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**Datasheet for the decision
of 18 March 2014**

Case Number: T 0812/12 - 3.3.09
Application Number: 06848573.9
Publication Number: 1949799
IPC: A23L1/304, A23L1/305, A23K1/16,
A23K1/175
Language of the proceedings: EN

Title of invention:

SALTS OF MINERAL NUTRIENTS STABILIZED WITH AMINO ACIDS AND/OR
AMMONIUM SALTS, PRODUCTS AND FOOD SUPPLEMENTS THAT CONTAIN
THEM AND METHODS FOR OBTAINING SAME

Applicant:

Ettlin, Eduardo Walter
Boccio, José Rubén
De Paoli, Adrián Tomás
Hager, Edgardo Adrián
De Paoli, Pablo Adrián

Headword:

Relevant legal provisions:

EPC Art. 54(2), 83

Keyword:

Novelty - (no)
Sufficiency of disclosure - (no)

Decisions cited:

Catchword:



**Beschwerdekammern
Boards of Appeal
Chambres de recours**

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Case Number: T 0812/12 - 3.3.09

**D E C I S I O N
of Technical Board of Appeal 3.3.09
of 18 March 2014**

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(Applicant 2)

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Decision under appeal:

**Decision of the Examining Division of the
European Patent Office posted on 27 October 2011
refusing European patent application No.
06848573.9 pursuant to Article 97(2) EPC.**

Composition of the Board:

Chairman: W. Sieber
Members: M. O. Müller
R. Menapace

Summary of Facts and Submissions

I. European patent application No. 06848573.9, filed on 13 October 2006 as international application PCT/IB2006/003840 in the name of E.W. Ettlin, J.R. Boccio, A.T. de Paoli, E.A. Hager and P.A. de Paoli, and claiming priority from AR P20050104763 (11 November 2005), was refused by decision of the examining division issued in writing on 27 October 2011.

II. In the examination proceedings, the following documents were cited:

D1: US 4,900,561; and

D6: US 5,061,815.

III. The examining division's decision was based on claims 1-13 as filed with letter of 2 July 2010, of which claims 1 and 11 read as follows:

"1. Salts of mineral nutrients stabilized with an amino acid and/or an ammonium salt, where the salts are obtained with anions of organic acids or inorganic anions associated to metallic cations which are associated with an amino acid and/or an ammonium salt, characterized because metallic cations and the anions are bonds by a union ionic and a covalent dative bond with the amino group of the amino acid and/or ammonium salts, having as a general formula:

$$[\text{ANION}]^{-n} \cdot [\text{METAL}]^{+n} \leftarrow [\text{AMINOACID and/or AMMONIUM SALT}]_n \cdot x\text{H}_2\text{O}$$

Where:

n takes values from 1 to 3, and

x takes values from 0 to 10."

"11. A procedure to stabilize a mineral salt using an amino acid and/or an ammonium salt according to claim 1, characterized by having the following steps:

- Dissolve the salt to stabilize in an aqueous solution of amino acid and/or an ammonium salt
- Heat at a temperature between 60 and 85°C
- Adjust the pH in the range between 4.5 and 6.8 units
- The process of evaporate is the correct [sic] to obtain the product in the dray [sic] form."

IV. In its decision, the examining division reasoned essentially as follows:

The amendments in the claims introduced subject-matter which extended beyond the content of the application as filed, contrary to Article 123(2) EPC. Furthermore, several claims lacked clarity. Finally, the claimed subject-matter lacked novelty. In this respect, the examining division held *inter alia* that the product of claim 1 was not novel in view of D6. This document described a complex comprising a zinc cation ionically bonded to an inorganic anion (sulphate) and covalently bonded to lysine. Since the method of preparation was effectively the same as that according to the claims, involving dissolving zinc sulphate in water, adding lysine hydrochloride, heating and drying, at least some

of the end product had to be expected to be the same as that claimed.

V. On 19 December 2011, the applicants (hereinafter: "the appellants") filed a notice of appeal against the above decision and paid the prescribed fee on the same day. A statement setting out the grounds of appeal was filed on 27 February 2012 together with new claims 1 to 16, of which claims 1 and 12 read as follows:

"1. Salts of mineral nutrients associated with amino acids and/or ammonium salt, where the salts are obtained with anions of organic acids or inorganic acids and metallic cations associated with amino acids and/or ammonium salt, characterized in that the metallic cations are bound to the anions by means of a ionic bond and by a covalent bond with the amino group of the amino acids and/or ammonium salt, having the following general formula:

$$\{[\text{Anion}]^{n-} \cdot \text{Metal}^{n+} \leftarrow \text{[Amino acid and/or ammonium salt]}\} \cdot x \text{H}_2\text{O}$$

Where:

Where (Anion)⁻ⁿ represents the anion of an organic acid or an inorganic acid

Where (Metal)⁺ⁿ represents the mineral nutrient metal n takes values from 1 to 3, and x takes values from 0 to 10."

"12. A procedure for the synthesis of mineral salts associated with amino acids and/or ammonium salt according to claim 1, characterized in that it comprises the following stages:

- a) in an aqueous solution of amino acids and/or ammonium salt, solubilizing the salt previously formed, in the established relationship of the general formula according to claim 1,
- b) heating at a temperature between 20 and 80°C and adjusting the pH to a value between 3,5 and 8,0, and
- c) obtaining the formed product in an aqueous solution."

VI. On 24 September 2013, the appellants were summoned to oral proceedings. In its preliminary opinion, annexed to the summons, the board observed *inter alia* that the process disclosed in D6 appeared to be identical to the process of claim 12, such that the product obtained in D6 had inevitably to be identical to the product of claim 1. D6 thus appeared to be novelty-destroying for the subject-matter of claim 1.

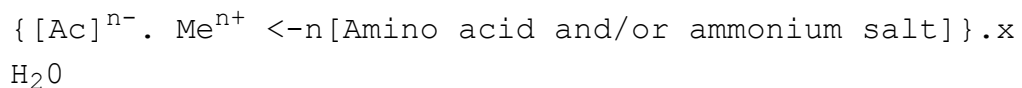
The board was furthermore of the opinion that the process of example 3 of D1 was identical to the process of claim 12. The board observed in this respect that if the salt obtained in D1 were not to be identical to the claimed salt, this would imply that carrying out the process described in claim 12 did not necessarily lead to the claimed product. This would imply in turn that certain additional technical measures would be necessary to obtain these products. The application as filed however did not provide sufficient guidance as to any such additional technical measures, implying that the invention as defined in claim 1 was insufficiently disclosed.

VII. With letter of 14 March 2014, the appellants filed

Annex 1: Collection of excerpts from various dictionaries

and a new main request, independent claims 1 and 10 of which read as follows:

"1. Salts of mineral nutrients associated with amino acids and/or ammonium salt, where the salts contain anions of organic acids or inorganic acids and metallic cations associated with amino acids and/or ammonium salt, characterized in that the metallic cations are bound to the anions by means of a ionic bond and by a covalent dative bond with the amino group of the amino acids and/or ammonium salt, having the following general formula:



Where:

Ac is an acid selected from the group consisting of: Fumaric Acid, Malic Acid, hydrochloric Acid or Sulfuric Acid, sole or any combination thereof.

Meⁿ⁺ is a metal selected from the group consisting of the following cations: Calcium, Magnesium, Iron, Copper, Manganese, Sodium, Potassium.

\leftarrow is covalent dative bond

n takes values from 1 to 3, and

x takes values from 0 to 10 and characterized in that the salt is not a chelate."

"10. A procedure for the synthesis of mineral salts associated with amino acids and/or ammonium salt according to claim 1, characterized in that it comprises the following stages:

- a) solubilizing salts previously formed according to claim 1 in an aqueous solution of amino acids and/or ammonium salt
- b) heating at a temperature between 60 and 85°C and adjusting the pH
- c) obtaining the formed product in an aqueous solution."

VIII. On 18 March 2014, oral proceedings were held before the board. The appellants maintained their main request and did not file any additional requests.

IX. During the oral proceedings, the board made the following observations:

Claim 10 (which is derived from claim 12 of the previous request) referred to the preparation of the product of claim 1, which comprised the steps of adding a salt, such as manganese sulphate, to water and an amino acid, heating to 60 to 85°C and adjusting the pH to obtain a product in aqueous solution. Since the process disclosed in preparation method 6 of D6 was identical to the process of claim 10, the product obtained in D6 had to be identical to the product of claim 1 as well. In this respect, the appellant's assertion that the product in D6 was a chelate complex contrary to that of claim 1 was not convincing since it was not supported by any evidence. Evidence was in particular missing that reworking preparation method 6 of D6 would lead to a complex with a chelate structure. Also, the appellant's reference to a difference between

the amino acid used in D6 (lysine) and that used in the method as described in the patent application (glycine) was not convincing since claims 1 and 10 were not restricted as regards the type of amino acid. Furthermore, in both cases it could be assumed that a covalent dative bond between the amino group of the amino acid and the metal would result, such that the formation of a chelate structure did not depend on the type of amino acid.

The board furthermore reiterated the insufficiency objection raised in its preliminary opinion, namely that if the process disclosed in for instance D1 and D6 did not result in a product as claimed in claim 1, and in particular did not avoid the formation of a chelate, then the invention underlying claim 1 would be insufficiently disclosed. In particular, in this case any teaching would be lacking as to how the formation of a chelate in a process such as disclosed in D1 and D6 and claimed in claim 10 could be avoided.

Finally, the board referred to the appellants' statement made in their letter of 14 March 2014 that the ammonium salts in the present claims could be inorganic. The board explained that it was not conceivable that a covalent dative bond could be formed between an inorganic ammonium salt and a metallic cation and that this was objectionable under Articles 83 and 84 EPC.

- X. The appellants' position in the written and oral proceedings, in as far as relevant to the present decision, was as follows:

According to the first paragraph on page 8 of the application as filed, the products of the prior art

were chelate complexes, while that according to the invention did not have a chelate-type structure. That the claimed product was not a chelate was proven by the ninhydrin test referred to in the first paragraph of page 15 of the application as filed. In this passage it was reported that the product of the invention gave a yellow colour with ninhydrin, while if it were to be present in the form of a chelate, it would not give any colour at all. A further difference was that in the method described in the application as filed, glycine was used as the amino acid, contrary to the lysine applied in preparation method 6 of D6.

In the written proceedings, the appellants had referred to a difference in stoichiometry between the claimed product and that of D6. The appellants did not elaborate on this difference during the oral proceedings.

The appellants did not elaborate either on the board's insufficiency objection in view of D1 or the board's objection under Articles 83 and 84 that the formation of a covalent dative bond between an ammonium salt and a metallic cation was not conceivable.

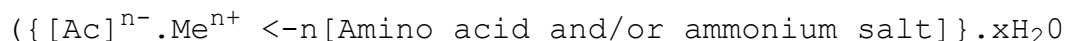
- XI. The appellants requested that the decision under appeal be set aside and that a patent be granted on the basis of the set of claims filed with letter dated 14 March 2014.

Reasons for the Decision

1. The appeal is admissible.

2. *Novelty*

2.1 Claim 1 refers to salts associated with amino acids and/or ammonium salt according to the formula



with Ac^{n-} being eg SO_4^{2-} , Me^{n+} being e.g. Mn^{2+} (ie $n=2$) and x taking values from 0 to 10, wherein the salt is characterised in that it is not a chelate.

According to claim 10, the salts associated with the amino acid of claim 1 are prepared by

- (a) solubilizing the salts in an aqueous solution of amino acids,
- (b) heating at a temperature between 60 and 85°C and adjusting the pH, and
- (c) obtaining the formed product in an aqueous solution (for the exact wording of claims 1 and 10, see point VII above).

2.2 Preparation method 6 disclosed in D6 comprises the steps of

- dissolving one molar equivalent of manganese sulphate and one molar equivalent of L-lysine monohydrochloride in water,
- adding hydrochloric acid to reduce the pH of the resulting solution to about 3.4 and heating the solution to a temperature of approximately 80°C

- to obtain a solution of a dark yellow colour.

2.2.1 The manganese sulphate (MnSO_4) in the first step of D6 corresponds to the salt referred to in step (a) of claim 10. The L-lysine monohydrochloride corresponds to the amino acid as mentioned in step (a) of claim 10. Consequently, the first step of dissolving manganese sulphate and L-lysine monohydrochloride in water in D6 corresponds to step (a) of claim 10.

The second step in D6 of adding hydrochloric acid to reduce the pH of the resulting solution to about 3.4 and of heating the solution to a temperature of approximately 80°C corresponds to step (b) of claim 10.

The third step in D6 of obtaining a solution of a dark yellow colour corresponds to step (c) of claim 10.

Thus, the process disclosed in preparation method 6 of D6 is identical to the process of claim 10. Therefore, without any proof to the contrary, the product obtained in D6 must be assumed to be identical to the product of claim 1.

2.2.2 The appellants argued that according to the first paragraph on page 8 of the application as filed, the products of the prior art were chelate complexes, while that according to the invention did not have a chelate-type structure. That the claimed product was not a chelate was proven by the ninhydrin test referred to in the first paragraph of page 15 of the application as filed, where it was reported that the product of the invention gave a yellow colour with ninhydrin, while if it were to be present in the form of a chelate, it would not give any colour at all.

The board acknowledges that, at least in view of the wording in claim 1 "characterized in that the salt is not a chelate", it can be accepted that the product as claimed does not have a chelate structure. However, the application as filed does not contain any comparative experiment in which the ninhydrin test is applied to the product obtained by preparation method 6 of D6. There is thus no proof in the application as filed, and none has been provided during the examination or appeal proceedings, that the product obtained in preparation method 6 of D6 contains a chelate structure. In view of this lack of evidence, and since the process steps of preparation method 6 in D6 are identical to those defined in claim 10, it must be assumed that the product of D6 does **not** contain a chelate structure.

2.2.3 The appellants further argued that while in the method described in the application as filed, glycine was used as the amino acid, it was lysine that was used in preparation method 6 of D6. However, the method as described in claim 10 does not require the use of any specific amino acid but refers to amino acids in general as does claim 1. Furthermore the board does not see any reason, and none has been provided by the appellants, why the use of lysine in D6 should lead to the formation of a chelate complex while the use of glycine in the application as filed does not.

2.2.4 In the written proceedings, the appellants argued that the product obtained in D6 differed from that of claim 1 in that the product of D6 did not have a "stoichiometric formulation" (presumably implying that the stoichiometric ratio of the product obtained in D6 is different from that required in claim 1).

The board acknowledges that the stoichiometric ratio of the amino acid to manganese salt applied as **starting materials** in preparation method 6 of D6 is 1:1, while it is 2:1 in the formula as defined in claim 1 for the **final complex** ($n = 2$ in the formula of claim 1; see point 2.1 above). However, it can at the very least not be excluded that with a stoichiometric ratio of the starting materials of 1:1, as is used in D6, some of the resulting complexes will have a stoichiometric ratio of 2:1, i.e. as defined in claim 1. More specifically, the stoichiometric ratio of the starting materials does not necessarily equate to the stoichiometric ratio in the end product, since the latter is not exclusively determined by the stoichiometric ratio of the starting materials but additionally depends on e.g. the pH (see for instance the paragraph bridging columns 3 and 4 of D6). It would therefore have been up to the appellants to prove that preparation method 6 of D6 does not lead to products that comprise complexes with a stoichiometric ratio as defined in claim 1. The appellants have however not provided any such evidence and in fact did not elaborate on the stoichiometric ratio as a distinguishing feature during the oral proceedings.

Therefore, the board's finding made above, and already expressed in its preliminary opinion, still applies, namely that in view of the fact that the process of D6 must be assumed to be identical to the process of claim 10, the product obtained by this process in D6 must be identical to that of claim 1.

2.2.5 D6 thus is novelty-destroying for the subject-matter of claim 1.

3. *Sufficiency of disclosure*

3.1 In view of the above, there is in principle no need to address the board's further objections in the present decision. Since, however, sufficiency of disclosure was an important point in both the written and oral proceedings, this issue is dealt with in the present decision as well.

3.2 As set out above, claim 1 requires that the claimed salt is not a chelate. For the invention underlying claim 1 to be sufficiently disclosed, the skilled person must therefore be able to prepare salts having the features of claim 1 and not being chelates. As set out in the board's preliminary opinion, in this respect, D1 is relevant.

3.2.1 D1 relates to the preparation of copper complexes (column 3, lines 3 to 4). In example 3, the copper complex is prepared by a process comprising the steps of

- preparing a solution of 800 pounds (2210 mol) lysine monohydrate in water and adding 600 pounds (1090 mol) of copper sulphate pentahydrate;
- heating the resulting solution to 160° to 180°F (71.1° to 82.2°C) to allow the complexing reaction to occur;
- to obtain a solution that is filtered.

The copper sulphate corresponds to the previously formed salt of claim 10. The solution of lysine monohydrate in water corresponds to the aqueous solution of amino acid of claim 10. Consequently, the

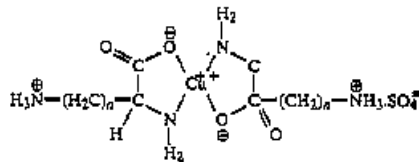
addition of the copper sulphate to the solution of the lysine monohydrate in example 3 corresponds to step a) of claim 10.

Heating to 71° to 82.2°C corresponds to the heating of claim 10. By way of the addition of the copper sulphate to the amino acid and the subsequent heating, a certain pH will result, corresponding to the pH adjustment of claim 10. Consequently, the step of heating the resulting solution to 71.1° to 82.2°C to allow the complexing reaction to occur in example 3 corresponds to step b) of claim 10.

The obtaining of a solution that is dried in example 3 corresponds to step c) of claim 10.

As set out in the board's preliminary opinion, the process of example 3 of D1 is thus identical to the process of claim 10.

3.2.2 The complex obtained in example 3 satisfies the following general formula (column 3, lines 55 to 65):



3.2.3 As stated by the appellant on page 5 of the statement of grounds of appeal (letter of 27 February 2012), this formula represents a chelate. Consequently, carrying out the process of claim 10 leads to a chelate rather than the salt of claim 1, which is not a chelate. This

implies that certain additional technical measures are necessary to obtain the salt of claim 1. The application as filed however does not provide any guidance as to any such additional technical measures. In view of this, the skilled person is not able to prepare salts with the constituents as defined in claim 1 and not having a chelate structure.

3.2.4 As mentioned above, this objection was already raised in the board's preliminary opinion. No comment was offered by the appellants either in their written response to the board's preliminary opinion or at the oral proceedings.

3.2.5 The invention as defined in claim 1 is thus insufficiently disclosed.

3.3 Claim 1 refers to "[s]alts of mineral nutrients associated with amino acids and/or ammonium salt" and requires *inter alia* that the metallic cations of these salts are bound "by a covalent dative bond with the amino group of the amino acids and/or ammonium salt". In its letter of 14 March 2014, the appellant stated that the ammonium salts can be inorganic. Thus, claim 1 covers a salt of mineral nutrients wherein the metallic cation of the salt is bound by a covalent dative bond with the "amino group" of an inorganic ammonium salt, such as ammonium sulphate.

The board acknowledges that a non-charged nitrogen atom in an amino group, such as the amino group of an amino acid, contains a non-bonded electron pair which it can give away in the form of a covalent dative bond to a metallic cation. As set out by the board during the oral proceedings, and not disputed by the appellants, it is however inconceivable that the same is true of

the ammonium group in an inorganic ammonium salt. More specifically, unlike in an amino group, the nitrogen in the ammonium group, due to its positive charge, does not contain an unbonded electron pair and hence cannot give away such an electron pair in the form of a covalent dative bond.

Since no teaching is present in the application as to how a covalent dative bond between an inorganic ammonium salt and the metallic cation can be achieved, and since the formation of such a bond in fact contradicts chemical principles, the alternative of claim 1 relating to a salt of mineral nutrients associated with an inorganic ammonium salt is not sufficiently disclosed.

- 3.4 In view of this finding, there is no need to address the further objections raised by the board with regard to the requirements of Articles 123(2) and 84 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



M. Cañueto Carbajo

W. Sieber

Decision electronically authenticated