, they were 5 ppm and 10 ppm, respectively. … By conducting three times, they became 1 ppm or less, and 1 ppm or less, respectively" (see page 25, line 22 to page 26, line 1). The fact that the amount of La and Y can be reduced by increasing the number of refining steps, raises the question whether or not these elements La and Y also act as a scavenger; actually these metals have a strong affinity to oxygen; although their oxides have a high melting point it may well be that their oxyfluorides or fluorides are the more volatile compounds which can be removed during the refining steps and the crystal growth step.

Furthermore, the interpretation of point 2.2.4 above is also in line with the aforementioned examples which specify a La and Y content of 5 ppm and 10 ppm, respectively, when conducting the refining step only once so that the total content of La and Y was 15 ppm, i.e. greater than 10 ppm.

1.2.6 The application as originally filed further discloses "concrete examples of scavengers include zinc fluoride, cadmium fluoride, lead fluoride, lithium fluoride,

bismuth fluoride, and sodium fluoride" (see page 12, lines 24-27) and "the scavenger to be used in the present invention includes lead fluoride, cadmium fluoride, zinc fluoride and the like" (see page 20, lines 15-17). Thus the lists of scavengers given are **not** exhaustive. Thus if said passages on pages 8 and 9 were to be interpreted in the manner proposed by the respondent, i.e. differing from the aforementioned interpretation, namely to cover all scavenger constituting metals, then it is not conclusively derivable that said threshold of metal atoms constituting a scavenger of "10 ppm or less" shall be limited to said six elements only.

1.2.7 Thus taking account of the considerations above the definition "with a total content of 10 ppm or less" of claim 1 of the main request - which combines the definition of a mere metal content with the total content of the scavenger-constituting metals, thereby limiting the total contents of said six elements and, based on the wording "a calcium fluoride crystal **containing** …", at the same time allowing for the presence of further - unspecified - contents of scavenger-constituting metals is not derivable directly and unambiguously from the application as originally filed.

1.2.8 Hence also this feature of claim 1 of the main request contravenes Article 123(2) EPC.

1.3 The Board therefore concludes that claim 1 of the main request contravenes Article 123(2) EPC. The main request is therefore not allowable.

Auxiliary requests 1 to 4

1.4 Claim 1 of auxiliary requests 1 to 4 contains the same objectionable feature as claim 1 of the main request (see points VII-X above):

For auxiliary requests 1, 2 and 3 ("wherein Zn is present in said calcium fluoride crystal and has a content of les than 0.5 ppm") only the word "said" has been deleted (in: "wherein **said** Zn is present …"), whereas in claim 1 of auxiliary request 2 the feature "… wherein Zn, **which constitutes zinc fluoride added as a scavenger**, is present …" has been amended.

These small alterations of the wording have no influence on the feature concerning the definition of the Zn content of less than 0.5 ppm as such. Therefore the conclusions of point 1.1.5 above apply *mutatis mutandis* for claim 1 of auxiliary requests 1 to 4 which therefore contravenes Article 123(2) EPC. The auxiliary requests 1 to 4 are therefore not allowable.

Auxiliary request 5

1.5 Claim 4 of auxiliary request 5 is considered to represent a product-by-process claim due to its wording "A calcium fluoride crystal **obtainable by the method according to claims 1 or 2 …**" which refers back to process claim 1. Claim 4 - likewise as product claim 1 of the previous requests discussed above - comprises the feature "**contains Zn of less than 0.5 ppm**".

Neither product claim 4 nor the process claim 1 to which it refers, however, specify all the process

parameters of examples 3 to 8, which are necessary to obtain the result of "contains Zn less than 0.5 ppm".

Consequently, the conclusions of point 1.1.5 above apply *mutatis mutandis* for claim 1 of auxiliary request 5 which therefore also contravenes Article 123(2) EPC. Auxiliary request 5 is therefore not allowable.

Auxiliary request 5a

2. *Admissibility of auxiliary request 5a*

As response to the Board's negative provisional opinion concerning the requirements of Article 123(2) EPC (see point II above) and one month before the oral proceedings the respondent filed a new main request and six auxiliary requests. During the oral proceedings the amendments carried out in the auxiliary requests 1 to 5 were objected to by the appellant and the Board for not complying with Article 123(2) EPC and for contravening Article 123(3) EPC (see points 1.4 and 1.5 as well as XIV above). For a response to these objections the Board allowed the respondent time to prepare a further auxiliary request indicating, however, that the admission of such a request was open to discussion. The respondent then filed a first version of auxiliary request 5a, which after another discussion and objections under Articles 123(2) and (3) EPC was further amended into the present version thereof, and an auxiliary request 6.

2.1 Auxiliary request 5a is another attempt to overcome the two Articles 123(2) EPC objections concerning said features "Zn … has a content of less than 0.5 ppm" and

"total content of 10 ppm or less" comprised in the product claim of the main request, while not contravening Article 123(3) EPC.

Auxiliary request 5a involves product claim 3 representing an amended version of the product-by-process claim 4 of auxiliary request 5, now specifying all process parameters of the examples necessary for obtaining said Zn content of less than 0.5 ppm in combination with the claimed transmittance value. Additionally, in order to overcome the objections made in point 1.2 above the term "total" was deleted.

2.1.1 The deletion of the term "total" raises the issue of Article 123(3) EPC, i.e. the scope of claim 3 of auxiliary request 5a extends beyond the scope of claim 1 as granted because claim 1 as granted limits the **total** content of said six elements to 10 ppm or less.

Taking account of the considerations under point 1.2.4 and particularly of point 1.2.5 above, it is obvious from the patent in suit that the process applied to achieve examples 3-8 does not automatically result in a product having a content of scavenger-constituting metals of 10 ppm or less. Hence claim 3 of auxiliary request 5a is *prima facie* not allowable, already for this reason alone.

In combination with the feature concerning the six metal elements, possibly intended to read "… from the group consisting of **scavenger-constituting metal elements including Zn, Cd, Pb, Li, Bi and Na with a content of 10 ppm or less**" (see point XII above) in the

final version of auxiliary request 5a the allowability of this amendment is even less evident.

2.1.2 In the Board's opinion where an amendment is filed during oral proceedings in the form of a new request it should not be necessary to carry out an extensive examination with respect to the compliance with the requirements of Article 123(2) and (3) EPC since this constitutes an unreasonable burden on the other party and the Board within the time constraints of an oral proceedings.

Taking account of the fact that the present auxiliary request 5a was filed at an advanced stage of the oral proceedings the Board applied its discretion under Article 13 RPBA, as in force of 13 December 2007, (former Article 10b) and did not admit this request into the proceedings.

2.1.3 Auxiliary request 6, however, was admitted into the proceedings for the reasons that follow.

Auxiliary request 6

3. *Amendments (Articles 123(2) and (3) EPC)*

Method claim 1 of auxiliary request 6 is based on method claims 4 and 6 as originally filed in combination with the disclosure of page 21, line 2 to page 22, line 11 of the description as originally filed. Thus the requirements of Article 123(2) EPC are met.

Since claim 1 of auxiliary request 6 is much more restricted than claim 3 as granted it meets also the requirements of Article 123(3).

The Board does also not share the appellant's opinion that the feature "said refining step being carried out at least once", where furtheron in the claim reference is made to "subsequent refining steps", would render claim 1 unclear. To the contrary, from the wording "said refining step being carried out **at least once**" it is clear that there can be only one refining step, but also more than one, the subsequent one(s) having to fulfil a specific requirement as to the amount of zinc fluoride added as scavenger in comparison with the amount of zinc fluoride added in the first refining step. Thus claim 1 is also considered to meet the requirement of Article 84 EPC.

4. *Novelty (Article 54 EPC)*

Novelty of the subject-matter of method claim 1 of auxiliary request 6 was not disputed by the appellant. The Board is satisfied that none of the prior art documents, particularly D1, D2 and D6, discloses a method for producing calcium fluoride crystal having all the features of claim 1. The same applies in respect of the prior uses as alleged by the appellant, and the evidence filed in support of that objection. In any case, the appellant no longer relied on the alleged prior use(s) for its objections against the present method claim 1.

The Board therefore concludes that the subject-matter of claim 1 is novel.

5. *Inventive step (Article 56 EPC)*

Document D2 was undisputedly considered as the closest prior art. It discloses a process for the production of CaF_2 single crystals for optical purposes without disturbing absorptions (over the whole spectral range from UV (125 nm) to IR (9000 nm)), opacities, colorations or other inhomogeneities, suitable for highest polarisation optical requirements. The process includes the addition of a scavenger (PbF_2) in an amount of 0.2-2 wt.% with respect to CaF_2 in the crystal growth step under vacuum and a subsequent tempering step at a temperature of 1200°C for 2-3 hours including cooling down to 900°C in a PbF_2-containing atmosphere (see page 4, line 16 to page 5, line 8 and line 16 to page 6, line 22; claim 1).

Said range of 0.2-2.0 wt.% PbF_2 as scavenger corresponds to 0.064-0.64 mol% and overlaps with the range of from 0.04-0.1 mol% for the scavenger in the first refining step according to claim 1 of auxiliary request 6 (in this context the Board remarks that 0.04-0.1 mol% of ZnF_2 plus 10-50% added later in the further refining steps and the crystal growth step as claimed results in a range of 0.044-0.15 mol% for the total amount of scavenger applied in all steps).

5.1 Thus the process of claim 1 of auxiliary request 6 differs from that according to D2 in that
a) ZnF_2 is used as scavenger, and
b) that a specific amount of 0.04-0.1 mol% of the scavenger based on the raw material is added in a first refining step which includes heating to 1360°C to melt

and gradually cool the mixture and removing the upper part thereof by a thickness of 1 mm, said refining step carried out at least once, and

c) that in the crystal growth step zinc fluoride is added to the refined raw material and maintained at vacuum degree of $2x10^{-6}$ Torr and 1360°C for 11 hours and that the crystal is grown using a crucible lowering method including lowering the crucible at a 2mm/hour rate, and

d) that 10-50% of the zinc fluoride, based on the amount of the first refining step, is added in all subsequent refining steps and the crystal growth step, and

e) that the annealing (tempering) is carried out by adding 0.1% by weight of zinc fluoride to the grown calcium fluoride crystal and exhausting the inside of the furnace and raising the crucible temperature from room temperature to 900°C at a 100°C/hour rate, maintaining at 900°C for 20 hours and lowering the crucible temperature at a 6°C/hour rate to room temperature, and

f) cutting and abrading the obtained calcium fluoride crystal to form a 10 mm thick disc.

5.2 No comparative examples with respect to the closest prior art D2 are present in the patent in suit nor have they been submitted by the respondent, as admitted in the oral proceedings. Consequently, it has not been demonstrated by the respondent that the claimed process actually results in a product having better properties and an improved deterioration ratio. Hence the respondent's definition of the objective problem, i.e. to provide a process for producing calcium fluoride

crystals having an improved deterioration ratio, cannot be accepted by the Board.

Therefore the objective technical problem is a less ambitious one and is defined as the provision of an alternative process for producing CaF_2 crystals for optical purposes which is harmless for the environment (see patent in suit, page 7, paragraph [0082]).

5.3 This problem is solved by the process as defined in claim 1 of auxiliary request 6.

5.4 The Board considers that the subject-matter of claim 1 is not rendered obvious for the following reasons:

5.4.1 D2 mentions in its description of the prior art the documents D1 and D4 (=US-A-2 550 173), the first one concerns the adding of a scavenger, preferably a metal fluoride having specific properties so as to be removed during the refining step, more preferably PbF_2, and the second one describes that the addition of a getter material, e.g. PbF_2, in the crystal growth step (i.e. after a first refining step in a fluorine atmosphere) further improves the optical qualities of CaF_2 crystals (see D2, page 2, second and third paragraph).

However, there is nowhere a hint to be found in the mentioned documents to choose ZnF_2 as scavenger. Moreover, there exist other - compared to Pb or Cd - harmless metal fluorides such as NaF or LiF. Hence there exists no one-way street situation to select zinc fluoride.

The appellant did not contest the respondent's argument that PbF_2 was used as scavenger until 1998, so that at the priority date (1997) the person skilled in the art had no reason at all to choose ZnF_2. But even if the person skilled in the art would have modified the process of D2 by using zinc fluoride as scavenger he would not have arrived at the process claimed in claim 1 since there are a number of further modifications necessary (see point 5.1 above, compare the further distinguishing features b) to f)) and the person skilled in the art has no reason to do so.

5.4.2 The appellant argued that the temperatures for such a process are known and normally are slightly below the melting point of the crystal and that the compulsory tempering step is also known by the person skilled in the art. First of all, the appellant has not submitted any supporting evidence for this submission to prove the same and secondly, he has not given any reason as to why the person skilled in the art would do so in the particular manner presently claimed. For example, the tempering (annealing) step according to D2 is carried out at 1200°C for 2-3 hours while according to the method of claim 1 it is done at 900°C for 20 hours. Assuming that both products have the same properties then the productivity of the process of claim 1 is about 6-10 times lower than that according to D2. Thus it does not appear to be credible that the skilled person would modify the tempering step of the process of D2. Consequently, these arguments cannot be accepted.

5.4.3 The appellant further argued that the amount of scavenger to be used in the refining step is likewise known, see e.g. D2, page 5, line 1 which specifies 0.2-

2.0 wt% PbF_2 corresponding to about 0.06-0.6 mol%; or see D1 which specifies 1-2 wt% PbF_2 corresponding to about 0.3-0.6 mol% (see column 3, lines 12 to 14 and lines 18 to 25). However, the amount of about 0.06-0.6 mol% according to D2 relates to that of the scavenger added in the crystal growth step whereas the amount of 0.3-0.6 mol% of D1 - which is by far too high compared to the range required by claim 1 of auxiliary request 6 - corresponds to the amount added in the first refining step but there is no disclosure or suggestion in D1 to further add PbF_2 in a separate crystal growth step (see D1, column 3, lines 9 to 28 and lines 43 to 70). There is likewise no suggestion to reduce said amount of scavenger in a further crystal growth step as compared to the refining step since D1 discloses that an excess is not critical and can be removed by volatilization (see column 3, lines 14 to 17).

Hence the appellant's arguments cannot be accepted.

5.4.4 Furthermore, it is apparent from the examples and particularly the comparative examples 1, 2, 9 and 10 of the patent in suit that impurity levels of 0.5 ppm or less of zinc and of 50 ppm or less of oxygen are obtainable only when the amount of scavenger added in the refining step and crystal growth step is within the ranges as defined in claim 1. If the amount of scavenger added in the first refining step is more than 0.1 wt% or less than 0.04 to 0.1 mol% (i.e. examples 1, 2 and 10), or if the amount to be added in the crystal growth is below the minimum of 10% based on the amount of the zinc fluoride added in the refining step (i.e. example 9) then the impurity concentration of Zn is too high with values of 20 ppm and 12 ppm, or the oxygen

content is 80 ppm or 150 ppm, respectively (compare patent, Tables 1 and 2).

5.5 For the above reasons claim 1 of auxiliary request 6 is considered to involve an inventive step.

The same conclusion applies to dependent claim 2 which defines a preferred embodiment of the method of claim 1.

6. *Remittal to the department of first instance*

The description of the patent in suit has not yet been adapted to the extensively modified method claim 1 of auxiliary request 6. Furthermore, product claims are no longer present.

Therefore the Board considers it appropriate, in accordance with Article 111(1) EPC, to remit the case to the department of first instance in order to bring the description and figures into appropriate agreement with the subject-matter of claims 1 and 2 of auxiliary request 6.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the department of first instance with the order to maintain the patent with claims 1 and 2 of auxiliary request 6 filed at the oral proceedings, description and figures to be adapted.

The Registrar: The Chairman:

G. Nachtigall H. Meinders