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**Datasheet for the decision
of 10 May 2021**

Case Number: T 2795/17 - 3.3.06

Application Number: 08718284.6

Publication Number: 2139979

IPC: C11D3/04, C11D3/20, C11D3/386,
C11D3/16, C11D3/02

Language of the proceedings: EN

Title of invention:
STABLE ENZYME SOLUTIONS AND METHOD OF MANUFACTURING

Patent Proprietor:
Novozymes A/S

Opponent:
BASF SE

Headword:
Stable enzyme solutions/NOVOZYMES

Relevant legal provisions:
EPC Art. 100(c), 123(2), 56
RPBA 2020 Art. 13(2)

Keyword:

Amendments - added subject-matter (yes) - Main request and auxiliary requests 2, 4, 6, 8-10

Inventive step - auxiliary requests 1, 3, 5, 7 (no)

Late-filed auxiliary requests 11 to 13 - Admitted (no)

Decisions cited:

Catchword:



Beschwerdekammern

Boards of Appeal

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Case Number: T 2795/17 - 3.3.06

D E C I S I O N
of Technical Board of Appeal 3.3.06
of 10 May 2021

Appellant: Novozymes A/S
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted on 19 October 2017 revoking European patent No. 2139979 pursuant to Article 101(3) (b) EPC.**

Composition of the Board:

Chairman J.-M. Schwaller
Members: G. Santavicca
E. Mille

Summary of Facts and Submissions

I. The appeal lies from the decision of the Opposition Division to revoke European patent No. 2 139 979 because the ground of opposition under Article 100(c) EPC prejudiced the maintenance of the patent as granted, claim 1 of which read as follows:

"1. A liquid composition comprising an enzyme constituent, a phenyl boronic acid constituent or a derivative thereof, and a dissolved salt constituent wherein the salt constituent comprises an anion which is chloride, sulphate, nitrate or acetate, and the salt constituent is present in an amount of 0.5 to 10 % by weight of the total composition."

Furthermore the subject-matter of the then pending auxiliary requests either contravened the requirements of Article 123(2) EPC, or was not novel or obvious over D3 (US 5,972,873 A) taken as the closest prior art.

II. In its grounds of appeal the patent proprietor took stance on the decision under appeal and enclosed auxiliary requests 1 to 10.

III. In its reply the opponent maintained all grounds of opposition and raised objections under Articles 123(2) and 56 EPC against auxiliary requests 1 to 10.

IV. In its preliminary opinion the board held that the grounds of opposition under Articles 100(c) and 100(a) EPC (lack of an inventive step) prejudiced the maintenance of the patent as granted, and that the claims of auxiliary requests 1-10 did not meet the requirements of Articles 123(2) and 56 EPC.

- V. With its response of 2 April 2020 the appellant filed auxiliary requests 11 to 13.
- VI. The respondent requested not admit these new requests and regarding the issue of obviousness, it pointed to documents D5 (WO 03/041680 A1) and D9 (WO 02/08398 A1), as well as to the common general knowledge of the skilled person.
- VII. At the oral proceedings held on 10 May 2021 the parties referred to their written submissions as regards the issues other than inventive step, which was discussed taking D3 as the closest prior art.

The final requests of the parties were as follows:

The **appellant (patent proprietor)** requested that the decision under appeal be set aside and the patent be maintained as granted (main request), or auxiliary, in amended form on the basis of the claims according to any of auxiliary requests 1 to 10 filed with the grounds of appeal or of auxiliary requests 11 to 13 filed with letter dated 2 April 2020.

The **respondent (opponent)** requested that auxiliary requests 11 to 13 not be admitted in the appeal proceedings and that the appeal be dismissed.

Reasons for the Decision

1. Main Request - *Amendments*
- 1.1 Claim 1 as granted, compared to claim 1 as filed, comprises the following amendments (made apparent by the Board): "A *liquid composition comprising an enzyme constituent, a phenyl boronic acid constituent or a*

derivative thereof, and a dissolved salt constituent wherein the salt constituent comprises an anion which is chloride, sulphate, nitrate or acetate, and the salt constituent is present in an amount of 0.5 to 10 % by weight of the total composition."

- 1.1.1 Concerning the restriction specifying the anions of the salt constituent, claim 10 as filed - which referred to any preceding claims and so implicitly contained all the features of claim 1 - disclosed the feature "*wherein the salt constituent comprises one or more anions, wherein the one or more anions are selected from the group consisting of chloride, sulphate, nitrate, and combinations thereof*". An identical disclosure can be found on page 8, lines 1 and 2.

As the indefinite article "an" (with respect to the anion) in granted claim 1 does not extend the breadth of the expressions "*one or more*" (anions) and "*and combinations thereof*", the only feature in granted claim 1 not present in original claim 10 is the anion "**acetate**".

- 1.1.2 As regards the restriction to the amount of the salt constituent, claim 13 as filed - which referred back to any preceding claims, and so implicitly comprised the features of claims 10 and 1 - disclosed the feature "*wherein the salt constituent is present in an amount of 0.1 to 20 % by weight of the total composition*", which is broader than in claim 1 as granted. An identical disclosure can be found on page 8, last paragraph of the application as filed.

- 1.2 It follows from the foregoing that an embodiment corresponding to the combination of original claims 1, 10 and 13 disclosed the claimed combination of features

in form of both a (short) list of three possible anions of the salt constituent and a (broader) range for the amount of the salt constituent in the composition.

- 1.3 It remains to be decided whether the addition of the anion "**acetate**" to the short list of anions already disclosed and the limitation of the range for the amount of the salt constituent from originally "0.1 to 20% by weight" to "**0.5 to 10% by weight**" has a basis in the application as filed.
 - 1.3.1 Page 8, last full paragraph, last sentence, discloses in a generic way that the amount of the one or more inhibitor boosters in the composition may indeed be "0.5 to 10% by weight of the total composition", so that this restriction to the amount specified also in original claim 13 does not appear per se to result in an inadmissible extension of the subject-matter of the original application.
 - 1.3.2 Instead, as apparent from claim 10, page 7 (lines 19, 22 and 26) and the table on page 18 of the application as filed, only sodium acetate was specifically disclosed originally as a salt in the composition of a particular embodiment (namely with the inhibitor phenyl boronic acid on page 4, line 15). It is however apparent from the combination of granted claims 1 with any of in particular dependent claim 7, that further specific acetates, in particular those of Zn, Mg and NH₄, are now defined in the claims, these being however not disclosed originally as salts of the claimed composition.
- 1.4 In this respect the appellant argued that not only sodium acetate was specifically disclosed originally as a salt, as there was a clear and unambiguous disclosure

in the sentence bridging pages 6 and 7 of the application as filed - irrespective of the cation - of the acetate anion. Furthermore the combination of granted claim 7 with granted claim 1 did not define further specific acetates (e.g. zinc acetate) not disclosed in the application as filed. Rather, granted claim 7 defined that the cation was Zn, Mg and/or NH₄, and, by its dependency on claim 1, that the anion was chloride, sulphate, nitrate or acetate. Hence, while granted claim 7 covered Zn, Mg and NH₄ acetates, along with the corresponding chlorides, sulphates and nitrates, it did not specifically disclose Zn, Mg and NH₄ acetates. And contrary to the Board's reasoning in its preliminary opinion referring to page 7, lines 19-26 of the application as filed, the sentence bridging pages 6 and 7 of the application as filed was the main basis for a direct and unambiguous basis for specifying in claim 1 as granted that the anion was chloride, sulphate, nitrate or acetate. Thus, the limitation in claim 1 that the salt constituent comprised an anion which was chloride, sulphate, nitrate or acetate represented the deletion of alternatives from a single list of anions in the sentence bridging pages 6 and 7 of the application.

In summary, there was a direct and unambiguous basis for claim 1 as granted in claim 1 as filed in combination with the sentence bridging pages 6 and 7 (for the anion) and page 8, lines 32-33 (for the amount of the salt) of the application as filed.

- 1.5 The Board does not accept these arguments because the sentence bridging pages 6 and 7 of the application as filed, which quotes a number of organic and inorganic anions, cannot be taken in isolation of its context, namely the whole paragraph bridging pages 6 and 7 of

the application as filed, otherwise this would already be a first selection. In fact, the first sentence of said paragraph makes it apparent that it concerns the most general disclosure of non-limiting examples of suitable soluble **salts**, and the second and third sentences merely concern non-limiting examples of the respective cations and anions of the said **salts**. This is even more apparent from the last sentence of the paragraph mentioning a number of specific **salts**, *inter alia* magnesium acetate. Thus, the passage bridging pages 6 and 7 does not represent an unambiguous disclosure of the acetate anion irrespective of the cation of the salt. The only unambiguous disclosure in this respect remains that of original claim 10, which however does not include the acetate anion.

Moreover, the combination of granted claims 7 and 1 implicitly and unambiguously disclose (by way of reference) that the anion acetate is combinable with any of the defined cations, so that e.g. zinc acetate and ammonium acetate are unambiguously identified as being encompassed. However, the original claims did not refer, not even implicitly, to acetate anions as such (let alone to salts thereof, see e.g. original claims 8 and 9 referring to formate instead), nor did the original application disclose or point to salts such as zinc or ammonium acetates.

It follows that the combination defined by granted claims 7 and 1 does not arise from a deletion of alternatives from a single list, but requires the deletion of alternatives in two long lists, to arrive at the claimed combination of four anions and three cations, which was neither directly and unambiguously disclosed nor pointed to in the original application, and which now clearly includes salts going beyond the

specific list of salts disclosed in original page 7 or in the examples.

1.6 As there is no direct and unambiguous basis in the application as filed for the subject-matter of claim 1 as granted, the latter extends beyond the content of the application as filed (Article 123(2) EPC) and the ground of opposition under Article 100(c) EPC prejudices the maintenance of the patent as granted. The main request therefore cannot be allowable.

2. *Auxiliary requests 2, 4, 6 and 8 to 10*

Claim 1 according to each of these requests including the anion "acetate", the objections raised against claim 1 of the main request apply *mutatis mutandis* under Article 123(2) EPC, so that these requests are not allowable either.

3. *Auxiliary request 1 - inventive step*

3.1 In assessing inventive step, the broadest meaning is to be given to the features "*composition*", "*enzyme constituent*" and "*dissolved salt constituent ... (whose) anion ... is chloride, sulphate or nitrate*".

In this respect, it is noted that claim 1 concerns a liquid composition which is not necessarily a detergent composition, and which comprises (and so does not exclude further components) an enzyme constituent (not necessarily a protease), any phenyl boronic acid constituent (not necessarily 4-fPBA), or any derivative thereof, and any salt constituent dissolved in the liquid composition and present in an amount of 0.5 to 10% by weight of the total composition, and which comprises chloride, sulphate or nitrate anions. The

specified amount thus refers to the total amount of chloride, sulphate or nitrate salts, hydrated or not.

- 3.2 The present invention has in particular the objective to provide a liquid enzyme composition with improved enzyme stability, as well as to provide a method for manufacturing the liquid enzyme composition (paragraph [0010]).

This objective is achieved by adding an inhibitor booster such as a soluble salt to a liquid composition including an enzyme and an inhibitor such as a phenyl boronic acid or a derivative thereof, which addition improves the inhibitors effect and the storage stability of the enzyme with regard to its activity (paragraphs [0011] and [0012]).

- 3.3 Considering the similarities in terms of problems addressed and solutions proposed in the prior art and the patent in suit, the Board holds D3 (see columns 1 and 2) as a suitable starting point for assessing the (non) obviousness according to Article 56 EPC. D3 also concerns a liquid detergent composition comprising an enzyme and an improved enzyme stabiliser (column 1, lines 16-18).

In particular, the composition illustrated in example 3 of D3, which is a preferred embodiment (see also column 2, lines 26-35) represents the closest prior art as it discloses the storage stability test of an enzyme in a liquid detergent composition comprising:

- a US-type detergent base, including the components listed in column 12, lines 5-23, *inter alia* 0,4 wt.% of sodium sulphate;
- an enzyme, namely 1% w/w Savinase (which is undisputedly a subtilisin, i.e. a serine protease); and

- a phenyl boronic acid, in particular a 4-formyl-phenyl-boronic acid (4-fPBA), which gives the best inhibition effectiveness (column 12, lines 47 to 53) compared to other phenyl boronic acid derivatives.

Thus, the liquid detergent composition of Example 3 of D3 has all the features of claim 1 at issue but an amount of sodium sulphate of only 0.4%, instead of at least 0.5% as required by claim 1 at issue.

- 3.4 For the appellant, the technical problem to be solved is to provide a liquid enzymatic composition with improved enzyme stability (in line with [0010] of the patent), the improvement being based on the addition of the salt constituent to the phenyl boronic acid.
- 3.5 As a solution thereto, the claim 1 at issue proposes in particular that the salt constituent is present in an amount of 0.5 to 10% by weight of the total composition."
- 3.6 As to the question whether the technical problem is effectively solved by this solution, the Examples in the patent in suit show the residual activity of liquid detergent compositions comprising a protease, a 4-fPBA and 3 wt.% of different specific salts with different cations and anions.
- 3.6.1 The board notes that the relative influence of the weight of salt on the residual activity is however not shown, in so far as only the amount of 3% by weight was tested. Experimental report D21 of the patent proprietor includes comparative tests based on a liquid detergent composition with Savinase, 4-fPBA and varying amounts (0.5, 1 and 3%) of NaCl, MgSO₄ and Mg(NO₃)₂. Although these tests (carried out at pH 9) are not

comparative over Example 3 of D3 (in view of the different, less loaded detergent and no comparative test with 0.4% salt), they appear to show that:

- an increase of the salt amount from 0.5 to 3% improves the protease stability after 7d at 40°C, which improvement is more pronounced for magnesium nitrate and magnesium sulfate than for sodium chloride; and
- an improvement in smaller scale is obtained also without the presence of 4-fPBA.

Hence, the specific salt constituents tested appear to stabilise the protease already without the phenyl boronic acid but especially with phenyl boronic acid.

However, Experimental report D22 (see Table A) of the opponent shows that an increase of the amount of sodium sulphate from 0.4 (as in D3) to 0.5% wt (as in claim 1), at pH values of 7.5 or 9.0, produces only a slightly better residual activity of the enzyme Savinase (respectively from 91 to 92 or from 81 to 86), which is not necessarily better than the reference (sodium formate) used in the examples of the patent. The patent proprietor has objected that the conditions of Experimental report D22 were deliberately chosen to produce less improvement, by the use of a more loaded or stabilised base composition, with components differing from those of the compositions of D3 and D21, and omitting a comparison with no salt and with no phenyl boronic acid. It nevertheless acknowledged that the composition tested in D22 falls under claim 1 at issue and argued that an improvement for sodium sulfate was apparent also from D22.

For the Board, even upon acknowledging that improved enzyme stabilisation has been shown by the comparative tests, in particular D21, the question however arises of whether the improvement in stabilisation is

plausibly achieved across the whole breadth of claim 1, in so far as the liquid composition of claim 1 is not necessarily a detergent composition and does not need to comprise 4-fPBA nor the protease as tested or the tested salts.

Moreover, it should be considered that:

- D3 was not acknowledged in the application as filed, and thus not considered when formulating the original technical problem, though D3 uses 4-fPBA, which is much more effective than other phenyl boronic acids, as apparent from column 12, lines 40-45 of D3;
- the compositions of D21 or D22 are not identical to that of example 3 of D3, which anyhow already comprises 4-fPBA and a Savinase enzyme.

Thus, in view of the generic phenyl boronic acid, salts and enzyme, and of the missing pH, defined therein, the invoked improved stability cannot be plausibly achieved across the whole breadth of claim 1 at issue.

- 3.6.2 It follows that the problem over D3 should be reformulated less ambitiously, namely in the provision of "a further liquid enzyme composition stabilised with phenyl boronic acid derivatives".

It is not in dispute that such a reformulated problem is effectively solved across the breadth of claim 1.

- 3.7 It remains to be decided whether the proposed solution is obvious or not from the prior art, and so whether the skilled person starting from example 3 of D3 and faced with the less ambitious problem formulated above would increase the amount of sodium sulphate in the composition of Example 3 to a value of at least 0.5% by

weight or higher, on the basis of D3 or in view of common general knowledge or cited prior art.

In this respect, the appellant stressed that even if the technical problem was formulated less ambitiously, the solution was not obvious, as D3 was an accidental disclosure, which gave no importance whatsoever to the sodium sulphate content, disclosed no purpose thereof, thus no motivation to modify, let alone to increase its amount, and that in any case the disclosure of a higher content of sodium sulphate in D3 concerned a dishwashing composition rather than a base detergent as in example 3 of D3.

- 3.7.1 For the Board, D3 is not an accidental disclosure, because it pertains to the very same technical field of stabilisation of enzymes such as proteases in liquid compositions (column 1, lines 17-19 and 26) based on the same reversible inhibitor (phenyl boronic acid) (column 1, lines 61-64).

D3 discloses (Table bridging columns 7 and 8) that a liquid dishwashing composition, which contains cleaning surfactants as the composition of Example 3 of D3, can have a content of sodium sulphate up to 12.5 %wt.

The appellant's argument that this disclosure of D3 concerns a dishwashing rather than a detergent base as used in Example 3 is not convincing. For the Board, apart that the limitation "detergent", if present, is to be understood broadly as "cleaning" composition, also the detergent base composition of example 3 of D3 contains cleaning surfactants and sodium sulphate. D3 moreover does not teach that sodium sulphate should only be used with dishwashing compositions. The general disclosure of D3 (see said table) unambiguously teaches

that a liquid enzyme composition with cleaning surfactants tolerates up to 12 wt.% of sodium sulphate. As the composition of D3 is liquid, sodium sulphate cannot be understood to mean that it is used to simply bulk up the composition, as for solid detergent compositions. The fact that D3 does not disclose any purpose therefor is not decisive, in view of the less ambitious problem faced by the skilled person. D3 discloses that sodium sulphate can be present in cleaning enzymatic compositions in higher amounts than that used in example 3.

Still for the Board, the skilled person facing the less ambitious technical problem of providing a further enzyme composition stabilised with phenyl boronic acid derivatives has at its disposal all the options disclosed in D3 and he would have tried higher amounts of sodium sulphate within the limit of 12.5 wt.%, at least in the expectation that it is tolerated. He would thus have tried to use an amount of at least 0.5% wt or higher of sodium sulphate. Thereby, he would have arrived in an obvious way at the claimed liquid composition. D3 suggests (Table bridging columns 7 and 8), albeit in the context of a general formulation for liquid dishwashing compositions, that a sodium sulphate content of up to 12.5 wt.% is compatible with increasing amounts of enzymes (mentioned just below sodium sulphate in the same table). Already for this reason the skilled person would have considered that an amount of sodium sulphate higher than 0.4% by weight merely provides a further liquid enzyme composition according to D3 itself.

- 3.7.2 The Board also shares the position of the respondent that the skilled person also knew, as common general knowledge, or from the same technical field, that an

increase of the concentration of salts such as sodium sulphate not only is tolerated by a liquid enzyme composition but also provides a beneficial effect in the stabilisation of the enzyme. In this respect, attention is particularly drawn to:

- D5, which concerns a liquid enzyme (e.g. subtilisin, see page 31, last paragraph) stabilised composition with phenyl boronic acid, which might contain up to 25 wt.-% surfactant (claims 37 to 39), and teaches that such compositions allow up to **5** wt.-% of salts of alkali metals such as sodium sulphate (see page 15, Point C, first two paragraphs, as referred to by the opponent in the oral proceedings before the Board). The fact that these salts are used as lyotropic stabilisers does not diminish the relevance of D5, in so far as D3 does not directly disclose which purpose should be served by the use of up to 12 wt.-% of sodium sulfate;
- D9, concerning the stabilisation of proteases enzymes (e.g. subtilisin, see claim 5) in liquid compositions (claim 1 and page 1), which may be cleaning/detergent products (claim 7), and which teaches (see page 2, first two paragraphs, in particular lines 1-4, 7 and 16-18; and the paragraph bridging pages 3 and 4) that such compositions allow up to **12** wt.-% of salts of alkali metals such as sodium sulfate (see page 3, line 30). The fact that these salts are used as stabilisers in combination with polyols such as propylene glycol does not diminish the relevance of D9, in so far as D3 does not specify the purpose of using up to 12 wt.-% sodium sulfate but specifically hints at also using other, conventional stabilisers such as propylene glycol (column 6, lines 9-12, Example 3). Hence, in D3 as well sodium sulphate and conventional stabilisers are compatible.

3.7.3 It follows from the foregoing analysis of the prior art that the liquid enzyme composition of claim 1 according to auxiliary request 1 was obvious for the skilled person starting from D3. The composition of claim 1 according to this request thus lacks an inventive step under Article 56 EPC and is not allowable.

4. *Auxiliary requests 3, 5 and 7 - Inventive step*

4.1 The liquid enzyme composition of claim 1 of auxiliary request 3 differs from that of claim 1 according to auxiliary request 1 only in that "*the salt constituent **consists of cations and anions** and the anions are selected ...*".

The composition of claim 1 of auxiliary request 5 differs from that of claim 1 according to auxiliary request 1 in the specification of "*A liquid **detergent** composition*" and in the restriction "*... the salt constituent is present in an amount of ~~0.5~~ **1** to **10%** by weight ...*".

The composition of claim 1 of auxiliary request 7 differs from that of claim 1 according to auxiliary request 5 only in that "*the salt constituent **consists of cations and anions** and the anions are selected ...*".

4.2 The board notes that the amendments "**detergent**" and "**consisting of cations and anions**" in claim 1 of these requests do not distance themselves from the closest composition of Example 3 of D3, which is a detergent composition containing a salt (sodium sulfate) consisting of a sodium cation and a sulfate anion. Moreover, the further limitation of the salt amount to at least **1%** by weight in claim 1 of auxiliary requests 5 and 7 is disclosed or hinted at in a generic way in

D3, and is specifically known in the same technical field, as apparent from D5 and D9 commented above, all of which teach the use of more than 1% by weight of salts such as sodium sulfate.

4.3 Therefore the conclusion of lack of an inventive step drawn for the subject-matter of claim 1 according to auxiliary request 1, especially in view of D3, possibly taken in combination with D5 or D9, still applies *mutatis mutandis* against the subject-matter of auxiliary requests 3, 5 and 7. Consequently, these requests are not allowable either.

5. *Auxiliary requests 11 to 13*

5.1 The admittance of these requests, filed with the response of 2 April 2020 to the board's preliminary opinion, is at the board's discretion under Article 13 RPBA 2020, as they represent an amendment to appellant's case made after the issue of the summons for oral proceedings.

5.2 According to paragraph (2) of Article 13 RPBA: "*Any amendment to a party's appeal case made ..., after notification of a summons to oral proceedings shall, in principle, not be taken into account unless there are exceptional circumstances, which have been justified with cogent reasons by the party concerned.*"

5.3 In its letter of 2 April 2020, the appellant, provided the following reasons: "*We are submitting these new claim requests in view of the Board's preliminary opinion concerning Articles 123(2) and 56 EPC. In particular, the deletion of "or acetate" in auxiliary claim requests 11, 12 and 13 would appear to render these claims allowable accordingly to the Board's*

preliminary view. Moreover, as noted above the Board places a reliance on the data in D22 in its preliminary opinion on inventive step, we submit that the pH limitation in auxiliary claim requests 11, 12 and 13 highlights the greater relevance of the data in the opposed patent and D21 compared to D22. This pH limitation also further distinguishes the claimed subject matter from the disclosure of D3. We request that the Board exercises its discretion to allow late-filed auxiliary requests 11, 12 and 13 into proceedings."

- 5.4 For the board, these reasons are not sufficient to show that exceptional circumstances have arisen, which have been justified with cogent reasons, in so far as these late filed requests have neither been filed in reaction to the decision under appeal, nor do they arise from an amendment of the respondent's appeal case, let alone can they be considered as a reaction to the Board's notification, since the Board has merely given its provisional opinion on the cases made until then by the parties. Moreover, the issue under Article 100(c) or 123(2) EPC due to the feature "or acetate" was dealt with in the decision under appeal, and so was under discussion across the opposition proceedings. D3 was the closest prior art also in opposition proceedings. Furthermore, the deletion of the feature "or acetate" in auxiliary request 8, which defines also the pH, could and should have been recognised earlier if held to be important over D3.

It follows from the foregoing that if the appellant aimed at submitting further fall-back positions it could and should have provided them at the latest with its statement of grounds of appeal.

5.5 Therefore the Board exercised its discretion and decided not to admit late filed auxiliary requests 11 to 13 into the appeal proceedings (Article 13(2) RPBA).

6. As none of the requests filed by the appellant is allowable, its appeal cannot succeed.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



A. Pinna

J.-M. Schwaller

Decision electronically authenticated