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Datasheet for the decision of 11 November 2015

Case Number: T 0764/14 - 3.3.06

Application Number: 03718116.1

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Language of the proceedings: ΕN

Title of invention:

ETHYLENE OXIDE CATALYST

Patent Proprietor:

SD Lizenzverwertungsgesellschaft mbH & Co. KG

Opponent:

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

Headword:

Silver catalyst / SD

Relevant legal provisions:

EPC Art. 83

Keyword:

Sufficiency of disclosure - (yes)

Decisions cited:

G 0003/14, T 0608/07

Catchword:



Beschwerdekammern **Boards of Appeal**

Chambres de recours

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Case Number: T 0764/14 - 3.3.06

DECISION of Technical Board of Appeal 3.3.06 of 11 November 2015

SD Lizenzverwertungsgesellschaft mbH & Co. KG Appellant:

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SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. Respondent:

Carel Van Bylandtlaan 30 (Opponent) NL-2596 HR The Hague (NL)

Decision of the Opposition Division of the Decision under appeal:

European Patent Office posted on 24 January 2014 revoking European patent No. 1492619 pursuant to

Article 101(3)(b) EPC.

Composition of the Board:

Chairman B. Czech P. Ammendola Members:

S. Fernández de Córdoba

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Summary of Facts and Submissions

- I. This appeal is against the decision of the Opposition Division revoking European patent No. 1 492 619.
- II. The granted patent had been opposed on the grounds of added subject-matter (Article 100(c) EPC), insufficient disclosure (Article 100(b) EPC), lack of novelty and lack of inventive step (Article 100(a) EPC).
- III. During the opposition proceedings the Patent Proprietor had filed three sets of amended claims labelled Main Request and Auxiliary Requests I and II.
- IV. Claim 1 according to said Main Request reads:
 - "1. A process for the preparation of a catalyst for the production of ethylene oxide comprised of silver supported on an alumina carrier containing both sodium and silicate ions on the surface thereof, comprising
 - (i) a carrier pretreatment with an aqueous lithium salt solution at a temperature lower than 100 °C, wherein the weight ratio of removed Si/Na is less than 5.0, and wherein the treated carrier is dried prior to the impregnation with the silver solution and wherein the treated carrier is washed with water before or after said drying step, wherein the treatment removes at least 25 % of the sodium ions on the surface of the carrier and partially replaces the removed sodium with lithium to yield a surface with up to 10 ppm lithium ions, and
 - (ii) impregnating the pre-treated carrier."

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- V. More particularly, the Opponent's objections under Article 100(b) EPC focused on the features of the pretreatment step "(i)". The Opponent had essentially argued that it was not possible for a skilled person to establish whether such a pretreatment step complied with the following three requirements (hereinafter requirements (A), (B) and (C), respectively) imposed by claim 1:
 - (A): "[A]t least 25% of the sodium ions on the surface of the carrier" had to be removed. This requirement also necessarily implied the previous knowledge or the experimental determination of the baseline to which this percentage referred, i.e. the initial amount of Na ions on the surface of the untreated carrier.
 - (B): The pretreatment "partially replaces the removed sodium with lithium to yield yield a surface with up to 10 ppm lithium ions".
 - (C): "[T]he weight ratio of removed Si/Na" had to be "5.0 or less".

The following abbreviations will also be used below:

- the "sodium ions on the surface of the carrier" are indicated as surface Na;
- their relative amount on the untreated carrier as surface Na baseline;
- the surface Na removed from the carrier during step "(i)" are indicated as surface Na removed and their relative amount expressed as percent of the surface Na baseline is indicated as % of surface Na removed; and
- the "lithium ions" on the surface of the pretreated carrier are indicated as **surface Li**.

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VI. In the appealed decision, the Opposition Division held (Reasons, 2.4.3.1) that the compliance with requirement (C) could be verified since its literal meaning was that of the weight ratio between the Si and Na ions that were found in the "pretreatment solutions". This expression "pretreatment solutions" was used by the Opposition Division, and is also used herein below, to designate all the liquid phases collected at the end of the step "(i)", i.e. the totality of the Li salt solution(s) and washing water(s) used in the pretreatment.

However, the Opposition Division held (Reasons, 2.4.1.12 and 2.4.2.8) that the patent in suit lacked a clear disclosure of how to determine the relative amounts of surface Na removed and of the resulting surface Li, and that the skilled person was thus unable to carry out the process at the borderlines of claim 1 of the Main Request when:

- "intending to remove close to 25% of the sodium ions", i.e. at the borderlines of requirement (A), and
- "intending to yield a surface with up to 10 ppm lithium ions", i.e. at the borderlines of requirement (B).

Since the same objections also applied to both Auxiliary Requests, the Opposition Division concluded that all pending requests of the Patent Proprietor were objectionable under Article 83/100(b) EPC.

VII. With its statement of grounds, the Appellant (Patent Proprietor) (re-)filed, as its Main Request and Auxiliary Requests I and II, three sets of claims identical to those already considered in the decision under appeal. In its statement, the Appellant contested the findings of the Opposition Division regarding

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sufficiency and requested that the case be remitted to the Department of First Instance for further prosecution, including examination of novelty and inventive step.

- VIII. In its reply, the Respondent (Opponent) maintained objections regarding sufficiency (all requests).

 Referring also to a further prior art document, it argued inter alia that the analytical technique X-ray Photoelectron Spectroscopy (XPS hereinafter) was not suitable to determine accurately, without undue burden, the amounts of Na, Li and Si present on, deposited on and/or removed from the carrier surface.

 It also requested the remittal of the case for continuation of the opposition proceedings in relation to novelty and inventive step, in case the Board were to set aside the revocation decision of the Opposition Division.
- IX. The Parties were summoned to oral proceedings. In a communication issued in preparation for the oral proceedings, the Board indicated *inter alia* that it intended to remit the case if it were to conclude that the patent as amended according to one of the pending requests complied with Article 83 EPC.
- X. With further written submissions, both parties filed further items of evidence, in support of their respective arguments regarding analytical methods, including XPS, that may be used for analysing the surface of the carrier.
- XI. Oral proceedings took place as scheduled in the presence of both Parties. The issue of sufficiency of the disclosure was extensively debated.

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XII. The Appellant **requested** that the decision under appeal be set aside and that the patent be maintained in amended form on the basis of the claims according to one of the Main Request or the Auxiliary requests I or II, all filed with the statement of grounds of appeal.

The Respondent requested that the appeal be dismissed.

XIII. The submissions of the Appellant of relevance here, i.e. regarding the sufficiency objections raised with respect to the Main Request, may be summarized as follows.

The decision under appeal was erroneous because the skilled person had no difficulty in carrying out many embodiments of the process of claim 1 of the Main Request. Contrary to the Opposition Division's conclusions, the relevant disclosure in the patent in suit regarding requirements (A) and (B) to be met in the carrier pretreatment step "(i)" were sufficient to permit carrying out the process, at least in view of common general knowledge. The finding that the skilled person would be unable to carry out the claimed process at the borderlines of claim 1 pertained to the requirement of clarity (Article 84 EPC) and was irrelevant as regards sufficiency.

General considerations

The mild pretreatment of the carrier according to step "(i)" of claim 1, i.e. using an aqueous Li salt solution at low temperature,

- provided the carrier surface with certain low-alkali characteristics (requirements (A) and (B) being met, see also paragraphs [0024] and [0025]) that were beneficial for the subsequent deposition of the silver catalyst, - while ensuring that only a limited amount of silicate

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ions was removed from the carrier surface (requirement (C) being met, see also paragraphs [0029] and [0047]) so as to preserve the particular structure of the carrier surface and, thus, to retain the carrier strength also in the final catalyst.

Hence, it was also evident that the requirements (A) to (C) had to be complied with just before starting the deposition of the silver catalyst in step "(ii)".

Accordingly, the requirements concerning the percentage of surface Na removed had to be complied with at the end of step "(i)", i.e. after the washing(s) with water.

As to requirement (A)

The patent in suit explicitly defined the surface Na as the Na ions that could be rapidly removed during the "Acid-Leachable test" described in paragraph [0031]. In this test the untreated carrier was digested for a "short period of time" with a concentrated nitric acid solution.

Such nitric acid solution was, concededly, so aggressive that it progressively dissolved the carrier (alumina) and, thus, progressively also dissolved Na ions initially present in the subsurface of the carrier (hereinafter subsurface Na).

The argument of the Respondent that a skilled person would expect substantial migration of relevant alkali ions (Na or Li) from the subsurface to the surface of the carrier (or vice versa) to occur during the "Acid-Leachable test", was merely an unsupported allegation and, therefore, not to be accepted. A possible migration of subsurface Na was mentioned in paragraph [0048] of the patent in suit only with reference to the drying of the (pretreated) carrier at high temperatures. Hence,

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there was no reason to also expect such a migration to occur upon contacting the carrier with an acid solution at much lower temperatures for a "short period of time".

On the contrary, any release of subsurface Na into the nitric acid solution used in the "Acid-Leachable test" could only occur after disruption of the carrier surface and was, thus, manifestly slower than the dissolution of the surface Na. Therefore, the "short period of time" mentioned in paragraph [0031] was the time necessary for dissolving, and thus permitting measuring, (almost) exclusively those Na ions that were exposed on the surface and, thus, dissolving very rapidly, before the occurrence of any substantial disruption of the carrier surface, i.e. before the subsurface Na might contribute substantially to the amount of Na dissolved in the nitric acid solution.

For the skilled person, it was thus self-evident that the test described in paragraph [0031] also required (implicitly) some monitoring of the speed at which the concentration of Na ions in the acid solution increased. The skilled person would expect that, at a sufficiently low temperature, this monitoring allowed to detect a manifest drop of the speed of Na dissolution, corresponding to the point at which substantially all "fast dissolving" surface Na had been removed and only "slow dissolving" subsurface Na gradually passed into the nitric acid solution. Hence, the amount of Na present in the nitric acid solution at the moment of that drop of the Na dissolution speed, was the surface Na baseline.

The Appellant also maintained that only a very limited dissolution of subsurface Na could possibly occur during such "short period of time", resulting only in a very

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limited "error" in the measured surface Na baseline value. In particular, such "error" could only render this latter value slightly higher than its actual value.

The relative amount of surface Na removed was defined in paragraph [0032] of the patent in suit. Considered in the context of the patent as a whole, this paragraph implied that such amount corresponded to the overall amount of Na ions found in the totality of the pretreatment solutions.

Concededly, some minor amounts of subsurface Na ions might also be dissolved during the pretreatment "(i)" (i.e. by the Li salt solution(s) and washing water(s)), e.g. due to some local excessive disruption of the carrier's structure, but not because of spontaneous migration of subsurface Na to the surface, as alleged by the Respondent.

As explicitly stated in the patent in suit (see in particular paragraphs [0027] to [0029] and [0047]), requirement (C) imposed that the conditions of the pretreatment step "(i)", and in particular of the low temperature of the treatment with the Li salt solution, be set such that excessive dissolution of the silicate ions present on the surface of the carrier was avoided and, thus, the integrity of the carrier's surface was preserved. Therefore, only a very limited amount of subsurface Na was possibly exposed to and dissolved in the pretreatment solutions.

In addition, it was reasonable to expect that also during the treatment with the Li salt solution(s) any dissolution of subsurface Na was anyway much slower than that of the surface Na. Hence, the amount of subsurface Na dissolved during the minimum duration of the

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pretreatment required for removing the minimum amount of 25% of surface Na could be predicted to be particularly low. Hence, all pretreatment steps "(i)" lasting substantially longer than such minimum duration would necessarily also remove more than 25% of the actual surface Na, and would thus necessarily comply with requirement (A).

The very limited "errors" possibly resulting from the dissolution of some subsurface Na and affecting the measured percentage of surface Na removed, would at most cause a lack of clarity as to whether or not processes in which the amount of Na found in the pretreatment solutions was exactly 25%, or just above 25%, of the baseline fell within the ambit of claim 1. However, any such uncertainty at the boundaries of the claimed process did not amount to an insufficiency.

It was also conceded that the wording in the initial part of the claim appeared to require (possibly unintentionally, but literally) the presence of surface Na in the final product of the claimed process. However, such requirement was inevitably always met, because it was impossible to arrive at a pretreated carrier not containing any detectable surface Na at all, even when the conditions of the pretreatment step "(i)" were set with the intention to remove 100% of the surface Na baseline.

As to requirement (B)

After having initially maintained that the surface Li could be measured by the skilled artisan by using XPS, the Appellant ultimately argued that the skilled person reading the patent in suit would in any case also understand that all the Li comprised in the carrier at

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the end of the pretreatment step "(i)" had to be present on the surface of the carrier, i.e. was essentially all surface Li. Indeed, the claimed process was clearly a surface treatment, i.e. a treatment avoiding any substantial disruption of the particular carrier structure used (alumina containing silica).

The relative amount of Li in ppm was thus directly determinable by chemical analysis of the (entire) pretreated carrier, or from the difference between the starting amount of Li in the used Li salt solution(s) and the overall amount of Li ions found in the totality of pretreatment solutions at the end of step "(i)".

Moreover, since this pretreatment was a mild procedure at low temperature, there was no evidence or theoretical reasoning rendering plausible that, as alleged by the Respondent, Li might also migrate into the subsurface of the carrier. In any case, any such hypothetical migration could only affect the measured amount of surface Li to a very limited extent. Thus, it could at most generate some uncertainty at the boundaries of the claim (i.e. when aiming to provide a carrier with about 10 ppm of surface Li). Such uncertainty did not, however, amount to insufficiency of the disclosure either.

The further requirement in step "(i)" according to which Li only "partially replaces the removed sodium" (emphasis added), could only mean that the amount of surface Li (i.e. all the Li that could be found in the carrier) had to be less than the amount of surface Na removed (i.e. all the Na that could be found in the pretreatment solutions). Any "errors" possibly due to the dissolution of some subsurface Na and/or to some (hypothetical) migration of Li into the carrier

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subsurface, could at most raise an issue of clarity for those embodiments of the claimed process in which the measured amount of Na actually removed during the pretreatment and the measured amount of Li remaining on the pretreated carrier were found to be very close to each other. In addition, as apparent from Table 1, many commercial carriers had a surface Na baseline of more than 40 ppm. For all these starting materials the claimed process (requiring removal of at least 25% of the surface Na) mandatorily required that surface Na was removed in an amount corresponding to more than 10 ppm (at least 25% of at least 40 ppm). Hence, given that 10 ppm was the maximum amount allowed for the surface Li, when using such a commercial carrier a "partial" replacement of surface Na by surface Li was merely the inevitable consequence of meeting requirements (A) and (B).

Finally, already upon reading paragraphs [0036], [0039], [0090] and, in particular, [0107] of the patent, the person skilled in the art was taught that washing with water allowed to reduce the amount of Li remaining on the carrier to the desired relative amount of "up to 10 ppm". The same was confirmed by the comparison between the two examples of Table 11, which were comparable since their preparation was very similar. Hence, the patent in suit clearly taught that the required low level of Li on the carrier could be achieved by (repeated or prolonged) washing.

As to requirement (C)

Concededly, the patent in suit consistently referred to the Si atoms and Na ions removed during the pretreatment step "(i)" by indicating explicitly, or implying, that they were removed from the surface of the carrier, i.e.

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that prior to their removal from the carrier they were, respectively, Si atoms initially present in silicate ions on the carrier's surface (surface Si hereinbelow) and surface Na. However, in the patent in suit (e.g. paragraphs [0032], [0074], [0080] and [0096] in combination) the amounts of surface Si and surface Na removed were clearly equated to the amounts of Si and Na that could be found in the pretreatment solutions. Since in the patent in suit requirement (C) was explicitly stated to correspond to those conditions of the pretreatment step "(i)", and in particular the low temperature of the treatment with Li salt solution, under which the structure of the carrier surface was preserved, it was apparent that only minor amounts of dissolved subsurface Na or subsurface Si were to be expected in the pretreatment solutions. Again, the small "errors" resulting therefrom would not make the patent insufficient. There was at most some uncertainty (lack of clarity) as to whether or not processes in which the removed Si/Na ratio was measured to be about 5 fell within the boundaries of claim 1.

As to the undue burden allegedly implied by requirements (A) to (C) having to be met simultaneously

This allegation of the Respondent was unfounded. The patent in suit disclosed several examples of suitable carriers, suitable Li salt solutions and conditions under which the pretreatment step may be performed, such as temperatures, duration of the treatments with the Li salt solution(s) as well as abundant information regarding the washing(s). In this connection, reference was made to, inter alia, paragraphs [0031] to [0039], [0051], [0052], [0074], [0079] and [0080]. In addition, the importance of monitoring the Na, Li and Si concentrations in the pretreatment solutions was

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apparent from the whole disclosure of the patent. Hence, the patent in suit contained sufficient information enabling the skilled person to carry out many alternative embodiments of the claimed process and required at most few routine experiments possibly necessary for identifying the appropriate temperatures and durations for the "Acid-Leachable test".

XIV. The submissions of the Respondent of relevance here may be summarized as follows.

As to requirement (A)

After having initially maintained that claim 1 of the Main Request omitted to specify whether or not the amount of surface Na removed was to be measured taking into account also the Na ions removed during the washing step(s), the Respondent ultimately no longer disputed at the oral proceedings that requirements (A) to (C) had to be complied with at the end of step "(i)", i.e. that any Na possibly removed during the washing(s) had also to be taken into account when determining the amount of surface Na removed.

Requirement (A) was, however, totally unclear because the disclosure in patent in suit comprised no precise definition of the "surface" of the carrier (e.g. in terms a specific number of atomic layers). Moreover, contrary to a statement in paragraph [0031] of the patent in suit, there was no standard or conventional procedure for measuring the surface Na baseline of carriers. Additionally, the producers of commercial alumina carriers did not normally provide any information regarding the surface Na contents of these carriers.

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Hence, the skilled reader of the patent in suit could only attempt to determine the surface Na baseline based on the disclosure of the "Acid-Leachable test" mentioned in paragraph [0031] and also referred to in Table 1 (providing the surface Na baseline of several carriers). However, such disclosure was manifestly incomplete because it indicated neither the temperature nor the length of the "short period of time" for the digestion with the nitric acid solution. This missing information was, however, important because the concentrated nitric acid solution used was also able to dissolve the whole carrier and, thus, also to dissolve rapidly large amounts of subsurface Na. Moreover, as evident from paragraph [0048], Na ions could also migrate from the subsurface to the surface of the carrier. Thus, simultaneous dissolution of subsurface Na occurred inevitably right from the start of the digestion and proceeded to a possibly very large extent, depending on the (arbitrarily) chosen digestion temperature and length of the "short period of time". This deprived of any meaning the measure of the surface Na baseline according to the incompletely disclosed "Acid-Leachable test".

The Respondent initially disputed the Appellant's allegation that it would be possible to identify the moment in time at which all the surface Na is removed by monitoring the speed at which the Na concentration in the acid solution increases, and by measuring the amount of Na that is more rapidly dissolved by the nitric acid solution. It argued that surface Na dissolved instantaneously at the very beginning of the "Acid-Leachable test". Thus, there was no detectable "short period of time" characterised by a relatively faster speed of Na dissolution. However, at the oral proceedings, the Respondent ultimately did not dispute the Appellant's argument that the skilled person

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confronted with such an "instantaneous" dissolution, would routinely attempt to lower the temperature and to monitor the Na concentration in the acid solution at shorter intervals, in order to render clearly detectable the end of the fast dissolution of surface Na.

The dissolution of subsurface Na also occurred to a large extent during the pretreatment step, because of the migration of Na ions from the subsurface to the surface of the carrier and because the pretreatment was disrupting to some extent the carrier structure. Hence, subsurface Na also substantially contributed to the amount of Na to be found in the pretreatment solutions and, thus, affected with an "error" the measure of the amount of surface Na removed according to the teaching in paragraph [0032] as well.

Hence, the surface Na baseline (allegedly measurable using the insufficiently disclosed "Acid-Leachable test" of [0031]) and the amount of surface Na removed (measurable as possibly implied in [0032]) were not exclusively expressing the amount of Na ions initially present on or removed from the <u>surface</u> of the carrier, respectively. Thus, their comparison did not permit to determine with accuracy the % of surface Na removed, i.e to verify the compliance with requirement (A).

Finally, the wording of the preamble of claim 1 also required surface Na (and silicate ions) to be present in the final product, but the patent in suit provided no guidance on how to conduct the process so that between 25% and less than 100% of the surface Na is removed.

As to requirement (B)

This requirement clearly referred exclusively to the Li

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present on the surface of the carrier. However, the patent in suit gave no guidance on how to determine and quantify the amount of such surface Li. Considering that the patent itself also mentioned in Table 9 "atomic %" of surface ions measured by XPS, the Appellant's attempt to present as self-evident that the relevant amount of surface Li corresponded to the total amount of Li that could be found by chemical analysis of the composition of the treated carrier, or that was missing from the Li salt solution(s) and not recovered during the washing(s), was unjustified. XPS was, moreover, not suitable for determining very low Li concentrations on the surface of carriers as used according to the patent.

Moreover, Li ions that could be found in the pretreated carrier at the end of step "(i)" could also be present in the carrier matrix, i.e. as subsurface Li, due to migration from the surface to the subsurface.

Finally, there was no teaching in the patent in suit on how to target a final Li content of "up to 10 ppm". The comparison between samples 16 and 15 in Table 11 did not suggest to repeat or prolong washing, because these two samples were based on different carriers and, thus, not directly comparable.

As to requirement (C)

The process according to the claim at issue implied keeping below a specific value the weight ratio between the Si and Na ions removed <u>from the surface</u>, because the claim had to be read in the light of the specifications (see first sentence in paragraph [0026], third sentence in [0027], second sentence in [0085] and first sentence in [0101]).

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Again, the method possibly suggested in the patent in suit to determine the content of Na and Si present in the pretreatment solutions, did not permit to distinguish between ions stemming from the subsurface and ions stemming from the surface of the carrier.

Hence, the patent did not sufficiently disclose how requirement (C) was to be met. Carrying out an embodiment of the claimed process such that it certainly complied with this requirement imposed an undue burden of experimental work, and/or of theoretical speculations as to the extent of "errors" arising from the dissolution of subsurface Na and Si.

The undue burden required for carrying out a process simultaneously complying with requirements (A) to (C)

None of the methods described in the examples of the patent in suit expressly complied with all the requirements A to C. Hence, reproducing the invention had to be based on a number of theoretical considerations and required an experimental effort to find the appropriate setting of interdependent variables (such as the temperature and the duration of the treatment with the Li salt solution(s), or the number of washing steps). Thus, identifying appropriate pretreatment conditions for a given carrier required to preliminarily carry out a research program to understand how these variables influenced - and to which extent the desired final compositional requirements of the pretreated carrier. Hence, the incomplete and/or obscure disclosure of the patent in suit imposed an undue burden on the person skilled in the art seeking to reproduce the claimed invention.

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Reasons for the Decision

Main Request - Sufficiency

- 1. Claim 1 (wording under IV, supra) is directed to a process for the preparation of a silver-containing catalyst for the production of ethylene oxide, starting from an alumina carrier comprising Na and silicate ions on its surface. In particular, the wording of claim 1 per se expresses that:
 - in step "(i)" of this process the carrier is pretreated with aqueous Li salt solution at a temperature lower than 100°C, washed with water and dried;
 - this pretreatment removes Na ions from the carrier surface and partially replaces them with Li ions, while also removing silicate from the carrier, and
 - in step "(ii)" silver is deposited on the surface of the pretreated carrier by impregnation.
- 2. The Board holds that the skilled person reading claim 1 in the context of the whole patent (see e.g. paragraphs [0024] to [0029] and [0033] to [0048]) understands that requirements (A) to (C) of the pretreatment step identified above determine compositional characteristics to be imparted to the carrier before subjecting it to the subsequent impregnating step "(ii)". In other words, requirements (A) to (C) have to be met at the end of step "(i)" and, thus, after the washing(s) with water. This was ultimately no longer disputed by the Respondent at the oral proceedings.
- 3. Requirement (A) ("the treatment removes at least 25 % of the sodium ions on the surface of the carrier")
- 3.1 The initial determination of the surface Na baseline

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The determination of the % of surface Na removed from the carrier during the pretreatment step "(i)" requires knowing the surface Na baseline, i.e. the amount of surface Na ions initially present in the untreated carrier.

3.1.1 The Board notes that the following statement in the patent in suit (paragraph [0031]) provides indications regarding the way in which the surface Na baseline is determined:

"The concentration of the surface Na ions of the untreated carrier is determined by the carrier manufacturer using the 'Acid-Leachable test'. In the standardised acid leachable test, the carrier sample is digested for a short period of time in 30% nitric acid solution. The sodium, potassium, calcium and silicon concentrations in the resulting solution are determined by atomic absorption spectro-photometry ..." (emphasis added).

Numerical values for the sodium baseline of several "low-sodium carriers made essentially of alpha-alumina" are reported in Table 1 under the heading "Surface analysis (ppm): Acid-Leachable test".

- 3.1.2 The Board also takes into account that at the oral proceedings the Appellant ultimately no longer disputed the Respondent's arguments that manufacturers of alumina carriers did not usually provide an indication of surface Na content thereof and that there was no "standardised" set of conditions for carrying out the "Acid-Leachable test".
- 3.1.3 Hence, for the Board, the above-cited paragraph [0031] indeed represents an (albeit somewhat vague) definition

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of the surface Na baseline provided to the skilled person reading the patent in suit. For the Board, the key question as regards the determinability of the Na baseline value of a given carrier is thus whether or not the skilled person, taking into account relevant common general knowledge, is able to identify the appropriate temperature and the length of the "short period of time" applicable in the digestion with 30% nitric acid solution.

- 3.1.4 It is plausible, and also common ground between the Parties, that such nitric acid solution is, depending on the duration of the treatment and the temperature applied, able to progressively disrupt the whole carrier matrix and, thus, to also gradually dissolve subsurface Na, but that surface Na is necessarily leached-out at much faster rate than subsurface Na.
- 3.1.5 The Respondent alleged that during such "Acid Leachable test", as well as in the actual pretreatment step "(i)", the liquid phase will also extract from the carrier substantial amounts of subsurface species without disrupting the carrier matrix, e.g. because ions may migrate from the subsurface to the surface.

This allegation was, however, not corroborated by suitable evidence and disputed by the Appellant. Therefore the Board rejects it as unconvincing. Indeed, paragraph [0048] of the patent in suit, referred to by the Respondent, merely teaches that Na ions may migrate from the subsurface to the surface of the carrier at very high drying temperatures of up to 1000°C (which should, therefore, be avoided when drying the pretreated carrier). For the Board, this indication does not imply that such a migration of subsurface Na ions would also

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occur during the treatment with a nitric acid solution at much lower temperatures.

3.1.6 Based on the considerations under 3.1.4, supra, the Board is thus convinced that the skilled person reading paragraph [0031] understands that the "surface Na ions of the untreated carrier" mentioned in that paragraph are those Na ions that dissolve very rapidly into the nitric acid solution, i.e. during said "short period of time". On the contrary, the subsurface Na ions can be expected to dissolve more slowly, because their exposure to the nitric acid solution requires the previous disruption of at least part of the carrier surrounding them.

Thus, the Board considers plausible that when all the fast-dissolving surface Na has passed into the nitric acid solution, the rate at which further Na ions are leached from the carrier must inevitably drop. Hence, the Bord is convinced that the skilled person reading paragraph [0031] would immediately understand

- that the test described therein requires monitoring the Na dissolution rate, i.e. the rate at which the Na ions are leached-out from the carrier into the nitric acid solution;
- that the end of the "short period of time" mentioned in that paragraph corresponds to the moment at which such dissolution rate shows a significant drop and, thus,
- that the surface Na baseline value is determinable (possibly with some slight uncertainty) as the amount of Na ions that can be found in the nitric acid solution at that moment.
- 3.1.7 The Respondent initially stated at the oral proceedings, without providing any supporting evidence, that the

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dissolution of the surface Na in the nitric acid solution would occur occur very rapidly, i.e.

"instantaneously". Thus, there would be no detectable drop of the rate at which Na ions pass into the nitric acid solution and, therefore, no possibility to identify an end point of the "short period of time". However, the Respondent ultimately no longer disputed that, as convincingly argued by the Appellant, the skilled person possibly confronted with an "instantaneous" dissolution of the surface Na, would routinely consider lowering the temperature of the digestion step as well monitoring the Na concentration in the acid solution at shorter intervals, so as to render detectable said drop in the Na dissolution rate.

- 3.1.8 Hence, the Board concludes that the skilled person is able, based on common general knowledge and corresponding routine variation of experimental conditions, to complement the information contained in paragraph [0031] of the patent in suit and, thus, to determine (possibly with some slight uncertainty but) without undue burden the surface Na baseline value for a given carrier.
- 3.2 Checking compliance with requirement (A)
- 3.2.1 The next question is how the skilled person, knowing the so-determined value of the surface Na baseline of the given carrier, can ascertain whether or not a certain setting of the conditions applied in the pretreatment step "(i)" of the claimed process meets requirement (A), i.e. whether or not at least 25% of the surface Na baseline are removed during step "(i)".
- 3.2.2 The Board notes in this respect that it is indicated in paragraph [0032] of the patent in suit that the amount

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of surface Na removed is "measured through analysis of the solution used in the pretreatment". It is undisputed (see 2, supra) and apparent from the rest of the patent description (see in particular paragraphs [0038] and [0039]) that the expression "solution used in the pretreatment" in this passage must reasonably be understood as referring to the totality of the Li salt solution(s) as well as the water phase(s) used for washing.

Hence, the Board concludes that the patent in suit implicitly defines the amount of surface Na removed as the overall amount of Na that can be found in all the pretreatment solutions. It is undisputed that this latter amount can be measured by conventional analytical methods.

- 3.3 The Respondent nevertheless argued that even if it were possible to determine a Na baseline by means of the "Acid-Leachable test", as well as the amount of Na present in the pretreatment solutions, these two measured values would not correspond to the initial amount of surface Na and to the amount of surface Na removed. Instead, the very aggressive nature of the nitric acid solution implied that relatively large amounts of dissolved subsurface Na contributed substantially to the measured surface Na baseline. In addition, also the overall amount of Na that can be found in the pretreatment solutions possibly comprised large amounts of subsurface Na dissolved, because of migration phenomena and of some unavoidable disruption of the carrier structure.
- 3.3.1 The Board accepts as plausible, and this was ultimately also no longer disputed by the Appellant at the oral proceedings,

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- i) that the measured surface Na baseline may encompass a detectable amount of subsurface Na, e.g. because some disruption of carrier surface may even take place during the "short period of time"; and
- ii) that detectable amounts of subsurface Na may also end up in the pretreatment solutions, e.g. due to some possible local dissolution of parts of the carrier surface.
- 3.3.2 For the following reasons, the Board, however, finds unconvincing the Respondent's allegations, unsupported by any experimental evidence, that the amounts of subsurface Na possibly dissolved during the "short period of time" of the test for the baseline, or those that can found in the pretreatment solutions at the end of a pretreatment step "(i)" as defined in claim 1, should be expected to be rather large, and possibly so large as to deprive of any meaning the % of surface Na removed, measured as indicated above:
 - i) For the Board, the key question as to the reproducibility of the invention as regards requirement (A) is not whether it is possible to determine with high accuracy the actual amount of surface Na that the nitric acid solution is able to rapidly remove, and the actual amount of surface Na that is removed during step "(i)". The relevant question is rather whether or not the skilled person is able to set the conditions (e.g. of time and temperature) of step "(i)" of the process of claim 1 so that this pretreatment results in the removal of at least 25% of the surface Na.
 - ii) Due to the undisputedly much faster dissolution of surface Na in the nitric acid solution, any "error" or uncertainty possibly produced by a simultaneous (but

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slower) dissolution of subsurface Na can only affect the surface Na baseline as measurable from the "Acid-Leachable test" to some limited, albeit possibly detectable, extent. In particular, such limited "error" can only render the measured surface Na baseline value slightly higher than the actual amount of surface Na.

- iii) The surface Na is primarily, if not exclusively, dissolved during the pretreatment "(i)", as this step is a mild treatment at low temperature. Indeed, as stressed by the Appellant, it is explicitly indicated in the patent in suit (see in particular paragraphs [0027] to [0029] and [0047]) that requirement (C) implies setting the conditions of step "(i)" so that only a limited amount of silicate ions should be removed from the carrier surface, in order to preserve the carrier structure, and thus the carrier strength. Since the dissolution of subsurface Na ions requires, however, such unwanted disruption and dissolution (of at least a portion) of the carrier surface, it is plausible that as long as the pretreatment is carried out so that the Si/ Na ratio remains below 5, the dissolution of subsurface Na can only contribute to a limited extent to the amount of Na that can be found in the pretreatment solutions.
- iv) Finally, it is plausible that any dissolution of subsurface Na possibly occurring during the mild pretreatment step is also a relatively slow process, as it requires additional time for the unwanted local disruption/dissolution of the carrier's surface and, possibly, also of other portions of the carrier matrix surrounding subsurface Na, in comparison to the dissolution of the surface Na that is immediately accessible to the pretreatment solutions. Hence it is plausible, that the dissolution of subsurface Na is very limited, in particular during the phases of the

pretreatment intended to remove surface Na. This applies, more particularly, to the initial phase of the pretreatment required for removing the first 25% of the surface Na baseline. The amount of subsurface Na that possibly dissolves during such initial phase is thus particularly limited. Of course, any "error" due to the dissolution of subsurface Na might become more relevant the longer the pretreatment is carried out after such minimum time, and the higher the temperature applied, but in all such cases the amount of surface Na actually removed will certainly have crossed the threshold of 25% of the surface Na baseline and, thus, requirement (A) will certainly be complied with.

3.3.3 Hence, for the Board, at least in all those embodiments of the claimed process in which the (overall) amount of Na ions found in the pretreatment solutions is well above 25% of the measured surface Na baseline (possibly slightly higher than the actual amount of surface Na in the untreated carrier), the fraction of this amount that actually derives from surface Na must also inevitably be above the 25% of the actual amount of surface Na present in the untreated carrier. In other words, any "errors" possibly associated to the procedures implied or described in the patent in suit for measuring the surface Na baseline and the % of surface Na removed, do not prevent the skilled person from carrying out all those embodiments of the claimed process wherein the measured amount of Na in the pretreatment solutions is well above 25% of the measured Na baseline, certainly comply with requirement (A).

The Board thus holds that the ambiguity deriving from such possibly unavoidable but limited "errors" does not "permeate the whole claim" and hence does not "deprive the skilled person of the promise of the invention" in

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the sense of e.g. decision T 0608/07 of 27 April 2009, Reasons, 2.5.2).

- 3.3.4 Already for this reason, the Board concludes that based on the information contained in the patent in suit the skilled person is in the position to carry out, without undue burden, embodiments of the claimed process wherein the conditions applied in step "(i)" are such that requirement (A) is met.
- 3.4 For the sake of completeness, the Board nevertheless considers it appropriate to additionally comment the case of a skilled person seeking to carry out processes according to the invention at the borderlines of requirement (A) of claim 1, in which the % of surface Na removed is either exactly 25% or slightly above this value.
- 3.4.1 This particular case was considered in the decision under appeal (Reasons, 2.4.1.9 and 2.4.1.10). The Department of First Instance concluded that the disclosure in the patent did not allow to carry out such processes (Reasons, 2.4.1.12 in combination with 2.6).
- 3.4.2 The Board finds instead that the above-discussed possible "errors" originating from the dissolution of subsurface Na during the "Acid-Leachable test" and during the actual pretreatment, do not make it impossible to actually carry out (technically speaking) processes at the borderlines of requirement (A). They might at most make it impossible to come to an unambiguous conclusion as to_whether or not such processes (that can be carried out and for which the measured amount of surface Na removed is 25% or just above about 25%) are embodiments of the claimed process, i.e. whether they fall within the ambit of claim 1.

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Thus, these "errors" would at most give to issues of clarity (Article 84 EPC) regarding the exact boundaries of the claim. However, since this issue is not caused by a post-grant amendment it is not relevant in the present opposition/appeal proceedings (G 3/14, OJ 2015, A102, Order).

- 3.5 For the above reasons, the Board is not convinced that the disclosure in the patent in suit as regards requirement (A) is so incomplete and/or ill-defined that the skilled person is unable to carry out the claimed process.
- 4. Requirement (B) (i.e. "the treatment ... partially replaces the removed sodium with lithium to yield a surface with up to 10 ppm lithium ions")
- 4.1 As pointed out by the Respondent, the patent in suit provides no explicit indication on how to measure the amount of Li on the surface of the carrier and does not explicitly state the basis reference for the (relative) threshold value of 10 ppm.
- 4.2 The Board is, however, convinced that the only technically reasonable interpretation of the threshold value of "up to 10 ppm" in the context of the patent is that it defines the weight parts of Li present on the carrier surface (i.e. the weight parts of surface Li) per one million weight parts of the whole pretreated carrier.
- 4.2.1 The Respondent argued that other meanings could also be theoretically plausible as regards the required maximum amount of surface Li (expressed in "ppm"), because the patent in suit also referred in Table 9 to the XPS method for measuring the amounts of Na and Si atoms on

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the carrier's surface. Thus, requirement (C) could also refer to the weight or molar ratio, expressed in "ppm", of Li atoms to all the atoms forming the carrier "surface".

In this respect, the Board notes preliminarily that the direct quantitative investigation at atomic level of the superficial composition of a carrier does not appear to be conventional, let alone the determination of Li in concentrations of less than 10 ppm by XPS. The only passage in the patent in suit reporting values apparently determined by "XPS" (in Table 9) concerns (undefined) "atomic %" of Na or Si, and not "ppm" of Li. If only for this reason the Board does not find it plausible that the skilled person would attribute to requirement (C) the meanings considered theoretically possible by the Respondent.

4.3 Moreover, the Board holds that the patent in suit must be understood to imply that substantially all the Li present in the carrier is located on the surface of this latter, i.e. is all surface Li.

As a matter of fact, the whole process of claim 1 is essentially a surface treatment, and nothing in the whole patent suggests that the Li remaining on the carrier could also migrate into the subsurface of this latter. As already mentioned (3.1.5, supra), the only disclosure of migration in the patent in suit in (paragraph [0048]) refers to Na ions possibly migrating from the subsurface to the surface at very high drying temperatures.

Hence, the Board finds unconvincing the Respondent's unsupported allegation, disputed by the Appellant, that

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Li might instead also migrate into the subsurface of the carrier during the pretreatment step "(i)".

- 4.4 The Board concludes that the patent in suit equates the amount of surface Li to the overall content of Li in the pretreated carrier. It is apparent and undisputed that this latter can easily be determined, e.g. using conventional chemical analysis of the carrier's overall composition or from the difference between the amount of Li present in the initial Li salt solution(s) and the overall amount of Li that can be found in the totality of the pretreatment solutions at the end of step "(i)". The arguments exchanged as regards the suitability of XPS or other methods for analysing directly the composition of the carrier surface, and more particular, for determining quantitatively very low amounts of Li on the surface of the carrier, need thus not be dealt with here.
- 4.5 The Respondent also objected that the patent in suit did not disclose how to set of the pretreatment conditions so as to ensure that the amount of surface Li in the pretreated carrier complies with requirement (B).
- 4.5.1 In this respect, the Board holds, on the one hand, that the skilled person finding that a certain setting of the pretreatment "(i)" results in a treated carrier comprising an amount of Li that is too high, would, based on common general knowledge in chemistry, routinely consider the possibility of lowering the Li concentration in the Li salt solution(s) used and/or the duration of the contact between the carrier and such solution.
- 4.5.2 However, in the present case, the patent in suit even indicates explicitly that the aim of the final washing

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of the pretreatment "(i)" is to limit the extent of contamination e.g. also by residual Li salt solution which leads to (possibly "unbound") Li on the surface of the carrier, the latter interfering with the catalytic function and stability (see, in particular, paragraphs [0029], [0035], [0038], [0039], [0046], [0090] and [0107]).

In other words, the patent in suit explicitly teaches that the washing(s) can be used for reducing the amount of surface Li, if only because these washing treatments remove any trace of the Li salt solution possibly remaining in the pores of the carrier.

- 4.6 Hence, based on the above considerations, the Board concludes that the patent in suit clearly suggests repeated or prolonged washing with water in order for requirement (B) to be met.
- As to the other aspect of requirement (B), i.e. that the Li only "partially replaces the removed sodium" (emphasis added), the Board holds that in the context of the patent in suit it must be understood to mean that the amount of surface Li (i.e. all the Li remaining in the pretreated carrier) must be less than the amount of removed surface Na. Here again,
 - the "error" possibly due to subsurface Na affecting the measured amounts of surface Na removed,
 - and even any "error" hypothetically arising according to the unsupported allegation of the Respondent that some of the Li in the pretreated carrier could be subsurface Li,

could at most give rise to an issue of clarity for those embodiments of the claimed process in which the measured amounts of surface Na removed and of surface Li in the pretreated carrier are very similar.

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In addition, for the Board, the very fact that the initial amount of surface Na is above 40 ppm in all "low sodium" carriers exemplified in the patent (Table 1), implies the suggestion to use carriers with a surface Na baseline of 40 ppm or more. Starting from any of these suggested carriers and performing the pretreatment so as to achieve the required removal of at least 25% of such baseline, inevitably results in an amount of surface Na removed distinctly above 10 ppm. Hence, given that 10 ppm is the maximum amount allowed for the surface Li, the "partial" replacement of the removed Na by Li will be - in all such embodiments - the inevitable consequence of requirement (A) being met and of the limitation on the amount of Li in the carrier also imposed by the remainder of requirement (B).

- 4.8 Accordingly, the Board is not convinced that the disclosure in the patent in suit as regards requirement (B) is so incomplete and/or ill-defined that the person skilled in the art is unable to carry out the claimed process.
- 5. Requirement (C) ("the weight ratio of removed Si/Na is 5.0 or less")
- 5.1 For the Board, it is implicitly, but nevertheless clearly, described in the examples of the patent in suit that this ratio corresponds to the weight ratio of Si to Na in the pretreatment solutions (see also the decision under appeal, Reasons, 2.4.3.1). However, it is also apparent to the Board that, as convincingly argued by the Respondent, in the context of the patent in suit this ratio is also meant to correspond to the ratio of the amounts of Si to Na removed from the surface (see in the patent in suit paragraph [0026]).

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Hence, the Board concludes that the patent in suit equates the ratio of Si and Na removed from the surface to the ratio Si/Na in the pretreatment solutions.

- 5.2 The Respondent nevertheless argued that the value for the Si/Na ratio measured in the pretreatment solutions would not correspond to the ratio of remove surface Si to removed surface Na, due to the contribution of Na and Si possibly stemming from the subsurface of the carrier matrix.
- 5.2.1 In this respect, the Board stresses again (see also 3.3.2 iii, supra) that requirement (C) implies setting the conditions of step "(i)" so as to preserve the integrity of the carrier surface (see, for instance, paragraphs [0027], [0035] and [0047]). This also implies that pretreatment conditions (e.g. lower temperatures) resulting in lower amounts of dissolved Si also limit reduce the possible exposure of subsurface species (and thus also of subsurface Na and subsurface Si) to the pretreatment solutions.
- 5.2.2 Hence, in the absence of any supporting evidence to the contrary, the Board has no reason to doubt that only very limited amounts of subsurface Na and subsurface Si are dissolved in the pretreatment solutions, at least when keeping their Si/Na ratio well below 5 by using appropriate conditions. Thus, a possible simultaneous dissolution of some Na and Si stemming from the subsurface will not be so pronounced that it would result in an ambiguity permeating the whole disclosure relating to requirement (C).
- 5.2.3 Therefore, the Board accepts that at least in all those embodiments of step "(i)" wherein the Si/Na ratio measured in the pretreatment solutions is substantially

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below 5, it will correspond to the actual ratio of removed surface Si / surface Na.

Again, the consequence of the alleged ambiguities or "errors", if any, would at most be an issue of clarity, but not insufficiency, and only for those embodiments of the claimed process wherein the measured weight ratio Si/Na in the pretreatment solutions is about 5.

- 5.3 Accordingly, the Board is not convinced that the disclosure in the patent in suit as regards requirement (C) is so incomplete and/or ill-defined that the person skilled in the art is unable to carry out the claimed process.
- 6. The mandatory presence of surface Na in the final catalyst.
- 6.1 The Respondent stressed that claim 1 requires the presence of surface Na in the pretreated carrier supporting the silver component, i.e. in the final catalyst, considering that its preamble reads (emphasis added):
 - "A process for the preparation of a catalyst for the production of ethylene oxide comprised of silver supported on an alumina carrier containing both sodium and silicate ions on the surface thereof".
- 6.2 The Board notes that this wording does not define any minimum value for the surface Na of the catalyst. This requirement is thus clearly complied with even if the catalyst comprises just detectable traces of surface Na. Thus, already upon considering that the physical separation of the carrier from the Li salt solution leaves traces of this solution on the treated carrier

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surface and in its pores, and also considering that the subsequent washing only achieves "reducing the amount of surface contamination" (paragraph [0039] of the patent specification; emphasis added), the Board accepts that the Appellant is correct in stating that some residual surface Na must inevitably always be present in the carrier at the end of step "(i)" and even of step "(ii)".

- 6.3 Thus, the Board concludes that the requirement in claim 1 as to the presence of surface Na also in the produced catalyst, is manifestly always complied with when carrying out process steps (i) and (ii) following the teaching of the patent in suit.
- 7. Undue burden in terms of experimental work required requirements A , B and C in combination
- 7.1 Finally, according to the Respondent, the absence of clear technical definitions and indications as to requirements A, B and C imposed an undue burden on the person skilled in the art seeking to reproduce the claimed invention. In particular, based on a number of complex technical considerations he had to carry out extensive experimental work, prior to be able to repeat the claimed process. Thus, the invention had not been sufficiently disclosed.
- 7.2 The Board acknowledges that the explicit technical disclosure of the patent in suit lacks the desirable precision under some aspects.
- 7.2.1 However, as set out above, the Board holds that the person skilled in the art would not be confronted with particular difficulties when seeking to identify the appropriate temperature and length of time for the

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- "Acid-Leachable test" that allow to determine with sufficient certainty the surface Na baseline.
- 7.2.2 Moreover, the patent in suit contains valuable technical information and guidance as to the nature of suitable carrier materials (paragraphs [0027], [0051], [0052] and [0072]), as well as of suitable Li salts and their concentrations (paragraph [0036] and examples).
- 7.2.3 Thus, the Board is also convinced that the person skilled in the art is able to identify, among commercially available low-sodium carriers based on alpha-alumina with silicate on its surface, suitable starting materials having, for instance, a surface Na baseline comparable to those reported in Table 1. Also suitable Li salt solutions (e.g. similar to those used in the examples) can be provided without difficulty.
- 7.2.4 The remaining instructions contained in the patent in suit (in particular those in paragraphs [0031] to [0039]) further suggest to the skilled person (who would also be aware of the possibly unavoidable but limited contribution of subsurface species to any measure to be made when checking compliance with requirements (A) to (C) to perform, inter alia, the following operations:
 - i) monitoring, by conventional analytical means, the amount of Na, Li and Si in the aqueous phase during the treatment with aqueous Li salt solution(s) (of known starting Li content), and carrying out such treatment at a temperature sufficiently below 100°C and for a length of time that allow to obtain an amount of Na in the Li salt solution well above 25% of the surface Na baseline, but still ascertaining that that amount of removed Si remains well below 5 times that of the removed Na, and then

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- ii) carrying out washing with water of the carrier while monitoring the amount of Na, Li and Si in the wash water phase(s), so as to achieve sufficient removal of Li contamination (i.e. leaving in the pretreated carrier a amount of Li which is clearly less than 10 ppm), but not to continue such washing so long that too much of the Si is removed (i.e. so as to still keep the removed Si/Na ratio clearly below 5).
- 7.3 In the absence of any evidence to the contrary, the Board sees no reason for considering that the person skilled in the art would experience serious difficulties in performing any of the above operations. Thus, the Board has no reason to consider that an undue amount of experimental work is needed to carry out many different embodiments of the claimed processes across the breadth the claim that certainly comply with the requirements given for step "(i)".

Moreover, carrying out specifically those processes for which the measured % of removed surface Na is very close to 25% and/or the measured surface Li is very close to 10 ppm and/or the measured removed Si/Na ratio is very close to 5, does not involve any undue burden of experimental work, but only generates doubts as to whether or not such processes at the borderlines of the invention as defined in claim 1 actually fall within the ambit of claim 1.

8. Hence, in the Board's judgement, the invention as defined in claims 1 to 3 of the Main Request is disclosed in the patent in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art. The requirements of Article 83 EPC are thus met.

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9. Remittal of the case

Since the decision under appeal only addressed the ground of opposition of Article 100(b) EPC, the Board considers it appropriate to remit the case to the Opposition Division pursuant to the provisions of Article 111(1) EPC, in accordance with the corresponding requests of both Parties.

Order

For these reasons it is decided that:

- The decision under appeal is set aside.
- The case is remitted to the Department of First Instance for further prosecution.

The Registrar:

The Chairman:



D. Magliano

B. Czech

Decision electronically authenticated