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D E C I S I O N
of 5 November 2002

Case Number: T 0584/99 - 3.3.5

Application Number: 91909631.3

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IPC: C01B 11/02

Language of the proceedings: EN

Title of invention:

Electrochemical production of acid chlorate solutions

Patentee:

Sterling Canada, Inc.

Opponent:

Eka Chemicals AB

Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (yes): Exclusion of hindsight"

Decisions cited:

-

Catchword:

-



Case Number: T 0584/99 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 5 November 2002

Appellant: Eka Chemicals AB
(Opponent) S-44580 Bohus (SE)

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Decision under appeal: Interlocutory decision of the Opposition Division
of the European Patent Office posted
11 March 1999 concerning maintenance of European
patent No. 0 532 535 in amended form.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: A. T. Liu
J. H. Van Moer

Summary of Facts and Submissions

I. European patent No. 0 532 535 was granted with a set of claims consisting of an independent claim 1 for a method with claims 2 to 24 depending thereon, and an independent method claim 25.

II. A notice of opposition was filed against the patent on the grounds of Article 100(a) EPC. During the opposition proceedings, seven documents were cited, of which reference shall be made to the following two in the present decision:

D1: US-A-4 129 484

D3: Chem. Ing. Tech. 61 (1989), Nr.12, Dec. 1989,
pages 933 to 935

III. At the end of the oral proceedings which were held on 19 January 1999, the opposition division came to the conclusion that, account being taken of the amendments according to auxiliary request 2, the patent and the invention to which it related met the requirements of the Convention.

IV. The decision of the opposition division was based on a set of claims consisting of an independent method claim 1 and claims 2 to 22 depending thereon. Claim 1 read as follows:

"A method for the production of chlorine dioxide, comprising the steps:

providing an aqueous acid chlorine dioxide-generating reaction medium containing sulfuric acid and

alkali metal ions in a reaction zone,

maintaining said aqueous acid reaction medium at its boiling point while a subatmospheric pressure is applied to the reaction zone,

forming an aqueous solution of a mixture of alkali metal chlorate and alkali metal sulfate from solid phase materials, said aqueous solution containing alkali metal ions and hydrogen ions in a molar ratio of from 1000:1 to 1:2,

electrochemically acidifying at a current efficiency of at least 70%, preferably at least 80%, said aqueous solution,

electrochemically removing alkali metal ion from said aqueous solution to produce an acidified alkali metal salt feed solution,

forwarding said acidified alkali metal salt feed solution as an acidified chlorate ion-containing solution to said reaction zone to provide chlorate ion and hydrogen reactants to said aqueous acid chlorine dioxide-generating reaction medium,

crystallizing an alkali metal sulfate from said aqueous acid reaction medium in said reaction zone, and recovering from said reaction zone an alkali metal sulfate."

- V. A notice of appeal was filed by the opponent who, in his statement of the grounds of appeal, submitted the reasons as to why, in his view, the subject-matter of all the claims on file lacked an inventive step in view

of D1 and D3.

VI. The appellant's arguments may be summarised as follows:

- The problem with regard to the closest prior art D1 could be seen in an improvement of the current efficiency in the electrolytic acidification step.
- The solution as proposed in claim 1 was essentially the stipulation that the aqueous solution contained, besides alkali metal sulfate, also alkali metal chlorate obtained from solid phase material.
- Considering that D3 revealed that a high molar ratio $\text{Na}^+:\text{H}^+$ improved the current efficiency, there was a clear incentive for a person skilled in the art to add alkali metal chlorate before the acidification step.
- The current efficiency stipulated in claim 1 was only a desideratum which should not be taken into consideration for the assessment of inventive step.

VII. The respondent's arguments filed in response were briefly as follows:

- The technical problems associated with such processes as known from D1 were not only the low current efficiency but also the large volume of liquid in circulation.
- This water balance problem was not mentioned in D1.

- There was no suggestion in D1 that an aqueous solution of both alkali metal chlorate and alkali metal sulfate made up from solid phase materials should be electrochemically acidified.

- There was no indication in D3 that the addition of alkali metal chlorate to alkali metal sulphate prior to the acidification step would lead to the improvement of current efficiency.

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

The respondent (patentee) requested that the appeal be dismissed.

Reasons for the Decision

1. *Amendments*

The Board is satisfied that the present claims meet the requirements of Article 123(2) and (3) EPC. There is no dispute on this point.

2. *Novelty*

It is also common ground that the process of claim 1 is new with respect to the available prior art. This will be clear from the following discussion on inventive step.

3. *Inventive step*

3.1 Claim 1 is directed to a method for the production of

chlorine dioxide by maintaining an aqueous acid reaction medium of alkali metal chlorate and alkali metal sulfate at its boiling point, while applying a subatmospheric pressure to the reaction zone. The process in particular includes the electrochemical processing of an aqueous solution to produce that acidic reaction medium (see also patent in suit, page 2, lines 5 to 6).

- 3.2 The Board can accept the view of both parties that the closest prior art is represented by D1.

D1 is directed to a process for working up residual solutions from a reactor in which sodium chlorate is reduced to chlorine dioxide in the presence of an acid, in particular sulphuric acid, whereby acid is consumed. The residual solution is led to an electrolytic cell, at the anode region of which an acid enriched fraction of the residual solution is prepared (this step will be referred to hereinafter as the "electrolytic acidification step") and recycled to the chlorine dioxide reactor for repeated use as acidifying agent (abstract; column 3, lines 3 to 13; column 7, line 17 to column 8, line 51; Examples 1 to 3 and Figures 1 to 3

- 3.3 The respondent has submitted that one of the problems associated with the production of chlorine dioxide at subatmospheric pressure (which is the case of the present process) is the water balance, which problem would arise from the large volume of the residual solution in circulation as is proceeded in D1 (patent in suit, page 2, lines 47 to 49 and page 5, lines 22 to 24).

Another drawback of the process of D1 is that the residual solution from the chlorine dioxide process has a low acid strength. Although higher sulfuric acid concentrations can be achieved in the electrolytic cell, the current efficiency for this electrolytic acidification step is low due to the leakage of H⁺ ions through the ion-exchange membrane. This problem, known in the art, is discussed in the patent in suit (see patent in suit, page 2, line 47 to page 3, line 9).

The Board therefore accepts the respondent's submission that, with respect to D1, the technical problem to be solved is two-fold, namely to improve:

- (a) the water balance of the whole process and
- (b) the current efficiency at the electrolytic acidification step.

3.4 To solve the technical problem as stated above, claim 1 essentially proposes a process comprising the following distinguishing features:

- (i) forming an aqueous solution of a mixture of alkali metal chlorate and alkali metal sulfate from solid phase materials,
- (ii) said aqueous solution containing alkali metal ions and hydrogen ions in a molar ratio of from 1000:1 to 1:2,
- (iii) electrochemically acidifying said aqueous solution at a current efficiency of at least 70% then forwarding the acidified solution to the chlorine dioxide-generating zone.

The Board hereby interprets the requirement of current efficiency under (iii) not as a mere desideratum but as a "functional" feature stipulating that all the other parameters, including the molar ratio of alkali metal ions to hydrogen ions, must be selected within their stipulated range(s) such that the stipulated current efficiency is obtained. In effect, feature (iii) at least restricts the range defined in (ii).

3.5 Claim 1 of the patent in suit requires forming an aqueous solution from solid phase materials. Therefore, the solution is not, as in D1, restricted to the concentration as it comes out of the reactor but the concentration of the solution to be acidified is made to specification (see for example patent in suit, page 4, lines 50 to 51 and page 5, lines 22 to 24). The Board therefore holds that feature (i) not only allows a decrease in the amount of water introduced into the chlorine dioxide generator but also the $\text{Na}^+:\text{H}^+$ ratio of the solution containing alkali metal chlorate and alkali metal sulfate be adjusted within the range specified in feature (ii) (which is further restricted by feature (iii), see point 3.4 above). There is also no dispute that the resulting electrolytic acidification step in claim 1 being more efficient than that disclosed in D1. The Board therefore has no doubt that the technical problem as stated in point 3.3 above is indeed resolved by the process according to claim 1.

3.6 It remains to be elucidated whether the solution proposed in claim 1 is obvious in view of the available prior art.

3.6.1 In both D1 and D3, the spent liquor from the reactor, in which chlorate is reduced to chlorine dioxide in the

presence of acid, is led directly to the electrolytic vessel for acidification. Neither of the documents discloses or suggests removing the water or part of it prior to the electrolytic acidification. Indeed the total volume of the recycle solution is not at issue, even though it is also envisaged in D1 that the electrolytic acidification be applied to residual solutions from processes which are performed at subatmospheric pressure (see D1, column 2, lines 3 to 8 and column 10, lines 40 to 47 and column 10, line 59 to column 11, line 2).

- 3.6.2 The appellant has argued that, as alkali metal chlorate must be added in order to produce chlorine dioxide, there are only two options available to the skilled person, namely to add the chlorate to the system either before or after the acidification step. Since D3 suggests that a high molar ratio $\text{Na}^+:\text{H}^+$ is desirable at the electrolytic acidification step, the skilled person would be led to choose the alternative of adding the chlorate before that acidification step.

As is not refuted by the appellant, D1 only discloses that sodium chlorate is fed directly to the chlorine dioxide reactor (see Figures 1 to 3 and column 7, line 27 to column 8, line 51). This prior art does not consider any alternative to that mode of feeding, let alone suggest adding chlorate to the spent solution.

Although it is known from D3 that a high molar ratio $\text{Na}^+:\text{H}^+$ improves the current efficiency, the solution to be acidified here is again an aqueous solution which contains only sodium sulfate (page 935, paragraph bridging left hand and right hand columns and Figure 3). There is no teaching as to how a desired

Na⁺:H⁺ molar ratio should be achieved, in particular there is no mention that any further component should be added to the solution to be treated.

3.6.3 The Board therefore holds that, without the benefit of hindsight, the skilled person cannot derive from D3 any incentive for adding chlorate to the solution to be acidified electrochemically, let alone to do so with a view to solve the present two-fold technical problem. The appellant has not submitted any argument to convince the Board that the electrolytic acidification of an aqueous solution of both alkali metal chlorate and alkali metal sulfate made up from solid phase materials would be rendered obvious by additionally taking into consideration any of the other prior art documents on file. On the basis of the available evidence, it cannot therefore be denied that the process according to claim 1 involves an inventive step.

3.7 Claims 2 to 22 are dependent claims relating to specific embodiments of the process according to claim 1. Their subject-matter is therefore also novel and involves an inventive step.

Order

For these reasons it is decided:

The appeal is dismissed.

The Registrar:

The Chairman:

U. Bultmann

R. Spangenberg