DECISION of 16 January 2003

Case Number: T 0444/00 - 3.3.5

Application Number: 93913716.2

Publication Number: 0644853

IPC: C01B 11/02

Language of the proceedings: EN

Title of invention: Method of producing chlorine dioxide

Patentee: Eka Chemicals AB

Opponent: Sterling Canada, Inc. OLIN CORPORATION

Headword: Chlorine dioxide/EKA

Relevant legal provisions: EPC Art. 123(2), 54, 56

Keyword: "Inventive step (amended claims, yes)"

Decisions cited: T 0219/83

Catchword: -
Case Number: T 0444/00 - 3.3.5

DECISION
of the Technical Board of Appeal 3.3.5
of 16 January 2003

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Decision under appeal: Decision of the Opposition Division of the European Patent Office posted 22 March 2000 revoking European patent No. 0 644 853 pursuant to Article 102(1) EPC.
Composition of the Board:

Chairman: R. K. Spangenberg
Members: M. M. Eberhard
J. H. Van Moer

Summary of Facts and Submissions

I. The appeal is from the decision of the opposition division revoking European patent No. 0 644 853. The patent was granted in response to European patent application No. 93 913 716.2. The decision was based on three sets of amended claims submitted on 15 February 2000.

II. During the opposition proceedings, opponents 1 and 2, now respondents 1 and 2, relied inter alia on the following documents:

D4: US-A-3 810 969


D7: WO-A-92/03374

D8: US-A-5 084 148


D17: Copy of Notice of opposition to EP-B- 514 427


In its decision the opposition division took the view that the subject-matter of claim 1 of the main request
lacked novelty over the disclosure of *inter alia* D5. The subject-matter of claim 1 of the 1st auxiliary request did not involve an inventive step in view of the disclosure of D4 and D5. The process of claim 2 of the 1st auxiliary request was obvious in view of the teaching of D7 and D5. The subject-matter of claim 1 of the 2nd auxiliary request was considered to lack an inventive step taking account of the teaching of D4 and D7.

III. The appellant (proprietor of the patent) filed a set of amended claims, as the main request, with the statement of grounds of appeal and additional comparative examples. Three sets of amended claims were submitted on 13 December 2002, as three auxiliary requests, as well as revised comparative examples. Oral proceedings took place on 16 January 2003. Respondent 2 had informed the board that he would not attend the hearing and was accordingly not represented. During the oral proceedings the appellant filed a set of claims as the main request in replacement of all the previous requests on file. Claim 1 of that request, which is identical to claim 1 of the 1st auxiliary request filed on 13 December 2002, reads as follows:

"1. A continuous method of producing chlorine dioxide in a single vessel generator-evaporator-crystalliser at subatmospheric pressure, characterised in that the method comprises supplying to a reaction medium sulfuric acid and a solution containing chloric acid and alkali metal chlorate, maintaining an acidity in said reaction medium of from 0.5 to below 5 N, and reacting said chloric acid and chlorate in said reaction medium with hydrogen peroxide as a reducing agent in such proportions that chlorine dioxide is produced and withdrawing a salt cake of alkali metal
sulfate, wherein the amount of $H^+$ in the reaction medium exceeds the amount of $H^+$ supplied with the sulfuric acid."

IV. The appellant requested that the decision under appeal be set aside and that the patent be maintained with claims 1 to 3 according to the main request filed on 16 January 2003. Respondent 1 requested that the appeal be dismissed. Respondent 2 did not present any request.

V. The appellant presented inter alia the following arguments:

The subject-matter of claim 1 was novel over the disclosure of D7. D7 could in no way be interpreted as unambiguously disclosing the supply of sulfuric acid to the reaction medium and no sulfate salt cake was withdrawn. D7 did not disclose using hydrogen peroxide as the reducing agent in the process thereof. D5 represented the closest prior art. The technical problem to be solved with respect to D5 was to provide a process of producing chorine dioxide with an improved production rate. The additional comparative examples showed that by replacing some of the alkali metal chlorate and sulfuric acid feeds with chloric acid in a continuous process for the production of chlorine dioxide and a salt cake of alkali metal sulfate such as the process of D5, the chlorine dioxide production rate could be significantly increased at the same acidity and temperature. None of D4, D7, D8 and D12 suggested that such an improvement of the production rate could be achieved by using chloric acid in the process of D5. The respondent's arguments that this advantage could be expected taking account of the known buffering effect of the sulfate ions could not be followed. In a chlorine dioxide generation process producing a salt
cake of alkali metal sulfate, the concentration of the
alkali metal sulfate in the reaction medium had
necessarily to be at its saturation in the steady
state. As in both runs of the comparative examples the
reaction medium was saturated in respect of sodium
sulfate, the buffering action of the sulfate ions would
be expected to be the same. D13 did not disclose a
kinetic model applicable to hydrogen peroxide at
acidities < 5 N, nor that chloric acid was the most
important factor for the production rate. It taught
that chloride ions were necessary, which was not in
agreement with the disclosure in D5. The respondent's
assumption that the production rate depended on the
free acidity at the reagents feed point to the
generator was neither common general knowledge, nor
supported by documents. The appellant believed that
this assumption was not correct since not only the
sulfate was diluted at the feed point, but also the
rest of the components.

VI. Respondent 1's arguments can be summarised as follows:

The incorporation of the term "chlorate" in claim 1 of
the main request contravened Article 123(2) EPC since
the original disclosure required complete conversion of
all the chlorate ions. The process of claim 1 lacked
novelty over the disclosure of D7. The supply of a
mineral acid to the reaction medium was disclosed on
page 5 and sulfuric acid was used in example 5 of D7.
Withdrawal of a salt cake was inherent to all SVP
processes and hydrogen peroxide was indicated at page 1
as a possible reducing agent. D5 was the closest prior
art. The only difference with respect to D5 was the
replacement of part of the chlorate and sulfuric acid
feeds by chloric acid. However, the equivalence of the
chloric acid formed in situ in mixtures as per D5 and
the externally produced chloric acid was obvious to any skilled person, including to the patentee as shown by D17. Therefore, it was obvious to combine the disclosure of D5 with the teaching of any of D4, D7, D8 or D12, in particular D8, which disclosed mixed feeds containing chloric acid and sulfuric acid and referred to the SVP process. The patent in suit was silent on the alleged increase of the production rate. This advantage, if obtained, was discovered during the opposition proceedings. The comparative examples included only a single experiment, which was not sufficient to show an advantage over the whole range of claim 1. Furthermore, the person who performed the comparative experiments had no exact re-collection of where the reagents were fed. If all the components were fed to the generator, then an increase of the production rate could not be achieved and would be contrary to the fundamentals of reaction kinetics. Assuming for the sake of argument that the advantage were obtained, then the increased production rate associated with the partial substitution of the sulfuric acid/sodium chlorate feed with the chloric acid feed was foreseeable. It was common general knowledge that chloric acid was the actual reagent involved in the chlorine dioxide generation step in all types of chlorate based chlorine dioxide generators and that the production rate depended on the acidity (see e.g. D13). The buffering action of the sulfate ions and the resulting effect on the free acidity and rate of production of chlorine dioxide were known for example from D7 and further confirmed by D18. It was irrelevant whether the concentration of sodium sulfate in the reaction medium was at its saturation since the rate of the chlorine dioxide generation reaction was governed by the acidity at the point where the reaction actually took place, i.e. at the reagents feed point to the
generator. At the point of feed of chloric acid in the re-circulation loop, the concentration of the alkali metal sulfate was no longer at saturation because of the dilution effect by the chloric acid.

**Reasons for the Decision**

1. The appeal is admissible.

2. Amended claim 1 meets the requirements of Articles 123(2) and (3) EPC. A continuous method of producing chlorine dioxide in a single vessel generator-evaporator-crystalliser at subatmospheric pressure, which comprises supplying sulfuric acid and a solution containing chloric acid and alkali metal chlorate to the reaction medium, maintaining the acidity of the reaction medium at values from 0.5 to below 5 N and withdrawing a salt cake of alkali metal sulfate, is disclosed in the PCT application as published: see page 6, line 24 to page 7, line 7; page 4, lines 17 to 30; original claim 8. The feature that the amount of H\(^+\) in the reaction medium exceeds the amount of H\(^+\) supplied by the sulfuric acid finds a support on page 4, lines 30 to 33, of the PCT application. Respondent 1 argued that the incorporation of the term "chlorate" in the step of reacting the chloric acid and chlorate with hydrogen peroxide contravened Article 123(2) EPC since the PCT application required complete conversion of all chlorate ions when a mineral acid such as sulfuric acid was added. These arguments cannot be followed by the board for the following reasons. On page 4 of the PCT application, it is disclosed that "at least in processes run under subatmospheric pressure, it is possible to increase the acidity by adding also a
mineral acid such as sulfuric acid, thus obtaining less of a salt cake by-product than in conventional processes without chloric acid. Mineral acids can be supplied in an amount sufficient for substantially all chlorate to react”. The first sentence thus disclosed the possibility of adding a mineral acid to the reaction medium, and the second sentence the possibility of adding an amount of acid sufficient for substantially all chlorate to react. It is not directly and unambiguously derivable from the second sentence that it is mandatory to use an amount of mineral acid sufficient for substantially all chlorate to react, since the terms "can be supplied" are stated in this sentence and these terms cannot be construed as meaning "must be supplied" in the context of the whole patent application. Respondent 1's affirmation that "can be" had to be interpreted as "must be" was not substantiated and the board finds no support in the PCT application for this construction. The features in dependent claims 2 and 3 are disclosed in original claims 3 and 4 and on page 7, lines 20 to 23 of the PCT application. The protection conferred by the present claims is clearly restricted with respect to that of the granted claims.

3. At the oral proceedings, respondent 1 maintained his objection of lack of novelty over the disclosure of D7. Respondent 1's arguments in this respect are not convincing for the following reasons. D7 discloses on page 5, lines 30 to 35, that the chloric acid is generally the sole acid species in the chlorine dioxide generating reaction medium and that it may be desirable in certain cases to blend the chloric acid with one or more additional acids to provide the desired total acid normality level up to a maximum of about 7 normal. It is not stated in this context that sulfuric acid is one
of the possible mineral acids. Respondent 1 referred to example 5 in which chloric acid was blended with sulfuric acid. However, in example 5 the reduction of chloric acid to form chlorine dioxide is not effected using methanol as the reducing agent according to the first embodiment of D7 (Figure 1), but it is performed electrolytically in accordance with the second embodiment of D7 (Figure 2) (see also page 6, lines 7 to 29). Furthermore, the total acid normality of the reaction medium is 6.0 N in example 5, ie it lies outside the range stated in present claim 1. Regarding the reducing agent, hydrogen peroxide, methanol and chloride ions are mentioned on page 1 of D7 as examples of reducing agents (see page 1, lines 10 to 22). In this context commercial processes are referred to in which the acidity for the process is provided by sulfuric acid while the chlorate ions are provided by sodium chlorate, and which lead to the formation of some form of sodium sulfate as a by-product. However, this paragraph does not concern the invention in D7 itself but the background of the invention. D7 contains no information from which the combination of the following features would be directly und unambiguously derivable: use of hydrogen peroxide as the reducing agent in combination with the supply of sulfuric acid and a solution containing chloric acid and alkali metal chlorate to the reaction medium. Therefore the claimed process is new with respect to D7. It is also novel over the disclosure of the remaining documents cited by the respondents. As this finding was not disputed, further considerations in this respect are not necessary.

4. Turning to the issue of inventive step, the board considers in agreement with the parties that D5 represents the closest prior art. D5 discloses a
continuous method of producing chlorine dioxide by reducing alkali metal chlorate with hydrogen peroxide in an aqueous reaction medium containing sulfuric acid. The process is performed in a single vessel generator-evaporator-crystalliser at subatmospheric pressure and includes the step of maintaining an acidity within the range from about 2 to about 5 N in the reaction medium, and withdrawing a salt cake of alkali metal sulfate. This process makes it possible to obtain a very high reaction rate and efficiency at low acid normalities without the aid of catalysts (see column 2, lines 47 to 56; column 3, lines 50 to 67; column 5, claim 1).

4.1 Starting from this closest prior art, the problem underlying the claimed process is, according to the appellant, to provide a continuous process for the production of chlorine dioxide which makes it possible to produce chlorine dioxide at an improved production rate.

It is proposed to solve this problem by the process as defined in claim 1, i.e., a process which differs from that of D5 in that the amount of H⁺ in the reaction medium exceeds the amount of H⁺ supplied by the sulfuric acid, a solution containing chloric acid and alkali metal chlorate being supplied to the reaction medium. In other words, chloric acid produced externally is supplied to the reaction medium.

The additional comparative examples submitted by the appellant at the appeal stage show that by replacing part of the sodium chlorate and sulfuric acid feed by chloric acid, the maximum steady state production rate of chlorine dioxide is substantially improved (from 1.4 to 1.8 tonnes ClO₂ per m³ reaction medium and 24 hrs), the acidity, temperature, and chlorate concentration
being similar in both runs. In view of these comparative examples, it is credible in the absence of evidence to the contrary that the problem of improving the production rate stated above has actually been solved by the claimed process.

4.2 Respondent 1 argued that the patent in suit was silent on the alleged increase in the production rate and that this advantage, if obtained, was discovered during the opposition proceedings. Although respondent 1 did not indicate what the legal consequence of this argument was, it is assumed that he objected against taking into account this advantage for the definition of the technical problem since it is allegedly not disclosed in the patent in suit. The board observes in this respect that the patent in suit expressly deals with the problem of increasing the production rate of chlorine dioxide. Reference is made to D5 in the paragraph bridging pages 2 and 3 of the patent in suit, and then on page 3, lines 3 to 9, it is said that "the reaction between chloric acid and hydrogen peroxide is very rapid even at acidities as low as 1-2 N. The reaction is considerably faster than the corresponding reaction in other systems for chlorine dioxide production such as the chloric acid - methanol system or the sodium chlorate - hydrogen peroxide - sulfuric acid system acid". The latter system is the one disclosed in D5. Thus, the patent in suit deals with the improvement of the production rate with respect to D5. As pointed out by respondent 1 chloric acid alone (and not a mixture of chloric acid and sodium chlorate) is considered in the passage on page 3, lines 3 to 5, of the patent in suit. However, on page 3, lines 16 to 18, it is stated that a preferred embodiment of the invention involves supplying the reaction medium with a solution containing chloric acid and alkali metal
chlorate. In these circumstances, there is no reason to consider that the improved production rate indicated in connection with chloric acid would not apply in the case of the said preferred embodiment. Furthermore, although the addition of sulfuric acid is not presented as a preferred embodiment in the patent in suit because of the formation of a sodium sulfate salt cake by-product (see page 3, lines 20 to 26), it cannot be inferred from the patent in suit that, in this case, no improvement of the production rate can be obtained although externally produced chloric acid is added. The comparative examples confirm that an improvement of the production rate is actually obtained with the sulfuric acid-externally produced chloric acid-sodium chlorate system. Under these circumstances, the board cannot accept respondent 1's arguments that the comparative examples show an advantage which was discovered first during the opposition proceedings. Therefore, the board sees no reason not to take this advantage into account for the formulation of the technical problem to be solved with respect to D5.

4.3 Respondent 1 contested for the first time at the oral proceedings that the comparative examples were sufficient to show an advantage over the whole range of claim 1. The comparative examples include only one run illustrating the process of D5 and one run according to the claimed process, these two runs being performed at a specific acidity, chlorate concentration and temperature. However, a single experiment can be sufficient to show an advantage over the whole range of a claim. This depends on the particular circumstances of the case. In the present case it is disclosed in the patent in suit that the reaction between chloric acid and hydrogen peroxide is very rapid even at as low acidities as 1-2N (see page 3, line 3) and the
comparative examples show a substantial improvement of the production rate at an acidity of about 3.1. Respondent 1 did not give any reason why an improvement would not be obtained at acidities other than the said values and falling within the claimed range of 0.5 to below 5N. He also provided no evidence showing that no improvement of the production rate would be obtained under different operating conditions falling within the claimed range, although the burden of proof lies on him regarding his allegation that an advantage would not be achieved over the whole range of claim 1. According to the appellant, the comparative examples were performed by feeding hydrogen peroxide and sulfuric acid separately into the chlorine dioxide generator, whereas the mixture of chloric acid and sodium chlorate was fed into the re-circulation loop. The appellant could not indicate at the oral proceedings where the separate feed points for the introduction of the hydrogen peroxide and the sulfuric acid into the generator were exactly situated. However, the board cannot conclude therefrom that the comparative examples are meaningless or do not support the improvement of production rate disclosed in the patent in suit. The exact locations of the feed points of the sulfuric acid and hydrogen peroxide are neither stated in claim 1 nor in the patent in suit, and it cannot be inferred from the latter that these features would be essential for the obtaining of the said improvement. Furthermore, both runs of the comparative examples were carried out using the same position for the feed points of the reagents, and the board has no reason to assume that the improvement of the production rate would be achieved only with certain locations of the reagents feed points, ie a feature not mentioned in the patent in suit. Here again, as respondent 1 has himself contested the achievement of the improved production rate over
the whole extent of claim 1, the burden of proof rests with him that the locations of the reagents feed points greatly influence the results as regards the production rate.

It follows from the above that respondent 1's allegation that the problem of improving the production rate is not solved over the whole extent of claim 1 cannot be accepted.

5. D5 itself does not contain information pointing towards the claimed solution of the stated technical problem. Respondent 1 argued that, in view of the teaching of D8, the skilled person would have arrived in an obvious manner at the claimed process by combining the teachings of D8 and D5. These arguments are not convincing for the following reasons. D8 discloses a process for electrolytically producing aqueous solutions of chloric acid and alkali metal chlorate in an electrolytic cell, and solutions having molar ratios of chloric acid/alkali metal chlorate of 0.3:1 to 200:1, preferably 1:1 to 100:1, which are suitable for use in the generation of chlorine dioxide. According to D8 these solutions are highly acidic and permit a reduction in the amount of acid required in the generation of chlorine dioxide in commercial processes which react a chlorate solution with an acid in the presence of a reducing agent. Reference is made to typical commercial processes using sulfuric acid or hydrochloric acid with a reducing agent such as sulfur dioxide or methanol in the presence of a salt such as sodium chloride. The Mathieson, Solvay, R2, R3, R8, Kesting, SVP, SVP/methanol are in particular cited (see claim 1, column 7, lines 5 to 18 and 32 to 42). D8 does not make reference to processes using hydrogen peroxide as the reducing agent. Furthermore, it does not deal
with the problem of improving the production rate of chlorine dioxide in the commercial processes explicitly mentioned therein, let alone in a sodium chlorate-hydrogen peroxide-sulfuric acid system of the kind disclosed in D5. In these circumstances, the skilled person confronted with the problem stated above would not have been encouraged, in view of the teaching of D8, to use the chloric acid and alkali metal chlorate containing solutions of D8 in the process of D5 since he could not have expected that this might lead to an improvement of the production rate.

5.1 D7 also does not contain information from which the skilled person could have inferred that, by using the solutions of D7 containing both externally produced chloric acid and sodium chlorate in the process of D5, the production rate of chlorine dioxide might be improved. Therefore, the skilled person would not have contemplated combining the teaching of D7 and D5 in order to solve the problem stated above. Regarding D12, it is not clear whether or not the method for the production of chloric acid disclosed therein leads to a solution of chloric acid still containing some alkali metal chlorate. Even in the affirmative, the preceding reasoning regarding the combination of D5 with D8, or D5 with D7 would apply likewise to the combination of D5 and D12 since D12 does not deal with the problem of improving the production rate of chlorine dioxide. It only mentions that the chloric acid formed according to the process of D12 is useful in the generation of chlorine dioxide in processes which do not produce a by-product salt of lower value (see column 3, lines 16 to 20).

5.2 D4 discloses a process for the production of chlorine dioxide in which an aqueous solution containing from
0.2 to about 11 moles/l of alkali metal chlorate is first reacted with a cation exchange resin to produce an aqueous solution containing from about 0.2 to about 4 moles of chloric acid/l. This solution is then reacted with a reducing agent to produce chlorine dioxide (see claim 1 of D4). Hydrogen peroxide is not cited in the list of conventional reducing agents given in column 3, lines 27 to 30. As this document neither discloses nor suggests that the use of the chloric acid containing solution disclosed therein leads to an improvement of the production rate of chlorine dioxide, the skilled person would not have been induced to combine the teaching of D4 with that of D5 to solve the problem of improving the production rate of chlorine dioxide in the process of D5.

5.3 In D17, it is indicated in a different context that "there is no technical prejudice against using chloric acid also containing alkali metal chlorate for production of chlorine dioxide, particularly since both chloric acid and alkali metal chlorate in aqueous solutions are fully dissociated in its ions" (see page 4, 2nd paragraph). However, it cannot be inferred from the fact that no technical prejudice exists against the said use that the skilled person confronted with the problem stated above would have combined the teaching of D5 with any one of D4, D7, D8 and D12 since, as already indicated above, none of them suggests that the combination might lead to an improvement of the production rate, and thus might solve the existing technical problem.

5.4 Respondent 1's arguments that the increased production rate would have been expected by the skilled person in view of the general knowledge concerning the influence of the free acidity on the production rate and the
buffering effect of the sulfate ions (see e.g. D13, D7 and D18) are not convincing. According to D13, if the hypothesis made in this document about the reaction mechanism is correct, then the rate of production of chlorine dioxide is dependent on the chlorate concentration, the chloride concentration, the acidity, and the temperature, and depends on the reducing agent concentration to the extent that this affects the hypochlorite and chloride concentrations (see page 556, right-hand column, point 7 referred to by respondent 1). Thus, the acidity is one of the parameters having an influence on the production rate, among others. The appellant did not contest at the oral proceedings that it was well-known (i) that the production rate increases with the free acidity of the reaction medium and (ii) that sulfate ions tend to buffer hydrogen ions in the solution (see in connection with the said buffering effect D7, page 18, line 30; D18, page 1454, right-hand column, lines 9 to 12 of the third paragraph concerning a publication of 1969). However, a salt cake of alkali metal sulfate is withdrawn from the single reaction vessel both in the continuous process of D5 and in the claimed process. As pointed out by the appellant, this means that the concentration of sodium sulfate in the reaction medium must be at saturation at a given acidity, chlorate concentration and temperature, and that the buffering action of the sulfate ions would be expected to be the same in both cases. Respondent 1 did not dispute these arguments but considered them as being of no relevance. He argued in this respect that the rate of production of chlorine dioxide was in fact governed by the free acidity at the point where the chlorine dioxide generation reaction actually took place, i.e. at the reagents feed point to the generator, and submitted that the concentration of the alkali metal sulfate was no longer at saturation at
the feed point of chloric acid because of the dilution effect of the chloric acid (HClO₃, 10% was used in the comparative examples) in the re-circulation loop. However, the appellant expressed doubts about the correctness of respondent 1's allegation concerning the said influence of the free acidity at the reagents feed point and pointed out that a dilution effect would apply to all components. The board observes that the contested allegation is not supported by any documents or evidence, and, as admitted by respondent 1 himself, it did not form part of the common general knowledge at the priority date. Furthermore, it is neither self-evident to the skilled person nor obvious that the rate of production of chlorine dioxide in a continuous process of the kind described in D5 would be governed by the free acidity at the reagents feed point to the generator. The fact that respondent 1, even on the basis of an analysis with hindsight, cannot find any scientific explanation other than the influence of the free acidity at the reagents feed point for the improvement of the production rate does not prove that this allegation was self-evident to the skilled person faced with the problem stated above, and that he would have combined the teaching of D5 and D7 with the expectation of improving the production rate. Under these circumstances, and further considering that the burden of proof rests on respondent 1 for the said allegation (see T 219/83, OJ EPO 1986, 211, point 12 of the reasons), the board cannot be persuaded by it in the absence of evidence. As the respondent's conclusion that the improvement of the production rate would have been anticipated by the skilled person is based on this allegation, it can also not be followed by the board.

5.5 At the oral proceedings respondent 1 did not rely on the remaining documents on file published before the
priority date. Respondent 2 did not present any comment at the appeal stage. The board is also of the opinion that these documents do not contain further information which, alone or in combination with the documents considered above, would point towards the claimed process.

5.6 It follows from the above that the subject-matter of claim 1 also meets the requirement of inventive step set out in Articles 52(1) and 56 EPC.

6. Claim 1 being allowable, the same applies to dependent claims 2 and 3, whose patentability is supported by that of claim 1.
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 3 submitted during the oral proceedings

   - a description to be adapted.

The Registrar: The Chairman:

U. Bultmann R. Spangenberg