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**D E C I S I O N**  
**of 17 October 2002**

**Case Number:** T 0678/99 - 3.3.5

**Application Number:** 93912503.5

**Publication Number:** 0644852

**IPC:** C01B 11/02

**Language of the proceedings:** EN

**Title of invention:**

Hydrogen peroxide-based chlorine dioxide process

**Patentee:**

STERLING CANADA, INC.

**Opponent:**

Eka Chemicals AB

**Headword:**

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**Relevant legal provisions:**

EPC Art. 123(2),(3), 54, 56

**Keyword:**

"Public prior use - not proven"

"Inventive step (yes) - after amendments non-obvious  
modification"

**Decisions cited:**

T 0877/90, T 0958/91

**Catchword:**

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Case Number: T 0678/99 - 3.3.5

**D E C I S I O N**  
**of the Technical Board of Appeal 3.3.5**  
**of 17 October 2002**

**Appellant:** STERLING CANADA, INC.  
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**Decision under appeal:** Decision of the Opposition Division of the  
European Patent Office posted 5 May 1999 revoking  
European patent No. 0 644 852 pursuant to  
Article 102(1) EPC.

**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. 0 482 102 was granted with a set of claims consisting of a process claim 1 with claims 2 to 10 depending thereon, and a further independent process claim 11 with claims 12 to 38 depending thereon.

II. A notice of opposition was filed against the patent on the ground of Article 100(a) EPC. Of the 18 documents which were cited in the course of the opposition proceedings, reference shall be made to the following in the present decision:

D2: Declaration signed by William E. Nearpass,  
14 March 1997, with Appendices A to J

D2a: Declaration signed by H. Falgen, 25 January 1999

D3: US-A-5 091 166

D4: US-A-5 091 167

D6: US-A-2 332 181

D8: US-A-5 066 477

D9: US-A-3 864 456

D10: A pamphlet entitled "Chlorine Dioxide, The SVP process", by Cellchem, Nobel Industries Sweden (undated)

D15: The Bleaching of Pulp, Third Edition, Ed. by R.P. Singh, pages 628 to 639, TAPPI press (1979)

- D16: A pamphlet entitled "SVP-HP™ ClO<sub>2</sub> process", by Eka Nobel, distributed at the TAPPI Conference, November 1992.
- III. At the oral proceedings before the opposition division, the patentee submitted two modified sets of claims as basis for a main request and an auxiliary request.
- IV. The opposition division held that the availability to the public of the documents filed in support of the allegation of public prior use, Appendices A to J of D2, was not proven. The process according to the amended claim 1 of both the main and auxiliary requests was however found to lack an inventive step with regard to D3 or D4 as closest prior art document, in combination with either of D8 or D9, each disclosing a process using a mixed feed with methanol or, respectively, sodium chloride as reducing agent.
- V. An appeal was lodged by the patentee against the decision of the opposition division revoking the European patent.
- VI. With the statement of the Grounds of appeal, the appellant filed a new set of claims 1 to 23 which were to form the basis for his main request. Three further sets of claims were submitted as auxiliary requests with a letter dated 16 September 2002.
- VII. In support of his arguments, the appellant introduced 7 additional documents for the first time into the proceedings.
- VIII. At the oral proceedings which took place on 17 October 2002, the appellant filed a new set of amended claims

consisting of a process claim 1 with claims 2 to 23 depending thereon. Claim 1 read as follows:

"A process for the production of chlorine dioxide comprising:

reducing chlorate ions with hydrogen peroxide in an acid aqueous reaction medium having a total acid normality of about 0.1 to about 11 N in a reaction zone,  
maintaining said reaction medium at its boiling point while a subatmospheric pressure is applied to said reaction zone, the reaction temperature being at least 50°C,  
maintaining substantially steady state conditions in said reaction zone by continuously feeding into said reaction zone an aqueous chlorate solution, hydrogen peroxide and strong mineral acid,  
pre-mixing the hydrogen peroxide feed to said reaction medium with at least a portion of said aqueous chlorate solution in a mixing zone which is a feed line only for said aqueous chlorate solution to said reaction zone or a vessel separate from the reaction zone, prior to feeding into said reaction zone to provide the hydrogen peroxide feed at a concentration of less than 15 wt%,  
and  
removing chlorine dioxide, oxygen and steam from said reaction zone."

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

- Claim 1 was based on claim 11 as filed and contained further restricting features derived from the description.

- There was no evidence that D2 was available to the public before the priority date of the patent in suit.
- The process of claim 1 was distinguished from that of D3 or D4 by the essential feature of pre-mixing of hydrogen peroxide with chlorate prior to their feeding into the reaction zone to obtain a concentration of less than 15 wt% hydrogen peroxide in the feed.
- The pre-mixing and the resulting dilution of the hydrogen peroxide feed was to provide a consistently efficient and predictably stable process.
- There was no prior art document suggesting such pre-mixing in a subatmospheric process for the production of chlorine dioxide.

X. The respondent's arguments were essentially the following:

- The amendments to claim 1 were not supported by the original application documents.
- The information contained in Appendices A to J of D2 was, in view of the decisions T 877/90 and T 958/91, not regarded as secret information. The process of claim 1 therefore lacked novelty in view of D2.
- It was queried whether the pre-mixing actually solved the stated technical problem with respect to D3 or D4.

- None of the prior art documents relating to hydrogen peroxide based chlorine dioxide production taught against pre-mixing hydrogen peroxide with the chlorate feed. In fact, this was explicitly recommended in D6.
- Both D3 and D4 explicitly referred to an SVP reactor as suitable for operating the disclosed process. As could be seen in D15 and confirmed by D2a, such SVP plants were designed for adding the chlorate and the reducing agent as a pre-mixed solution in the same feed line.

XI. At the end of the oral proceedings, the parties's requests were the following:

- The appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the set of claims 1 to 23 submitted during the oral proceedings (Main request) or, in the alternative, on the basis of the set of claims submitted with the statement of the Grounds of appeal or on the basis of the sets of claims submitted with the letter dated 16 September 2002.
- The respondent requested that the appeal be dismissed.

## **Reasons for the Decision**

### *Main request*

#### 1. *Amendments*

1.1 Claim 1 is essentially based on claim 11 as originally filed and granted. In addition, it contains the following stipulations, whose basis in the original application documents is given in parentheses:

- (i) the reaction temperature is at least 50°C (claim 19),
- (ii) the pre-mixing of the hydrogen peroxide with chlorate solution is in a the mixing zone which is a feed line only for the aqueous chlorate solution or a vessel separate from the reaction zone (page 7, lines 14 to 17; page 11, lines 28 to 32),
- (iii) the mixing is to provide the hydrogen peroxide feed at a concentration of less than 15 wt% (page 10, lines 31 to 34).

1.2 With regard to the feature (ii) above, the respondent has raised the objection that there is no basis in the original application documents for the stipulation that hydrogen peroxide is pre-mixed with chlorate only. Rather, the original description would disclose pre-mixing of hydrogen peroxide with chlorate and other chemicals.

The Board remarks that the description as filed indicates that the "hydrogen peroxide feed may be pre-mixed, prior to feeding into the reaction medium, with at least a portion of either or both of the aqueous chlorate solution or the strong mineral acid feeds or mixtures thereof" (description page 7, lines 14 to 17) and that "the hydrogen peroxide can be pre-mixed by feeding it into either one or both of the aqueous



chlorate solution feed line or the strong mineral acid feed line" (page 11, lines 28 to 33). In the Board's judgment, these two passages of the description, when taken together, discloses the following distinct options:

- (a) the hydrogen peroxide is pre-mixed with aqueous chlorate solution only, in the feed line for the latter, or
- (b) the hydrogen peroxide is pre-mixed with the strong mineral acid only, in the feed line for the acid, or
- (c) the hydrogen peroxide is pre-mixed with both the aqueous chlorate solution and the strong mineral acid, in the respective feed line.

The Board holds that the additional feature of pre-mixing of the hydrogen peroxide with chlorate solution in a mixing zone which is a feed line only for the aqueous chlorate solution corresponds to option (a). The stipulated feature is therefore found to be fairly based on the application documents as filed.

1.3 The dependent claims 2 to 23 essentially correspond to claims 16 to 18 and 20 to 38 as filed and granted. The present amended claims thus meet the requirements of Article 123(2) and (3) EPC.

2. Allegation of public prior use

2.1 In the opposition proceedings, the respondent has submitted D2 in support of his allegation that it was common practice before the priority date of the patent

in suit to pre-mix the reducing agent with the chlorate feed in SVP processes for producing chlorine dioxide. The opposition division, however, did not accept D2 and the related documents, Appendices A to J, as evidence in support of the contention of public prior use (see decision under appeal, page 6, item 5). In particular, it was queried by the opposition division as to whether the most relevant document (Appendix J) was actually received by the customer in due time.

The Board holds that, in view of the doubt expressed by the opposition division, the onus is on the respondent to provide confirmation from at least one of the numerous addressees as appeared in these Appendices. On the contrary, the respondent submitted at the oral proceedings before the Board that he would not produce any further evidence in this respect. The Board therefore does not see any reason for challenging the position taken by the opposition division in the impugned decision.

2.2 The present case is not comparable with the cases cited by the respondent where the Boards concerned accepted that the opponent has proved his allegation of public prior use beyond all reasonable doubt.

In the case T 958/91 of 25 March 1994, it was conceded by the patentee that about 150 copies of a pamphlet containing the relevant information were distributed before the priority date of the patent in suit. It was thus an undisputed fact that these pamphlets were received in due time (see point II and second paragraph of point 2 of the decision).

In the case T 877/90 of 28 July 1992, the Board found

that a document which was an abstract of a lecture presented at a scientific meeting constituted prior art within the meaning of Article 54(2) EPC. Again, it was an uncontested fact that the date of the meeting in question fell before the priority date of the patent in suit (see points 2.1.2, 2.1.4 and 2.1.8 of the decision).

The Board's findings in the present case are therefore not in conflict with the decisions cited by the respondent.

3. *Novelty*

The Board is satisfied that none of the remaining documents discloses a process as claimed. This will also follow from the discussion on inventive step (see in particular point 4.4).

4. *Inventive step*

4.1 The subject-matter of claim 1 is a process for the production of chlorine dioxide under subatmospheric pressure conditions, comprising reducing chlorate ions with hydrogen peroxide in an acid aqueous reaction medium having a total acid normality of about 0.1 to about 11 N in a reaction zone.

4.2 D3 and D4 are both directed to a subatmospheric process for producing chlorine dioxide involving the reduction of chlorate with hydrogen peroxide. In D3, the acidity of the reaction medium is 2 to 5N while in D4 it is from 5 to 11N (see respective abstract). In agreement with the parties, the Board can accept these prior art documents on an equal basis as comprising the closest

prior art. For simplicity, reference shall only be made to D3 for the purpose of the present decision.

4.3 The appellant has submitted that, with respect to D3, the technical problem to be solved is the provision of a consistently efficient and predictably stable process (see also patent in suit, column 2, lines 7 to 11 and column 6, lines 29 to 57).

4.4 The appellant's position was that the above indicated technical problem is solved by the provision of a process which is essentially distinguished from D3 in the dilution of the hydrogen peroxide feed to a concentration of less than 15 wt% by pre-mixing it with at least part of the chlorate solution **prior to feeding into the reaction zone** (point VIII above, claim 1). In the Board's view, it is therefore crucial to determine at this point what constitutes the reaction zone.

4.4.1 It is common ground that a suitable reactor for carrying out the prior art process as well as the present process is a so-called SVP (trade mark for "single vessel process") reactor. Such a reactor is illustrated in D10 and D16. Both documents show a generator which is a large vessel in which the sodium chlorate is reacted to form chlorine dioxide. The generator contents are circulated via a pump to promote good mixing and to circulate the fluid through a generator reboiler. The chemical feeds are injected into the recirculation line (see D10, page 4, left hand column: "Chlorine dioxide generation" and the accompanying Figure on the right hand column; D16, page 2 of 9, right hand column: "SVP-HP system description"; page 3 of 9: "SVP-HP Schematic" and page 4 of 9, left hand column: "Generator"). As was

confirmed by the respondent at the oral proceedings, whilst the generator is the heart of the process, the recirculation line is also part of the reaction zone.

4.4.2 The appellant has submitted that it is common usage that laboratory chlorine dioxide reactors also comprise a heart piece which is the generator itself and a recirculating line. Thus, although this is not explicitly indicated, in all the examples in D3, the reactants will also be fed into the recirculation line and not directly into the generator vessel. This is not refuted by the respondent.

4.4.3 As a consequence, the Board interprets the claimed process as being essentially distinguished from D3 in that the hydrogen peroxide is pre-mixed with at least part of the chlorate solution **prior to feeding into a reaction zone including the recirculation line.**

4.5 The respondent has, however, queried whether the claimed process actually solves the technical problem as stated above (see point 4.3).

The Board remarks that the description as filed provides three examples in support of the invention as originally claimed. Thus, the hydrogen peroxide is diluted in Example 1 by pre-mixing with both the chlorate and acid solutions and, in Example 2, with water. In the last example the hydrogen peroxide is fed to the generator as a 30% solution. Against this background, the emphasis of the claimed invention has now shifted to a pre-mixing of hydrogen peroxide with chlorate only. As is conceded by the appellant, none of the examples given in the patent in suit is encompassed by the wording of present claim 1. It is therefore

questionable whether the advantage proclaimed in the patent in suit is actually achieved by the present process. In the lack of convincing evidence, the Board is prepared to accept that the problem to be solved with regard to D3 is the provision of a further process for the production of chlorine dioxide. It is undisputed that the technical problem so stated is solved by the process of claim 1.

4.6 The only remaining question is whether the modification involving pre-mixing hydrogen peroxide with chlorate prior to feeding to the reaction zone is obvious with regard to the available prior art.

4.6.1 The respondent has advanced the argument that there is no explicit disclosure in D3 to feed the reactants separately or to pre-mix these prior to their feeding into the reaction zone. On the other hand, it is explicitly recommended in D6 to feed the chlorate and hydrogen peroxide as a pre-mixed solution (see page 2, left hand column, lines 5 to 10). Thus a person skilled in the art would have a clear incentive to pre-mix the reactants when operating the process of D3.

The Board remarks that D6 concerns a method for generating chlorine dioxide at atmospheric pressure, employing sparging with air to remove the product. Unless there were a pointer in that direction, there is a priori no reason for the skilled person to combine the disclosure of D3, which is directed to a process working at subatmospheric pressures, with any aspect of this prior art teaching which dates back to 19 October 1943 and involves an old generator type working at atmospheric pressure.

4.6.2 The respondent has also pointed out that D3 explicitly refers to a SVP reactor for conducting the disclosed process (column 3, lines 50 to 53 of D3 and point 4.4.1 above). A known SVP process is for example explained in D15 which is an article reviewing the five processes used in North America for generating chlorine dioxide (page 628, last paragraph). In the specific passage concerning the SVP process, it is indicated that "sodium chlorate and sodium chloride, as mixed or separate solution, are fed to the circulating liquid head of the of the reboiler" (page 635, last paragraph). It is thus known in the art to pre-mix the reducing agent (which is sodium chloride in the case of D15) with chlorate prior to feeding to the reaction zone. When an existing SVP plant is converted to the use of hydrogen peroxide as reducing agent, it would be most obvious to minimise the costs of conversion by maintaining the single feed line and thus also to pre-mix the hydrogen peroxide with the chlorate.

The appellant, on the other hand, has pointed out that the cited passage of the text has to be interpreted in the light of the Figure 23.11 to which it refers (page 635, first sentence of the last paragraph: "Figure 23.11 is a schematic illustration of the typical process."). On that Figure, one can see the following feed lines:

- (a) a feed line for a R-2 solution or chlorate solution and
- (b) a feed line for a brine alternate, if split feeds.

It is undisputed that the R-2 solution is a pre-existing solution containing both chlorate and chloride

ions. Thus, the Board concurs with the appellant in that, when the text of D15 is interpreted in the light of Figure 23.11, one must come to the conclusion that D15 discloses a process which only foresees the following two options for feeding the chlorate and the reducing agent chloride to the reaction zone:

- (i) a feed of an existing mixed solution containing both reactants ("R-2 solution") or
- (ii) separate feeds ("split feeds") of chlorate and chloride ("brine").

The Board therefore holds that the indication in D15 that sodium chlorate and sodium chloride can be fed as mixed or separate solution(s) does not include the option of pre-mixing previously separate solutions of these reactants with the aim to dilute the reducing agent.

4.6.3 The respondent has particularly relied on the declaration of Ms Falgén (D2a) for showing that the pre-mixing of chlorate and the reducing agent is common in the art. It is thus stated in this document that "it is my experience that most of the commercial SVP generators for chlorine dioxide production installed before 1992 were, for practical reasons, designed for pre-mixing of the reducing agent (chloride ions or methanol) with the sodium chlorate feed. If such a generator is converted to operate according to the SVP-HP process described in the US patents 5,091,166 and 5,091,167, it is normally most economical not to change the existing process equipment but feed the hydrogen peroxide in the original pipe for reducing agent. The SVP-HP process will then inevitably involve pre-mixing



the hydrogen peroxide with the sodium chlorate to a combined feed stream" (see D2a, item 5).

The assertions in D2a are strongly contested by the appellant with the argument that they are not congruent with the information disseminated to the general public. The explanation given was that, historically, the SVP process was developed to process R-2 solutions. When chloride was later replaced by methanol as reducing agent, the latter was always fed separately since there was no existing mixed solution of methanol and chlorate. Not only has the respondent thus consistently advocated the separate feed of chlorate and reducing agent (methanol or hydrogen peroxide) in documents placed in the public domain.

The appellant has observed that, in fact, there is no document which is definitely known to be available to the public, be it published up to the priority date of the patent in suit or even later, up to the present date, which shows a deliberate pre-mixing of chlorate and reducing agent to form a feed to a SVP generator. The Board remarks that this statement of the appellant, which also applies to the process of D9, has not been challenged by the respondent. Concerning the content of D2a, it is noted that the author has not named any particular commercial plant known to her in which the reducing agent (chloride ions or methanol) is actually pre-mixed with sodium chlorate to form a feed stream for SVP generators, let alone produced any evidence to that effect. The Board therefore cannot accept this unsupported presumption, much less the consequential argument to the effect of pre-mixing of hydrogen peroxide with chlorate.

5. As corollary of the above, the Board holds that the modification as proposed in claim 1 is not suggested in any of the available prior art documents. The process according to claim 1 therefore involves an inventive step. The dependent claims 2 to 23 are directed to preferred embodiments of that process; their subject-matter is also new and involves an inventive step. The patent can thus be maintained with the claims of the present main request, after the necessary adaptation of the description.

## **Order**

### **For these reasons it is decided that:**

1. The decision under appeal is set aside.
2. The case is remitted to the first instance with the order to maintain the patent with the following documents:
  - claims 1 to 23 submitted at the oral proceedings
  - a description to be adapted.

The Registrar:

The Chairman:

U. Bultmann

R. Spangenberg