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## Datasheet for the decision of 15 October 2008

Case Number:	т 0071/06 - 3.2.07
Application Number:	00979685.5
Publication Number:	1258535
IPC:	C22B 3/26
Language of the proceedings:	EN

## Title of invention:

Process for electrolytic production of highly pure zinc or zinc compounds from primary and secondary zinc raw materials

### Patent Proprietor: Tecnicas Reunidas, S.A.

rechicas keunitas,

## Opponent:

Outokumpu Oy

#### Headword:

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## Relevant legal provisions:

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EPC Art. 14(2), 54, 56, 70(2), 84, 113(1), 114, 123(2)(3), RPBA Art. 13
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# Relevant legal provisions (EPC 1973):

EPC Art. 150(3), 158(2)

#### Keyword:

"Admissibility of the four requests filed one day before the oral proceedings (all admitted) "Admissibility of the amendments (main and first auxiliary requests - no; second and third auxiliary requests - yes)" "Admissibility of late-filed document (yes)" "Inventive step (second and third auxiliary requests - no)" "Request for re-opening the discussion of admissibility of the amendments in the auxiliary requests (refused)" "Request to record matter in the minutes of the oral proceedings (refused)" "Request for adjournment of oral proceedings (refused)"

#### Decisions cited:

Т 0095/83, Т 0526/92, Т 0928/98, Т 0550/04, Т 0263/05

#### Catchword:

Procedural requests (see points 5 to 7.4 of the reasons)



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Beschwerdekammern

Boards of Appeal

Chambres de recours

**Case Number:** T 0071/06 - 3.2.07

#### DECISION of the Technical Board of Appeal 3.2.07 of 15 October 2008

Appellant:	Tecnicas	Reunidas,	S.A.
(Patent Proprietor)	Arapiles	, 13	
	E-28015	Madrid (	(ES)

Representative:

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Respondent: (Opponent) Outokumpu Oy Riihitontuntie 7-9 SF-02200 Espoo (FI)

Representative:

Zipse Habersack Kritzenberger Patentanwälte Wotanstraße 64 D-80639 München (DE)

Decision under appeal: Decision of the Opposition Division of the European Patent Office posted 23 November 2005 revoking European patent No. 1258535 pursuant to Article 102(1) EPC.

Composition of the Board:

Chairman:	P.	O'Reilly
Members:	н.	Hahn
	Ε.	Dufrasne

#### Summary of Facts and Submissions

- I. The patent proprietor lodged an appeal against the decision of the Opposition Division to revoke European patent No. 1 258 535.
- II. In this decision the following documents are cited:

D1 = 
$$WO-A-94/28184$$

D2 = International Symposium-World Zinc '93, Hobart, 10-13 October 1993, "Improved Jarosite Process"; pages 281-286

D4 = US-A-4 401 531

D5 = "Productivity and Technology in the Metallurgical Industries", TMS-Publication; Dr. Michael Koch and John C. Taylor; September 17-22, 1989

D6 = US-A-4 124 462

- D10 = Proceedings of International Symposium on Extractive Metallurgy of Zinc, October 14-16, 1985, Tokyo, Japan, chapter 48, pages 763-781
- D10a = Pb & Zn '05, International Symposium on Lead & Zinc Processing, http://www.mmij.or.jp/leadzinc2005/, pages 1-2, and Zinc '85 - Proceedings of International Symposium on Extractive Metallurgy of Zinc, October 14-16, 1985, Tokyo, Japan, The Mining and Metallurgical institute of Japan, pages i-viii
- D11 = WO-A-01 44520 (published Spanish language PCT application underlying the patent in suit)

- D12 = English translation of D11 as filed at the EPO on 11.07.2001
- Annex A = Internal report, Further Experimental Results
  as filed by the appellant with letter dated
  27 December 2007
- Annex A1= "Solvent Extraction, principles and applications to process metallurgy", G.M. Ritcey and A.W. Ashbrook, 1979, pages 62-63
- Annex A5= Espanola de Zinc Internal Report No. ITR/P-4506/010/1999
- Annex A6= H. Fuls et.al.: "Solvent Extraction in Zn production from a Primary Source: The Skorpion Zinc Experience", ISEC'05, Beijing, pages 1223-1228
- III. An opposition was filed against the patent in its entirety under Article 100(a) EPC, for lack of inventive step, and under Article 100(b) EPC, that the patent does not disclose the invention of claim 6 in a manner sufficiently clear and complete for it to be carried out by the person skilled in the art.

The Opposition Division held that the claims 1 to 18 of the main request and claims 1 to 16 of the auxiliary requests I and II, all as filed at the oral proceedings of 3 November 2005, met the requirements of Articles 123(2) and (3) EPC. The Opposition Division considered that the subject-matter of claim 1 of all three requests was novel and that novelty had not been disputed. However, the process of claim 1 of the main request was considered to lack an inventive step with respect to the closest prior art D1 and the common general knowledge and normal practice of the person skilled in the art, as proven by D2 or D3. The subjectmatter of claim 1 of auxiliary request I was considered to lack an inventive step with respect to D1 while claim 1 of auxiliary request II was considered to lack an inventive step in view of D1 and the common practice of the skilled person as exemplified by D4 and D6. The impugned decision was, however, silent with respect to the issue of Article 100(b) EPC although this issue had been discussed at the oral proceedings during which the Opposition Division had come to the opinion that the requirements of Article 100(b) EPC are fulfilled (see Minutes dated 23 November 2005, point 2).

IV. With a communication annexed to the summons to oral proceedings dated 28 July 2008 the Board presented its preliminary opinion based on claims 1-18 of the single request as filed with the grounds of appeal dated 31 March 2006 (which is identical with the main request underlying the impugned decision).

> With respect to the issue of inventive step D1 was considered to represent the closest prior art for the claimed process for the continuous production of high purity electrolytic zinc or zinc compounds starting from zinc concentrates to be leached by an aqueous acidic solution and subsequently to be extracted by an organic solvent.

The object of D1 is to provide a method for separating zinc and particularly zinc metal from zinc concentrates, and especially from lower grade zinc sulphide concentrates, in a manner which minimises input costs and unusable waste products and which is energy efficient when compared to the smelting and roast/leach techniques, and also does not produce toxic products such as  $SO_2$ .

The subject-matter of claim 1 appeared to be distinguished from the process according to D1 in that the neutralization is conducted in zones arranged in cascade mode and that a purification treatment of the zinc loaded organic solvent is carried out by a physical purification with an acidified water washing solution in an organic/aqueous rate between 5-50 followed by a chemical washing step with an aqueous zinc acid solution containing from 10 g/l to 100 g/l Zn and from 0.1 g/l to 1.0 g/l of equivalent H<sup>+</sup> (i.e. "a physical purification zone" and "a chemical purification zone").

Taking account of these distinguishing features the technical object to be solved - which is solved by the subject-matter of claim 1 - will have to be defined. Both parties have not yet defined an objective problem based on the above distinguishing features with respect to D1.

According to the impugned decision the object is to provide a process for high purity electrolytic zinc or zinc compounds being capable to treat zinc containing raw materials of different origin, whether primary or secondary raw materials, and, at the same time, to perform at high recovery yield and deliver zinc or zinc compounds of extreme purity.

Thus it needed to be discussed as to which technical problem is to be solved by the said distinguishing features and whether or not the solution chosen is

rendered obvious and/or suggested by the available prior art documents.

The Board stated that the following points should be considered.

According to the Figure and Table of the patent in suit only water - and no acidified water (see patent, paragraph [0049] and claim 22) - is used for the physical washing step (this view appears to be supported by Annex A6 which specifies demineralised water for use in the physical washing step; see page 1225, paragraph "Solvent-Extraction Circuit" and Figure 3). Furthermore, according to the example of the patent in suit said diluted aqueous electrolyte solution L10 has an acid equivalent of 3.4 g/l H<sup>+</sup> whereas according to claim 1 of the main request it should **only** have an acid equivalent of between **0.1-1.0** g/l H<sup>+</sup>. Thus said only example is **not** covered by claim 1. On the other hand the washing step according to the example of D1 (the scrub solution at stage 19 comprises 5 g/l Zn and 7.5 g/l  $H_2SO_4$  amounting to 0.15 g/l equivalent H<sup>+</sup> and is obtained by diluting zinc electrolyte from the electrowin step 20) does not represent chemical washing since the amount of zinc is outside the range of 10-100 g/l Zn according to claim 1 of the patent in suit. It is, however, remarked that the appellant has not shown that an effect occurs **only** in said concentration ranges 10-100 g/l Zn and 0.1-1.0 g/l of equivalent  $H^+$  according to claim 1.

From the text book Annex A1 the person skilled in the art knows that physical entrainment of the aqueous phase in the loaded organic phase can usually be overcome by scrubbing said loaded solvent with water or some suitable solution. He knows further from the same passage that co-extraction and particularly the chemical type thereof can be dealt with in two ways. Namely by either using an acidic solution having a sufficient pH at which the unwanted metal is stripped off while the wanted metal is left in the organic phase, or by using a solution of the metal of interest which by contacting the loaded solvent replaces the unwanted (co-extracted) metal with the wanted metal. Thus an acidic salt solution of the metal of interest having a specific pH range appears to combine these two effects.

The passage of D1 concerning the scrubbing needs to be interpreted. Does it mean that, if there is more than one scrubbing stage, i.e. there are at least two scrub stages, that all stages use the same scrub solution, i.e. either an acidified solution or a zinc sulphate solution, or does it mean that the at least two different stages use different solutions, e.g. one an acidified solution and the other one a zinc sulphate solution? It should be discussed as to how the person skilled in the art would interpret this passage?

The meaning of the feature in claim 1 "A process ... which **comprises** the following steps: ... the physical washing **comprises** the washing with **an acidified water** solution ..." should be discussed. In this context it needs to be considered that, if said acidified aqueous solution (or water) is used as scrub liquor for removing entrained aqueous phase from the organic solvent, i.e. an aqueous phase comprising zinc and sulphuric acid at a pH of between 3 and 5, that these ingredients will be enriched in the solution due to its circulation during the use in said scrubbing stage. Thus the composition of the solution of the physical washing step changes during the circulation and thereby will have an increasing Zn and  $H_2SO_4$  content.

In this context also the experimental tests of Annex "A" (Internal Report) as submitted by the appellant with letter dated 27 December 2007 will be discussed. According to these tests **the physical washing liquor was obtained by diluting the chemical washing liquor** (which contained between 10-100 g/l Zn and between 0.1-1.0 equivalent  $H^+$ ) to have a final composition with  $\leq$ 0.3 equivalents/l. Likewise the comparative tests as submitted with the grounds of appeal may be discussed.

A zinc purity of greater than 99.995% according to the patent in suit appeared to be only credible for electrowinning while claim 1 of the main request includes crystallization and/or precipitation of the zinc as well.

The appellant was requested to submit a translation of Annex A5 - which is a Spanish patent - in one of the three official languages of the EPO, at the latest one month before the date of the oral proceedings set (Rule 3(3) EPC) if the Board should consider it.

- V. With a letter dated 3 September 2008 the respondent submitted comments concerning the Board's annex to the summons together with the new documents D10a and D10.
- VI. With letter dated 11 September 2008 the representative of the appellant resigned from patentee's representation as requested by the patentee.

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With letter dated 15 September 2008 the new representative of the appellant/proprietor submitted amended main, and first to third auxiliary requests. Furthermore, it was requested to postpone the oral proceedings because further discussions would be necessary between the new representative just appointed and its client, particularly in view of the complexity of the case, which should require travel of the new representative to meet its client in Madrid. It further raised the need for the Board and for the respondent to deal with the amendments recently introduced, which should also constitute a reasonable statement for granting the request of adjournment, according to Article 13(3) of the Rules of procedure of the Boards of Appeal (RPBA).

With Fax of the appellant dated 16 September 2008 it was announced that the new representative would be accompanied by the appellant's Spanish attorney and three employees of the appellant at the oral proceedings.

VII. With communication dated 24 September 2008 the Board informed both parties that the reasons presented by the appellant for a postponement of the oral proceedings were not considered to fall under the serious substantive reasons in the meaning of items 2.2 and 2.3 of the Notice of the Vice-Presidents DG2 and DG3 dated 1 September 2000 concerning oral proceedings before the EPO. It further stated that all possible serious substantive reasons cited as examples under item 2.3 of the Notice are reasons which do not depend on any initiative or decision which the party has taken after the notification of the summons to oral proceedings. On the contrary, in the present case, the main reason provided by the appellant is the direct consequence of its own decision to change its representative. The Board cannot see such a free choice after the notification of the summons to oral proceedings as a serious substantive reason within the meaning of the above cited Notice.

As a further reason for postponement, the appellant raised the need for time for the Board and for the respondent to consider newly filed amended requests. Again, this situation only results from the appellant's own choice and cannot as such convincingly support its request for postponement of the oral proceedings. Article 13(3) RPBA, cited by the appellant, on the one hand intends to consider issues to be possibly raised by the other party or the Board in view of such late amendments and on the other hand does not provide support for postponement of the oral proceedings but for amendments not to be admitted.

Therefore the Board refused this request and the oral proceedings scheduled to take place on 15 October 2008 were maintained for that date.

- VIII. With fax dated 14 October 2008 the appellant submitted a new main, and first to third auxiliary requests in combination with an adapted description. It also submitted a reasoning for the amendments made to claims 1 thereof as well as the basis for them.
- IX. Oral proceedings before the Board were held on 15 October 2008. After discussion of the admissibility

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of the main and first to third auxiliary requests dated 14 October 2008 the Board considered these requests admissible. Thereafter the allowability of the amendments of all requests with respect to Articles 123 and 84 and Rule 80 EPC was discussed. After deliberation the Board concluded that claim 1 of the main and first auxiliary requests do not comply with Article 123(2) EPC while claim 1 of the second and third auxiliary requests were considered to comply with Articles 123(2) and (3) EPC.

Thereafter the admissibility of document D10 was discussed and the Board came to the conclusion that D10 was admissible.

At the start of the discussion on inventive step the respondent stated that its right to be heard has been breached and wished to return to the issue of Article 123(3) EPC of claim 1 of the second and particularly the third auxiliary request. A first discussion of this issue resulted in the respondent's written request: "It is herewith requested to reopen the discussion of admissibility of amendments in the auxiliary requests. In case this request is not admitted it is auxiliary requested to take account in the protocoll of the proceedings that the opponent has not been heard with regard to the question of admissibility of auxiliary requests 1-3 concerning amendments made therein".

After a further discussion to clarify whether the appellant had understood the chairman's original intention to deal with the formal aspects of all requests in one go, these two requests of the respondent were refused by the Board after deliberation.

Thereafter the issue of inventive step was discussed with respect to claim 1 of the second auxiliary request since novelty had not been disputed by the respondent. In the course of this discussion the appellant requested an adjournment of the oral proceedings in order to carry out comparative tests with respect to D10. Thereafter inventive step of claim 1 of the third auxiliary request was discussed as well as the request for adjournment of the oral proceedings which eventually was refused.

At the end of the oral proceedings the Board announced its decision.

X. The appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request or, in the alternative, on the basis of one of the first to third auxiliary requests, all filed with the letter dated 14 October 2008.

The respondent requested that the appeal be dismissed.

XI. Claim 1 of the main request under consideration reads as follows:

> "1. A process for the continuous production of a top purity electrolytic zinc or zinc compounds of high purity, starting from zinc containing raw materials to be extracted by a hydro-metallurgical treatment which comprises the following steps:

a) if the raw material is solid, leaching said raw material by an acidic aqueous solution in order to dissolve the zinc, wherein the pH of the aqueous media resulting in leaching is between 0 and 3 at the end of the leaching steps,

b) optionally, depending upon the raw material, if the leaching residue have any valuable component, a solid/liquid separation would be included after leaching,

c) neutralizing the aqueous solution or pulp, or the zinc containing solution when the raw material in liquid form, wherein the pH of the aqueous media resulting from the leaching is between 3 and 5 at the end of the neutralization steps,

d) solid/liquid separating of the zinc rich aqueous solution from the solids,

e) extracting the zinc contained in the pregnant aqueous solution by an organic acidic solvent, wherein the extraction of the zinc contained in the zinc rich neutralized aqueous solution is conducted by means of an organic acid solvent, chosen in the group constituted by alkyl phosphoric acids, alkyl phosphonic acids, alkyl phosphinic acids,

f) purifying the zinc rich loaded organic solvent coming from the extraction step e),

g) stripping the ionic zinc from the zinc loaded organic solvent by an acidic solution,

 h) recovering the zinc from the zinc containing acidic aqueous solution coming out the stripping stage by electrowinning and/or crystallization and/or

## precipitation,

wherein:

- for solid raw material case, the acid leaching of the raw material, according to step a) is conducted in "n"

leaching zones, arranged in a cascade mode, "n" being at least equal to 2,

- the neutralization according to step c), is conducted in "p" zones arranged in a cascade mode, "p" being at least equal to 2,

- the purification treatment of the zinc loaded organic solvent, according to step f), is conducted in "q" physical and "r" chemical purification (successive) zones, organic and aqueous streams operating in counter current mode, the number "q" of such physical purification treatment zones being comprised between 1 and 4 and the number "r" of such chemical purification treatment zones being comprised between 1 and 4, wherein the physical treatment comprises the washing with an acidified water solution of the zinc loaded organic solvent from extraction in an organic/aqueous rate between 5 and 50, and wherein the chemical purification treatment is conducted by means of an aqueous zinc acid solution containing from 10 g/l to 100 g/l of zinc and at least 0.1 g/L of equivalent  $H^+$ , and in that a part of the zinc rich neutralized liquid solution and/or of the aqueous zinc unloaded solution originating from extraction is treated by means of an alkali reagent, in order to adjust the water and/or alkaline element balance and increase the zinc recovery and control the building up of dissolved impurities, said part of the zinc rich neutralized liquid solution and/or of the aqueous zinc unloaded solution originating from extraction representing at most 25% of the volume of said solution in volume."

XII. Claim 1 of the first auxiliary request differs from claim 1 according to the main request in that the

feature of step h) "and/or crystallization and/or precipitation" has been deleted.

- XIII. Claim 1 of the second auxiliary request differs from that of the main request in that the concentration range of equivalent H<sup>+</sup> of "at least 0.1 g/l of equivalent H<sup>+</sup>" has been amended to read "and from 0.1 to 5 g/L of equivalent H<sup>+</sup>".
- XIV. Claim 1 of the third auxiliary request differs from claim 1 according to the second auxiliary request in that the feature of step h) "and/or crystallization and/or precipitation" has been deleted.

XV. The appellant argued essentially as follows:

The amendments made to claim 1 of the main and first to third auxiliary requests (which are based on the corresponding previous requests dated 15 September 2008) were made for two reasons. Firstly, to establish clarity of a feature amended during the opposition procedure (i.e. "r"+"q" being at least 2) which could be interpreted as meaning that either "r" or "q" could be 0. This lack of clarity was removed by introducing all features of dependent claim 21 as granted. Thereby the restriction of "more than 1" was changed to "1 to 4". Secondly, it was only very recently that the appellant became aware of the fact that the English translation (D12) of the Spanish language PCT application as originally filed (D11) contained a mistake. Due to this mistake the originally disclosed concentration range of acidity of the solution used in the chemical washing zone of "0.1 to 5 g/L equivalent  $H^+$ " according to D11 had been changed into "0.1 to 1 g/L

of equivalent H<sup>+</sup>" in D12. This erroneous value in the English translation was then used when filing further applications including the one at the EPO. In view of Articles 14(2), 70(2) and 158(2) and Rule 80 EPC this amendment "0.1 to 5 g/l of equivalent H<sup>+</sup>" made in claim 1 of the second and third auxiliary request is occasioned by a ground of opposition under Article 100 EPC and does not violate Article 123(2) EPC as laid down in Article 70(2) EPC, since the original language document defines the content of the application as originally filed.

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The subject-matter of claim 1 of all four requests is principally based on claims 1, 9, 13, 16, 17, 18, 21, 22 and 23 as granted with the following further amendments. The feature "0.1 to 1 g/l of equivalent  $H^+$ " comprised in claim 1 of the main request underlying the contested decision - which now has been replaced by either "at least 0.1 g/L of equivalent  $H^+$ " or "0.1 to 5 q/l of equivalent  $H^+$ " - was not comprised in claim 1 as granted so that the amendments do not violate Article 123(3) EPC. Furthermore, as the feature "0.1 to 5 g/l of equivalent  $H^+$ " in accordance with Articles 14(2), 70(2) and 158(2) EPC is taken from Spanish language original document D11 this amendment should also comply with Article 123(2) EPC. The same conclusion applies to the deletion of the feature "and/or crystallization and/or precipitation" from claim 1 of the first and third auxiliary requests which represented only an alternative. It is offered to reverse the order of the second and third auxiliary requests and the main and first auxiliary requests, respectively. The amendment made to claim 1 of the main and first auxiliary requests ("at least 0.1 g/L of

equivalent  $H^+$ ") is derived from the lower value 0.1 of said range in combination with the implicit upper acidity value of this solution. The upper limit of the range constitutes an unnecessary restriction of the scope of claim 1. The present case cannot be compared with the conclusion reached in T 526/92 dealing with changing of ranges. In the present case it has to be considered that it is important that the washing solution has to be an acidic **solution** based on the three components water, zinc sulphate and sulphuric acid. However, at certain zinc sulphate and sulphuric acid concentrations the solubility limit of zinc sulphate in this 3-component system is reached so that it would precipitate. Hence this feature "at least 0.1 g/L of equivalent H<sup>+</sup>" does not define an open end since the acidity is not unlimited for solubility reasons.

Three aspects have to be discussed with respect to the late filing of D10. It was filed about one month before the oral proceedings and a copy was received on 16 September 2008 with letter dated 3 September 2008. The complete case should, however, be presented at the beginning of the appeal procedure. The copy of D10 stemming from the proprietor was confidential and only given to clients. Thus the public availability is questioned and a publication proof was alleged to be presented. It is admitted that an International Symposium is open to the public and that it is reasonable to assume that the content of such an International proceedings will have been published within 5 years; however, the publication date of D10 is not known. Also its relevance is doubtful and its content does not go beyond the information already on

file. Therefore D10 should not be admitted into the proceedings.

It is true that, as stated by the Chairman, the formal aspects, i.e. Articles 84, 123(2) and (3) and Rule 80 EPC should be discussed for all requests in one go. Although the basis and justification of the amendments made to claim 1 of the second and third auxiliary request were presented first, those for claim 1 of the main and first auxiliary request were also given.

Novelty has not been disputed (see impugned decision).

With respect to the issue of inventive step it is requested to adjourn the proceedings to properly deal with the late filed D10 which besides the process fundamentals and the testing of a pilot plant also mentions a physical washing and a chemical washing of the organic phase after the extraction step (see pages 772-774 and 776). The chemical washing described in D10 is not a chemical washing with an acidic solution as required by claim 1 of the second auxiliary request which defines the concentrations of the incoming solutions. On the contrary a neutral zinc sulphate solution is used (see page 774). It is not known which exact conditions are used for obtaining the final zinc quality of 99.95% Zn. The impurity levels (such as those of Cu and Cd) of the process according to Table 3 do not allow obtaining zinc cathodes having the purities of 99.9959% Zn or 99.9971% Zn according to test run no. 6 (see Table 4). Thus there is no enabling teaching. These values may also not be correct since the analyses were made in-house. Furthermore, said test runs no. 6 lasting only 280 and 240 hours do not

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represent a long-term run of the process. According to D10 bleed-off streams are necessary to maintain the chloride content below 12 g/L which restriction is not necessary according to claim 1. Furthermore, D10 only deals with zinc secondaries. According to D10 gypsum precipitation takes place in the last settler (see page 770 and also Figure 3 at page 769) and the Ca concentration has to be kept at  $\leq$  0.3 g/L to avoid said precipitation (see pages 775-776). The technical problem to be solved by the subject-matter of claim 1 of the second auxiliary request is the provision of a process for the continuous production of electrolytic zinc or zinc compounds which allows to obtain the products with improved purity and in high yield starting from raw materials of different origins including primary and secondary raw materials (see patent in suit, paragraphs [0001] and [0015]). The example, which is now covered by claim 1, shows that this object is solved. The process is capable of producing an organic solvent containing an amount of Fe and Al of only 0.35 g/L (without Ca) after the washing step (see D11, page 13, Table A). Hence there exists an outstanding and surprising technical effect as proven by the comparative tests (see letter dated 31 March 2006, pages 7 and 8, Tables 1 and 2; and Annex A, Table 2.1). The solution according to D5 is equal to that used according to D10, i.e. a neutral zinc sulphate solution is used in the chemical washing step. The assumption of hydrolysis of zinc sulphate results in an acidity which is far below the range of claim 1. Claim 1 does not represent a selection invention since this would need a document disclosing a broader range from which then a smaller range has to be selected. It is admitted that the use of counter current in the

washing steps as well as the cascade mode are known. D1 discloses only scrubbing with a diluted zinc electrolyte containing 5 g/L of Zn and 7.5 g/L of  $H_2SO_4$ (corresponding to 0.15 g/L of equivalent  $H^+$ ) in two stages and mentions that acidified water or a zinc sulphate solution could be used (see page 10, lines 19 to 23; page 16, lines 24 to 34). However, the process results only in a purity of 99.99% Zn (see page 9, line 27). The remaining documents can also not suggest the subject-matter claimed for disclosing only water, or neutral solutions, or diluted (sulphuric acid) acidic solutions. The skilled person has no reason to amend the processes of D1 or D10. Only hindsight allows to arrive at the claimed subject-matter. Hence the process of claim 1 of the second auxiliary request involves an inventive step.

The above arguments are also valid for the fallback position of claim 1 of the third auxiliary request which had been limited to the electrowinning alternative.

The request for adjournment of the oral proceedings with respect to D10 is maintained. The appellant wishes to carry out comparative tests to show that the claimed process achieves a different purity of the resulting zinc product.

XVI. The respondent argued essentially as follows:

All requests dated 14 October 2008 have been late filed and should not be considered admissible. According to T 95/83 amendments in claims should only exceptionally be admitted if there exists a clear justification for

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the amendment and its late submission. Such a condition is not fulfilled in the present case. Furthermore, due to the amendment for the sake of clarity a new issue with respect to the selection of 1 to 4 washing zones in the physical and chemical washing steps is created. The question as to whether these amendments are formally admissible is not relevant for their admissibility. These amendments should have been filed one month earlier.

The feature "at least 0.1 g/L of equivalent H<sup>+</sup>" has no basis in the originally filed application and additionally has an undefined upper value. Furthermore, this amendment contravenes Article 123(3) EPC for extending the protection. Such an extension can also arise when only the description or a drawing of the granted patent has been amended.

With respect to D10 no further statements can be made. It was received from a competitor of the patent proprietor though not the opponent only shortly before its submission with letter dated 3 September 2008. The International Symposium took place on 14-16 October 1985 as proven by D10a. D10 was actually the book which was sent out and there is no question as to what has been disclosed. Its relevancy is proven by the cascade mode of the neutralization (see page 768) and the combination of physical and chemical washing (see page 774). Therefore D10 should be admitted into the proceedings. The content as such should be familiar to the proprietor since D10 belongs to it.

The respondent was deprived of its right to be heard with respect to the amendments of the second and third

auxiliary requests and particularly with respect to Article 123(3) EPC since the incorporated feature "0.1 to 5 g/L of equivalent  $H^+$ " extends the scope of protection of claim 1 of the second and third auxiliary requests beyond that of claim 1 as granted. This deprivation of right should be incorporated into the minutes of the oral proceedings. It was unclear which requests were actually discussed at that time. It was the representative's understanding that only the main request was to be discussed and thereafter would be decided by the Board. Therefore it is requested to reopen the issue of the formal aspects concerning the auxiliary requests; alternatively it is requested to take account in the minutes of this fact (see written request dated 15 October 2008). It is admitted that the arguments concerning the feature "at least 0.1 g/L of equivalent H<sup>+</sup>" of claim 1 of the main request likewise applied to claim 1 of the auxiliary request.

Most of the arguments of the appellant with respect to inventive step and the subject-matter of claim 1 of the second auxiliary request cannot be accepted, e.g. the patent in suit can also use zinc secondaries as a raw material (see paragraph [0001]) while the purity is not reflected as a feature of claim 1. The question to be answered is only whether or not it is possible to arrive at the technical features of claim 1. The cascade mode of the neutralization and the leaching of the raw materials belongs to the state of the art, as admitted by the appellant and can be found e.g. in D10. D10 further discloses the combination of physical (2 steps washing with acidified water) and chemical washing (1 step washing with a small stream of spent acid or spent electrolyte) to remove the impurities

from the organic phase resulting from the extraction step. The higher the acid content of the solution in the chemical washing step the higher will the amount of Zn washed out of the organic phase. The hydrolysis of zinc sulphate should also be considered. According to D1 the solution contained 0.15 g/L of equivalent  $H^+$ . D6 discloses that the washing is carried out in one or more steps with diluted acid but the acid content may not be so high as to result in any zinc being washed out from the organic phase (see column 3, lines 46 to 53). Taking account of the fact that e.g. cadmium is removed from the organic phase during the washing it is clear that the documents D1, D6 and D10 disclose a combination of physical and chemical washing in the sense of the patent in suit. The selection of an acid content for the chemical washing step which is sufficient to wash out the impurities but not so high as to wash out the zinc resides within the normal competence of the person skilled in the art. In this context the "rule of thumb", i.e. the higher the better, of the appellant should also be considered which was stated to cover the broad range of between 0.20 mol/L and higher as 5 mol/L for the total of dissolved components in the chemical washing solution. The acid concentration range of claim 1 of "0.1-5 g/L of equivalent H<sup>+</sup>" corresponds to a factor of 50 and is thus not small. Furthermore, as stated by the appellant "The washing is never 100% physical or 100% chemical. These effects go always together" (see the grounds of appeal dated 31 March 2006, page 5, first paragraph). The distinguishing features between claim 1 of the second auxiliary request and the process of D10 are the concentration ranges of Zn and equivalent  $H^+$ , and the organic phase/aqueous phase ratio. The problem to be

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solved for the person skilled in the art is thus to provide an optimum purification of the organic phase from the extraction and represents an optimisation problem. This problem can be solved by incorporating the washing stages of D10 into the zinc electrowinning process of D1. Thereby the person skilled in the art arrives at the subject-matter of claim 1 of the second auxiliary request without any inventive skill. Thus the subject-matter of claim 1 lacks an inventive step over the combination of the teachings of D1 and D10.

There is nothing to be added with respect to claim 1 of the third auxiliary request the subject-matter of which likewise lacks an inventive step for the same reasons as the second auxiliary request.

Legal grounds for an adjournment of the oral proceedings cannot be seen. It is also not understood as to how such proposed comparative tests would be relevant. They should be made with respect to D10 which discloses the use of an acidic solution in the physical washing steps and the use of an acidic solution having certain zinc content in the chemical washing step. Thus the suggested comparative tests shall be made to only prove what has been done by D10. Furthermore, the proprietor had sufficient time to carry out such comparative tests. Therefore this request represents a very late filed request and thus should be refused.

## Reasons for the Decision

1. Admissibility of the four requests filed one day before the oral proceedings (RPBA Article 13) - 24 -

- 1.1 On 14 October 2008, i.e. one day before the scheduled oral proceedings, the appellant withdrew all its previous requests dated 15 September 2008 and replaced them by a main and first to third auxiliary requests (see points XI to XIV above). It argued that the first amendment was necessitated to overcome a possible objection under Article 84 EPC with respect to an amendment made to claim 1 during the opposition proceedings. The second amendment in the second and third auxiliary requests served to remove a mistake which the patent proprietor only very recently had noticed: the English translation D12 of the Spanish language PCT application D11 underlying the patent in suit contained a mistake. Whereas the Spanish original D11 disclosed an acidity concentration range of the solution used in the chemical washing zone of "0.1 to 5 g/L equivalent  $H^+$ " (see page 9, lines 16 to 18 and claim 23) this range had been changed into "0.1 to 1 g/L of equivalent  $H^+$ " in D12 (see page 12, lines 35 to 38 and claim 23). The patent in suit having been based on D12 still contained this concentration range with said erroneous upper value when it had been eventually granted.
- 1.2 The first amendment, which was comprised in claim 1 of the main and the first to third auxiliary requests in an identical manner, concerned the feature "the purification treatment of the zinc loaded organic solvent, according to step f) is conducted in "q" physical and/or "r" chemical purification (successive) zone(s), organic and aqueous streams operating in counter current mode, "r"+"q" being at least equal to 1" of claim 1 as granted which had been amended during the opposition procedure to read "the purification

treatment of the zinc loaded organic solvent, according to step f), is conducted in "q" physical and "r" chemical purification (successive) zones, organic and aqueous streams operating in counter current mode, "r" + "q" being at least equal to 2". The appellant argued that the latter feature could be interpreted as meaning that either "r" or "q" might be 0 with the other being 2. This possible lack of clarity, which from the Board's view likewise implies a possible violation of Article 123(2) EPC since it was the intention of the appellant to delete the "or" alternative and there exists no basis for "at least 2 zones", was overcome by introducing all features of dependent claim 21 as granted into claim 1 whereby the restriction of number of zones "being at least equal to 1" of claim 1 as granted was changed to "comprised between 1 and 4". Therefore the Board comes to the conclusion that this clarification of the amendment made to claim 1 of all requests was necessary to avoid objections under Article 84 and 123(2) EPC.

1.3 The further amendment made to claim 1 of the second and third auxiliary requests concerned the acidity concentration range which was changed from "0.1 to 1 g/L equivalent H<sup>+</sup>" to "0.1 to 5 g/L equivalent H<sup>+</sup>".

When comparing the passages of the Spanish language PCT application D11 and its English translation D12 quoted by the appellant it is obvious that the originally disclosed range "0.1 to 5 g/L equivalent  $H^+$ " had been changed by mistake to read "0.1 to <u>1</u> g/L equivalent  $H^+$ " in D12.

1.3.1 In accordance with Article 150(3) EPC 1973 an international application for which the European Patent Office is a designated or elected Office, and which has been accorded an international date of filing, shall be deemed to be European application (Euro-PCT application). The EPO was the designated Office for the international PCT application D11 which has an international filing date of 1 December 2000 and which was filed in the Spanish language. Therefore D11 falls under the provisions of Article 150(3) EPC 1973. Furthermore, D11 is the application underlying the patent in suit.

1.3.2 If, however, the European patent application has been filed in a language which is not one of the three official languages of the European Patent Office, that text shall be the application as filed within the meaning of this convention (Article 70(2) EPC).

> The Spanish language PCT application D11 is considered as the European patent application in accordance with Article 70(2) EPC for the purpose of Article 123(2) EPC.

Consequently, the range of "0.1 to 5 g/l of equivalent  $H^+$ " as originally disclosed in D11 can form the basis for an amendment in the patent in suit since the original language document D11 defines the content of the application as originally filed.

1.4 It is credible that the appellant only very recently noticed said mistake so that the amendment resulting therefrom could not have been filed much earlier. Consequently, from the Board's view there exists a clear justification for the amendment and its late submission in the present case.

- 1.5 These amendments do not require any substantial change to the arguments on file because those brought forward with respect to the narrower range of "0.1 to 1 g/L of equivalent H<sup>+</sup>" and with respect to "two or three washing zones" of the prior art still apply. Consequently, the respondent's arguments that a new issue with respect to the selection of 1 to 4 washing zones in the physical and chemical washing steps would be created cannot be accepted.
- 1.6 Therefore the Board exercises its discretion according to Rule 13(1) RPBA and admits the four late filed requests dated 14 October 2008 into the proceedings.
- 2. Admissibility of the amendments of claim 1 of all four requests (Articles 84, 123(2) and (3) EPC)
- 2.1 Claim 1 of the main request is principally based on claims 1, 9, 13, 16, 17, 18, 21, 22 and 23 as granted but comprises the amendment of the concentration range of claim 23 from "0.1 to 1 g/L of equivalent H<sup>+</sup>" into "at least 0.1 g/L of equivalent H<sup>+</sup>". The said claims as granted correspond to the respective claims 1, 9, 13, 18, 21, 22 and 23 as originally filed (see D11). However, the application as originally filed does not explicitly mention anywhere said range of "at least 0.1 g/L of equivalent H<sup>+</sup>".

2.1.1 Contrary to the appellant's arguments this range is also not derivable from the application as originally filed, let alone directly and unambiguously as required by the longstanding Case Law of the Boards of Appeal (see Case Law of the Boards of Appeal of the European Patent Office, 5<sup>th</sup> edition, 2006, chapter III.A.2.1) for the following reasons.

2.1.2 First of all, the application as originally filed only discloses that "the chemical purification treatment is conducted by means of an aqueous zinc acid solution containing from 10 g/L to 100 g/L of zinc and from 0.1 to 5 g/L of equivalent H<sup>+</sup>" (see D11, page 9, lines 16 to 18 and claim 23). This solution therefore comprises water, a zinc salt, an acid and in addition it may contain certain amounts of impurities resulting from the use of the solution in the purification treatment. When simply considering the aforementioned general definition of this solution it is evident that it does not define a specific three component solution consisting of water, zinc sulphate and sulphuric acid as argued by the appellant.

Secondly, it likewise cannot be accepted, as argued by the appellant, that the maximum acid concentration of the said aqueous zinc acid solution (when constituted only by said three components) would be defined by the solubility limit of the zinc sulphate in the sulphuric acid/water mixture. As proven by D6 the limit for the acidity of this solution is actually defined by that acid concentration above which the zinc is washed out from the organic phase (see D6, column 3, lines 46 to 53). This concentration, however, will be lower than the solubility limit of the zinc sulphate. 2.1.3 Therefore, the feature of claim 1 of the main request "at least 0.1 g/l of equivalent H<sup>+</sup>" contravenes Article 123(2) EPC. The main request is thus not allowable.

- 2.2 The same conclusion as in point 2.1.3 above applies mutatis mutandis to claim 1 of the first auxiliary request which contains the identical concentration range (see point XII above). Therefore the first auxiliary request is not allowable under Article 123(2) EPC, either.
- 2.3 Claim 1 of the secondary auxiliary request is based on claims 1, 9, 13, 16, 17, 18, 21, 22 and 23 as granted. The feature "0.1 to 5 g/L of equivalent H<sup>+</sup>" - which replaces the feature "0.1 to 1 g/L of equivalent H<sup>+</sup>" of claim 23 as granted - has a basis in the application as originally filed (compare point 1.3 above). Consequently, claim 1 of the second auxiliary request is considered to meet the requirements of Article 123(2) EPC.

Since the subject-matter of claim 1 of the second auxiliary request is more restricted than that of claim 1 as granted - which for example was not restricted to any concentration of the aqueous zinc acid solution for the chemical purification treatment it is considered to meet also the requirements of Article 123(3) EPC.

The respondent argued that the feature when contained in the patent as granted in a dependent claim was in a narrow form, viz. "0.1 to 1 g/L of equivalent  $H^+$ " so that its incorporation into claim 1 in a broader form offends Article 123(3) EPC. The Board cannot agree with this argumentation. The respondent has failed to realize that the change in the range when incorporating the dependent claim into claim 1 cannot broaden the scope of protection since the incorporation in either form is always a narrowing of the scope of protection.

The Board also considers that the amendments made to claim 1 of the second auxiliary request do not render it unclear and thus meet the requirements of Article 84 EPC.

2.4 Claim 1 of the third auxiliary request is, besides the deletion of the feature of step h) "and/or crystallization and/or precipitation", identical with that of the second auxiliary request (see point XIII above). The deletion of these two alternatives is, however, not objectionable under Article 123(2) EPC.

Consequently, claim 1 of the third auxiliary request is likewise considered to meet the requirements of Articles 123(2) and (3) EPC. The amendments made to claim 1 of the third auxiliary request does not render it unclear and therefore also meets the requirements of Article 84 EPC.

- 2.5 Therefore, the amendments made to the second and the third auxiliary requests being considered to be formally admissible, these requests are admitted into the proceedings.
- 3. Admissibility of document D10 (Article 114 EPC)

3.1 D10 was submitted by the respondent with letter dated 3 September 2008 who stated that it obtained the document itself only recently (see point V above). The appellant stated during the oral proceedings that the copy of D10 was actually received on 16 September 2008, i.e. only about one month before the date of the oral proceedings.

- 3.2 D10 concerns the presentation "Using Zinc Secondaries to feed an Electrowinning Plant" produced by employees of the patent proprietor for the International Symposium on extractive Metallurgy of Zinc "Zinc '85" which took place on October 14-16, 1985 in Tokyo, and later published in the proceedings of said international symposium (see D10, first page and page 763). This symposium "Zinc '85" was one event of regularly held International symposia on lead and zinc which was followed by the Lead-Zinc '90 in Anaheim, the Zinc & Lead '95 in Sendai and the Lead-Zinc 2000 in Pittsburgh (see D10a, page 1).
- 3.3 In the Board's view it is reasonable, absent any contrary element, to consider that a) such an international symposium is open to the public so that everyone interested can attend it (compare D10a, pages 1 and 2), and b) the presentations submitted at such a symposium will have been published, most often in the form of a book "proceedings of ...", within a period of time of at most 5 years. This will be the truer in the present case where the next international symposium already took place 1990, i.e. after a period of 5 years. The appellant when questioned by the Board confirmed that these assumptions are reasonable. However, it argued

that for D10 no publication proof has been submitted. Although it is true that the respondent has not submitted any evidence in this respect the Board likewise considers that the appellant did not submit any arguments, let alone convincing ones, as to why such a proceedings document of 1985 having 54 chapters and comprising a foreword of the Chairman of the Organizing Committee dated 20 September 1985 (see D10a, page 1 and pages i-viii) would not be in the public domain. The Board therefore has no doubt that the publication was made earlier than 1990, i.e. when the next Lead-Zinc International Symposium took place.

3.4 The appellant argued that D10, having been filed about one month before the date of the oral proceedings, was clearly late filed so that it should not be admitted since the case of a party should be presented complete from the beginning. Furthermore, the relevance of D10 would be doubtful and would not go beyond information already on file since physical washing is mentioned in Annex A1, D4 and D6.

> These arguments cannot be accepted by the Board. First of all, the appellant could not be surprised by the content of D10 which has its origin in its own company. Secondly, D10 is considered to be *prima facie* relevant for disclosing a leaching procedure in combination with a solvent extraction process for electrowinning zinc from zinc secondaries. This process includes neutralization cascades of the leaching solution (see page 768) and comprises a combination of two physical washing steps with acidified water and one chemical washing step with spent electrolyte of the organic extract (see pages 773 to 774). Hence D10 supports the

assumption of the Opposition Division in its decision that such a combination of physical and chemical washing belongs to the prior art (see reasons, page 6, point "V. Further arguments").

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- 3.5 Taking account of the points above the Board considers that D10 is relevant and was available to the public before the priority date of the patent in suit, i.e. before 17 December 1999. The documents D10 and D10a are therefore admitted into the proceedings.
- 4. Inventive step (Article 56 EPC)

#### Second auxiliary request

4.1 D1 is considered to represent the closest prior art for disclosing a process for the continuous production of high purity electrolytic zinc or zinc compounds starting from zinc concentrates to be leached by an aqueous acidic solution and subsequently to be extracted by an organic solvent. The process of D1 includes subjecting the zinc concentrate to a bioleach step to form a solution containing zinc, at least partially removing the zinc from the solution, and returning at least part of the zinc depleted solution to said bioleach step. Said leaching is carried out in multiple leaching zones arranged in a cascade mode and takes place under acidic conditions, preferably at a pH of about 0.5-2.5, preferably between 20-80°C with a residence time of 0.3-8 days (= 7.2-192 hours; see page 4, line 21 to page 6, line 20 and lines 27 to 29; page 12, lines 30 to 35; page 19, lines 22 to 35 and page 21, lines 25 to 33). The leach solution is subjected to an iron precipitation step by typically

neutralising to a pH of about 3-5 e.g. with lime, typically at a temperature of 20-90°C (see page 8, line 16 to page 9, line 1). The zinc is then removed from the leach solution by a zinc solvent extraction process to separate zinc from the remaining impurities (particularly Cu, Cd, Ca and Mg) and can produce a good quality zinc electrolyte suitable for producing special high grade (SHG 99.99% min) cathode zinc at high current efficiencies (see page 9, lines 21 to 28). Commercially available zinc extractants such as D2EHPA and IONQUEST 801 can be used at concentrations between 10-50% extractant in a suitable diluent such as Shellsol 2046. The solvent extraction step comprises one or more stages and the aqueous feed solution is acidic to prevent or minimise zinc loss; typically a pH range of about 2-5 is used (see page 9, lines 29 to 33). The remaining solution (raffinate) can be neutralised or a part thereof can be recycled to the bioleach step. The organic solution containing extracted zinc is purified by scrubbing (see page 10, lines 1 to 35) and then stripped of zinc by contacting it with spent electrolyte from a zinc electrowinning stage (see page 10, line 36 to page 11, line 5). The zinc depleted aqueous solution can be recycled to the bioleach step and its acidity may be adjusted by adjusting the degree of neutralisation after the solvent extraction step. If the solution is recycled to the bioleach step, it is necessary to periodically remove some of the impurities (e.g. Cd, Mg and Cu) to prevent build-up in the process liquor. Therefore a bleed stream is provided which will remove the impurities, e.g. by a zinc dust treatment (see page 11, lines 10 to 34). Ammonia may be used as an acid neutralisation agent in the solvent extraction stage (see page 12, lines 6 to 13).

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4.2 D1 aims to provide a method for separating zinc, particularly zinc metal, from zinc concentrates, and especially from lower grade zinc sulphide concentrates, in a manner which minimises input costs and unusable waste products and which is energy efficient when compared to the smelting and roast/leach techniques, and also does not produce toxic products such as SO<sub>2</sub> (see page 4, lines 21 to 30).

4.3 The subject-matter of claim 1 of the second auxiliary request differs from the process according to D1 in that

> i) the neutralization is conducted in zones arranged in cascade mode, and

ii) a purification treatment of the zinc loaded organic solvent, according to step f), is conducted in "q" physical and "r" chemical purification (successive) zones, organic and aqueous streams operating in counter current mode, the number "q" of such physical purification treatment zones being comprised between 1 and 4 and the number "r" of such chemical purification treatment zones being comprised between 1 and 4, wherein the physical treatment comprises the washing with an acidified water solution of the zinc loaded organic solvent from extraction in an organic/aqueous rate between 5 and 50, and wherein the chemical purification treatment is conducted by means of an aqueous zinc acid solution containing from 10 g/l to 100 g/l of zinc and from 0.1 g/l to 5 g/l of equivalent  $H^+$  (i.e. it comprises between 1 to 4 so-called "physical washing" zone(s) in combination with 1 to 4 of socalled "chemical washing" zone(s)).

4.3.1 Feature i) serves to remove as much iron and aluminium as possible from the leach liquor before feeding it to the solvent extraction system by precipitating their trivalent hydroxides with milk of lime (see D10, page 768, last paragraph to page 769, third paragraph; and Figure 2).

- 4.3.2 Feature ii) provides that the co-extracted and entrained impurities contained in the zinc loaded organic extraction solvent are fully eliminated in order to achieve an extremely high purity level of the zinc metal or zinc compound resulting from the process (see patent in suit, page 5, lines 44 to 48; and page 6, lines 12 and 13).
- 4.4 The problem to be solved by the features distinguishing the subject-matter of claim 1 of the second auxiliary request over the process of D1 is thus considered to be the provision of a process for the continuous production of electrolytic zinc or zinc compounds which allows to obtain the products with improved purity and in high yield starting from raw materials of different origins including primary and secondary raw materials (see patent in suit, paragraphs [0001] and [0015]).
- 4.5 This problem is solved by the process as defined in claim 1 of the second auxiliary request.
- 4.6 The Board, however, considers that the subject-matter of claim 1 of the second auxiliary request is rendered obvious for the following reasons:
- 4.6.1 It is usual in the technical field of hydrometallurgy to conduct neutralization in a cascade mode in order to

improve the purity of the leach liquor and to obtain good settling characteristics of the precipitates. In the case of zinc this neutralization step with at least two zones serves to remove trivalent cations such as Fe, Al, As, Sb, etc. from the leach liquor (see D10, page 773, chapter "Oxidation and precipitation"). In the first zone air is dispersed into the leach liquor to get rid of the excess of reducing agent present in the leach liquor, before oxidizing ferrous to ferric iron to be precipitated as hydroxide with milk of lime while the second zone serves to further oxidize the partly oxidized slurry from the previous reactor using chlorine gas as the oxidant to complete the oxidation reaction. The precipitate is then removed in a thickener and the overflowing liquor is collected in the pregnant liquor tank (see D10, page 768, last paragraph to page 769, third paragraph).

The appellant did not contest that feature i) represents common general knowledge. Thus feature i) of claim 1 of the second auxiliary request is considered to be obvious to the person skilled in the art.

4.6.2 First of all, it belongs to the common general knowledge of the person skilled in the art that physical entrainment of aqueous phase in the loaded organic phase is usually overcome by scrubbing said loaded solvent with water or some suitable solution (see the text book Annex A1, page 62, third paragraph; see also D1, page 10, lines 19 to 21, or see D10, page 773, penultimate paragraph to page 774, second paragraph). This - first - scrubbing represents a "physical washing" of the organic solvent and removes the impurities comprised in the entrained aqueous phase. Likewise it belongs to the common knowledge of the person skilled in the art that impurities comprised in the organic solvent after the extraction stage due to chemical co-extraction (e.g. other metals) can be removed by scrubbing with a solution either causing a selective stripping by using an acidic solution having the required pH for stripping the metal impurities while leaving the metal of interest in the organic solvent, or by scrubbing with a solution of a salt of the metal of interest to replace said co-extracted metals due to a metal exchange process (see Annex A1, page 62, fourth paragraph to page 63, fourth paragraph).

4.6.3 According to D1 the organic solution containing the extracted zinc is purified from any entrained aqueous solution and any co-extracted impurities in one or more scrubbing stages which may utilise either an acidified solution or zinc sulphate solution. D1 states that "a small bleed of electrolyte can be used to acidify the scrub solution, if desired and it is found that at a scrub organic: aqueous ratio of 10-40:1, almost all of the copper, cadmium, and most of the calcium can be scrubbed" (see page 10, lines 19 to 27). The final electrolyte according to an example in accordance with Figure 2 of D1 using a scrubbing stage comprising two contacting units 33 and 34 for counter-current scrubbing in mixer-settlers with a diluted zinc electrolyte from the electrowinning step 20 comprising 5 g/l Zn and 7.5 g/l  $H_2SO_4$  (the latter corresponding to 0.15 g/l equivalent  $H^+$ ) as the scrub solution with an organic: aqueous ratio of 20:1 - contained 10 ppm Ca and the levels of Cu, Cd, Al and Mg were below detection levels (see page 15, lines 16 to 20; page 16, line 14

to page 17, line 13; page 18, lines 9 to 18; page 23, lines 6 to 36; Figures 1 and 2).

Hence D1 teaches the person skilled in the art to remove any entrained aqueous solution and any coextracted impurities by scrubbing in order to purify the organic solvent resulting from the extraction step, preferably in at least two counter-current scrubbing stages in mixer-settlers using an acidified solution. Said acidified solution is preferably obtained by diluting electrolyte bleed from the electrowinning step.

4.6.4 D10 discloses that the zinc loaded organic extract is contacted in a three-stage washing operation in mixers settlers, counter-currently with an aqueous stream made up of spent acid and fresh water (see page 770, second paragraph) to remove the entrained aqueous solution and co-extracted metals therefrom. D10 teaches that there is physical washing to remove entrained impurities, and chemical washing to remove the co-extracted impurities and that "although those washing effects overlap each other the first two washing steps are mostly devoted to physical washing the organic extract with acidified water. The third one is the chemical washing stage, where a small stream of spent acid is used for this purpose" (see page 773, last paragraph to page 774, second paragraph). D10 additionally discloses that "in the chemical washing, some zinc is stripped from the organic extract by the acid content of the spent electrolyte, producing an almost neutral solution of zinc sulphate containing as much as 130 g Zn/L. Mass action law is playing its role, and practically all the coextracted impurities, such as Ca<sup>2+</sup>, are exchanged by  $\operatorname{Zn}^{2+}$ " (see page 774, third paragraph) but does not

disclose the composition of the chemical washing solution. D10 further mentions that "the ultra clean extract containing zinc after both washing operations is subjected to zinc stripping in two stages using the spent electrolyte as the stripping agent" (see page 774, fourth paragraph).

The electrolysis of the aqueous zinc sulphate containing ("advanced") electrolyte generates a sulphuric acid acidity in the thereby resulting spent electrolyte which is sufficient to strip the zinc from the organic solvent to increase the zinc concentration in the spent electrolyte by more than 30 g/L in this stripping step (see page 772, first to third paragraph; page 774, fourth paragraph and page 775, Figure 6). Consequently, it is evident that the appellant's arguments - that the spent electrolyte or the spent acid, which is used in the third scrubbing stage, would be an almost neutral solution - cannot be accepted. To the contrary it is obvious that the free acidity of the scrubbing solution is even too high since D10 states that "some zinc is stripped from the organic extract by the acid content of the spent electrolyte" in the chemical washing stage.

4.6.5 Taking account of his common general knowledge (see point 4.6.2 above) the skilled person, aiming to solve the technical problem as defined in point 4.4 above, would apply the teaching of D10 concerning the purification of the zinc loaded organic solvent with a combination of two physical washing stages with one chemical washing stage. In order to then obtain the best results of purification of the organic solvent the skilled person further has to determine the optimum acidity of the acidic solution to be used in the chemical washing stage, which according to D1 is obtained by diluting a small bleed of zinc electrolyte, i.e. an aqueous solution containing zinc sulphate and sulphuric acid. The determination of such an optimum concentration, however, belongs to the ordinary skills of the person skilled in the art. Thereby the person skilled in the art would arrive at feature ii) of claim 1 of the second auxiliary request.

Consequently, feature ii) of claim 1 of the second auxiliary request is obvious to the person skilled in the art, either.

- 4.6.6 The respondent's allegation that the hydrolysis of zinc sulphate in aqueous solution would result in an acidity falling into the range of from 0.1 to 5 g/L of equivalent H<sup>+</sup> was not supported by any evidence and thus cannot be accepted.
- 4.6.7 The appellant's arguments that the comparative tests as submitted with the grounds of appeal and according to Annex A as filed with letter dated 27 December 2007 (see grounds of appeal dated 31 March 2006, pages 7 and 8, Tables 1 and 2; and Annex A, Table 2.1) would prove an outstanding and surprising technical effect cannot be accepted for the following reasons.

The comparative experiments with respect to D1 and D5 according to Tables 1 and 2 were made by taking only the parameters concerning the scrubbing stage of these documents without considering the other parameters such as e.g. the feed raw material, the extractant concentration, etc. of these documents. These other parameters, however, have an influence on the impurities which are later to be found in the organic phase. Consequently, these parameters likewise influenced the type and the composition of the solution(s) to be used in the following scrubbing stages.

The other comparative tests according to Annex A only concern results of chemical washing solutions having from 10 to 100 g/L Zn and from 0.1 to 1 g/L of equivalent H<sup>+</sup> and of physical washing solution obtained by diluting the chemical solution to have a final composition with  $\leq 0.3$  equivalents/L, i.e. in accordance with claim 1 of the patent in suit. Table 2.1 of these experiments shows that the desired result of purification is only obtained when a consecutive physical and chemical washing is applied (one stage each) whereas in the case that only one of the two washing solutions is applied in a single stage the desired result is not obtained.

Annex A does not reveal any test results of solutions being slightly outside the claimed concentration ranges, e.g. 9 g/L Zn or 101 g/L Zn, let alone comparative tests with respect to the closest prior art D1 according to the established jurisprudence of the Boards of Appeal (see Case Law of the Boards of Appeal of the European patent Office, 5<sup>th</sup> edition, 2006, chapter I.D.9.8).

Such tests, however, were implicitly suggested by the Opposition Division by stating that "the ranges for the zinc and equivalent  $H^+$  concentration in the chemical purification treatment lie within the experienced

parameters gained by experience carried out by the man skilled in the art. Moreover, the claimed ranges do not bring any technical effect. The amounts given in D1 for the zinc and equivalent  $H^+$  concentration (see page 15, lines 16-20) are only specific examples and the skilled person would at least carry out experiments with amounts different from those disclosed in the example of D1" (see reasons of the decision, page 5, first paragraph). The Opposition Division further stated that "in D1 the purification treatment is carried out with an acidified solution which may be realized additionally to the scrubbing in one or more stages, meaning that the use of an acidified solution is an additional chemical measure beside the physical scrubbing stages (see page 10, lines 19-35 and page 15, lines 16-10)" (see reasons of the decision, page 6, fifth paragraph).

4.6.8 However, the appellant has neither submitted acceptable comparative tests with respect to D1 nor any evidence which would prove that an effect occurs only within the concentration ranges as defined in claim 1 of the second auxiliary request. The latter deficiency, however, had been mentioned by the Board in its annex to the summons to oral proceedings (see point III above). Additionally, the Board remarks in this context that the appellant - with respect to the main request which defined an acidity of at least 0.1 g/L of equivalent  $H^+$  - had argued that the implicit upper acidity limit would have been defined by the solubility of the zinc sulphate in the aqueous solution of sulphuric acid. Such a value, however, would have been well above the upper limit of 5 g/L of equivalent  $H^+$ according to claim 1 of the second auxiliary request.

4.6.9 The subject-matter of claim 1 of the second auxiliary request thus lacks an inventive step, and hence does not meet the requirements of Article 56 EPC. The second auxiliary request is therefore not allowable.

#### Third auxiliary request

4.7 Although claim 1 of the third auxiliary request is narrower in scope than claim 1 of the second auxiliary request it still comprises the embodiment of electrowinning the extracted Zn (compare point XIII, above). Thus the above conclusion with respect to claim 1 of the second auxiliary request applies a fortiori to claim 1 of the third auxiliary request.

> The Board therefore concludes that claim 1 of the third auxiliary request does not meet the requirements of Article 56 either. Consequently, the third auxiliary request is not allowable, either.

5. Request for re-opening the discussion of admissibility of the amendments in the auxiliary requests

> The second issue to be discussed at the oral proceedings was the allowability of the amendments and the Chairman of the Board stated when opening the discussion that the amendments made in **all** requests should be discussed (see point IX above).

> In a first round of discussion the appellant specified the amendments made in claim 1 of all requests but presented initially only its arguments with respect to the second and third auxiliary requests. It then

suggested reversing the order of the discussion of the requests. This proposal was not accepted by the Board so that it also presented its arguments with respect to the main and the first auxiliary requests (see point XV, above).

The respondent presented in said first round its arguments and objections under Articles 123(2) and (3) EPC with respect to the feature "at least 0.1 g/L of equivalent  $H^+$ " (see point XVI, above).

In the second round of discussion the appellant responded with respect to the alleged extension of the scope of protection of claim 1 of the main and the first auxiliary requests. In this second round the respondent, when asked by the Chairman, stated that it did not intend to add anything.

Thereafter the oral proceedings were interrupted by the Chairman for the internal deliberation of the Board and subsequently after resuming the oral proceedings the conclusions with respect to all requests were given to the parties.

The issue of admissibility of document D10 was next discussed with the parties and it was announced by the Board that D10 and D10a are admitted into the proceedings.

When it was announced that the next matter to be discussed was inventive step the respondent argued that it has been deprived of its right to be heard with respect to the amendments of the second and third auxiliary requests and particularly with respect to Article 123(3) EPC since the incorporated feature "0.1 to 5 g/L of equivalent  $H^+$ " would extend the scope of protection of claim 1 of the second and third auxiliary requests beyond that of claim 1 as granted. The respondent wanted this alleged deprivation of right to be incorporated into the minutes of the oral proceedings.

- 5.1 The Board then discussed this new issue with both parties.
- 5.1.1 The respondent argued that its understanding was that only the main request was to be discussed and to be decided by the Board. Therefore it requested that the debate of formal aspects concerning the auxiliary requests be reopened; alternatively it requested to take account in the minutes of this request (see written request dated 15 October 2008).
- 5.1.2 On questioning by the chairman the appellant confirmed that it had understood that the formal admissibility aspects, i.e. Articles 84, 123(2) and (3) and Rule 80 EPC, should be discussed for all requests together. It further stated that although the basis and justification of the amendments made to claim 1 of the second and third auxiliary requests were presented first, those for claim 1 of the main and first auxiliary requests were also given.
- 5.2 From the above it can be concluded that the respondent either misunderstood the statements of the Chairman or that it escaped the respondent's attention that it like the appellant - should address all requests. Since the appellant had addressed all four requests it was

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the Board's understanding that the respondent did not intend to add anything to its objection concerning the feature of the main and first auxiliary requests (compare in this context points 2.3 and 2.4 above). The Board saw no reason to question the respondent specifically about the second and third auxiliary requests since the Board had no objection to these. Furthermore, as admitted by the respondent, the arguments concerning the feature "at least 0.1 g/L of equivalent H<sup>+</sup>" of claim 1 of the main request likewise applied to claim 1 of the first auxiliary request. Thus the respondent actually presented arguments at least with respect to the first auxiliary request contrary to the statement on its written requests that "the opponent has not been heard with regard to the question of admissibility of auxiliary requests 1-3" (see point IX above).

5.3 If the intention of the Board concerning the procedure was unclear to the respondent then it should have asked for clarification of the matter since no unclarity was apparent to the Board or the appellant. Similarly, if the intended procedure had escaped the respondent's attention this was likewise not apparent to the Board. In both cases the respondent had the opportunity to present its arguments but did not do so.

> Consequently, contrary to the allegations made, the respondent's right to be heard under Article 113(1) EPC has not been violated. The respondent's request to reopen the discussion for the admissibility of the amendments to the claims of the auxiliary requests was therefore refused.

#### 6. Request for recording a matter in the minutes

6.1 As an auxiliary request with respect to re-opening the discussion (see point 5 above) the respondent requested the recording in the minutes that "the opponent has not been heard with regard to the questions of admissibility of auxiliary requests 1-3 concerning amendments made therein" (see point IX above).

However, according to the jurisprudence of the boards of Appeal (see T 928/98 of 8 November 2000, points 5 to 5.5, not published in OJ EPO; T 263/05, OJ EPO 2008, 329, points 8 to 8.11; and T 550/04 of 2 April 2008, points 5 to 5.3, not published in OJ EPO) it is not the function of the minutes to record statements which a party considers to be possibly relevant in any subsequent proceedings. The desired statement does not relate to the surrender or abandonment of subjectmatter of the patent and does not otherwise have an impact on the definition of the subject-matter of the patent for the purposes of the questions the Board had to decide in these proceedings.

- 6.2 The desired statement was thus not a proper subject for the minutes. Consequently, the request was refused.
- 7. Request for adjournment of the oral proceedings
- 7.1 The appellant requested the adjournment of the oral proceedings in order to carry out comparative tests with respect to the process of the late filed document D10. These comparative tests should prove that the process of D10 results in a different purity of the

zinc cathode than that obtained according to the patent in suit.

The respondent objected to this request for being filed at a very late stage of the proceedings and requested that is should be refused. It also questioned the relevance of such tests since the use of an acidic solution in the physical washing step(s) and the use of an acidic solution having a certain zinc content in the chemical washing step was obvious to the skilled person.

- 7.2 With respect to inventive step the appellant argued that the chemical washing described in D10 is not a chemical washing with an acidic solution as required by claim 1 of the second auxiliary request since a neutral zinc sulphate solution would be used. It argued that it is not known which exact conditions are used for obtaining the final zinc quality of 99.95% Zn according to D10 since no details are given. Particularly the impurity levels such as those of Cu and Cd of the electrolyte according to Table 3 do not allow obtaining zinc cathodes having purities of 99.9959% Zn or 99.9971% Zn as specified in Table 4 according to test run no. 6. It further argued that in this respect there is no enabling teaching in D10.
- 7.3 Taking account of the fact that the opposition Division had argued that it was obvious to have consecutive physical and chemical washing stages and that it was obvious to experiment (see decision, reasons, page 6, point "V. Further arguments") comparative tests should have been made at the beginning of the appeal proceedings with such a combination of physical and

chemical washing stages in view of the closest prior art D1.

Considering that D10 does not disclose any concrete concentration values of the washing solutions and further taking account of the appellant's arguments concerning a non-enabling teaching for a specific purity in D10 - which view is not shared by the Board it is not apparent as to how such comparative tests with respect to D10 should be made at all, let alone credible ones.

7.4 Therefore the request for adjournment of the oral proceedings to carry out comparative tests with respect to D10 was refused.

## Order

## For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

G. Nachtigall

P. O'Reilly