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**D E C I S I O N**  
**of 25 August 2005**

**Case Number:** T 0375/02 - 3.3.05

**Application Number:** 94200268.4

**Publication Number:** 0612686

**IPC:** C01B 11/02

**Language of the proceedings:** EN

**Title of invention:**

A process for continuously producing chlorine dioxide

**Patentee:**

Eka Chemicals AB

**Opponents:**

- 1) Arkema
- 2) Superior Plus Inc.

**Headword:**

Chlorine dioxide/EKA

**Relevant legal provisions:**

EPC Art. 123 (2)(3), 84, 54 (3), 56

**Keyword:**

"Inventive step: no (all requests)"

**Decisions cited:**

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**Catchword:**

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Case Number: T 0375/02 - 3.3.05

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.05  
of 25 August 2005

**Appellant:** Eka Chemicals AG  
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**Decision under appeal:** Decision of the Opposition Division of the  
European Patent Office posted 18 February 2002  
revoking European patent No. 0612686 pursuant  
to Article 102(1) EPC.

**Composition of the Board:**

**Chairman:** M. Eberhard  
**Members:** B. Czech  
J. Willems

## Summary of Facts and Submissions

I. The appeal is from the decision of the opposition division revoking the European patent No. 0 612 686. The decision was based on the amended claims submitted on 17 January 2002 as a main request.

II. The documents cited in the opposition proceedings include the following:

A1/S9: JP-A-3-115 102 (translation into English)

A6/S12: The Bleaching of Pulp, Third Edition, 1979, Edited by R.P. Singh, TAPPI PRESS; pages 628 to 639

S2: US-A-5 091 167

S8: JP-A-63-8203 (translation into English)

S11: "Chlorine dioxide Generation Systems"  
(Japan Carlit)

S18: EP-B1-0 636 106

III. In the contested decision, the opposition division inter alia held that the subject-matter of amended claim 1 according to the main request was obvious in view of the combined teachings of S8 and A1.

IV. With its statement of grounds of appeal, the appellant submitted two fresh sets of amended claims as main and (first) auxiliary requests and contested the reasons given in the decision of the opposition division

concerning inventive step. Referring also to documents S11 and D1 (US-A-5 366 174), it argued that A1 could not induce the skilled person to modify the process of S8 so as to arrive at the claimed process, which was thus novel and inventive.

Claim 1 according to the main request reads as follows:

*"1. A process for continuously producing chlorine dioxide by reacting an alkali metal chlorate, sulfuric acid or another chlorine free mineral acid and hydrogen peroxide as reducing agent to produce chlorine dioxide in an aqueous reaction medium and withdrawing chlorine dioxide and oxygen, wherein the chlorine dioxide is generated in at least one reaction step carried out in two or more reaction vessels in the substantial absence of added chloride ions by feeding to a first reaction vessel alkali metal chlorate that contains not more than 0.05 weight percent of chloride, acid, and hydrogen peroxide, maintaining the reaction medium at a temperature from about 35°C to about 100°C and at an acidity within a range from about 4 to about 14 N and maintaining a chlorate concentration of between about 0.09 moles/l to saturation, maintaining the aqueous reaction medium in said reaction vessel at a pressure of from about 400 mm Hg (about 53 kPa) to about 900 mm Hg (about 120 kPa) and feeding inert gas to said reaction vessel, withdrawing inert gas and depleted aqueous reaction medium from said reaction zone without substantial crystallisation of alkali metal sulfate, wherein the depleted aqueous reaction medium from the first reaction vessel is brought to at least a second reaction vessel for further reaction with addition of hydrogen peroxide."*

Claim 1 of the (first) auxiliary request differs from claim 1 of the main request in that, after the phrase "*wherein the depleted aqueous reaction medium from the first reaction vessel is brought to at least a second reaction vessel*", it reads as follows:

***"with addition of inert gas and hydrogen peroxide, wherein more chlorate in the depleted medium is converted to chlorine dioxide and whereby the pressure in the second reactor is maintained at about atmospheric pressure and the temperature is the same as in the first reaction vessel."***

- V. In its reply, respondent 1 maintained that the claimed subject-matter lacked an inventive step in view of S8 and A1. It also submitted a sheet with calculations.
- VI. Respondent 2 considered the appeal to be inadmissible in view of the requests submitted by the appellant. It raised objections under Articles 84, 123 (2) and (3) EPC against the claims according to both of the appellant's requests. It also argued that the claimed subject matter lacked novelty over document S18 in view of S12. Referring to documents S8, A1/S9, S11 and D1, respondent 2 argued that the claimed subject-matter also lacked an inventive step. He filed another translation of S8 (to the contents of which the present decision refers when discussing S8) and two further documents.
- VII. In its last written submission dated 25 July 2005, the appellant rejected the objections raised by the respondents and also referred to document A6/S12. With

the same letter, it filed two further sets of amended claims as 2nd and 3rd auxiliary request, respectively.

Claim 1 of the 2nd auxiliary request differs from claim 1 of the (first) auxiliary request by the replacement of the word "reactor" by "**reaction vessel**"

Claim 1 of the 3rd auxiliary request differs from claim 1 of the 2nd auxiliary request

- by the insertion, after the expression "*in the substantial absence of added chloride ions*", of the feature "**the amount of chloride added being not more than about 0.05 weight percent of the alkali metal chlorate**";
- and by the insertion, after the phrase "... *chlorate in the depleted medium is converted to chlorine dioxide*", of the feature "**and whereby the amount of added hydrogen peroxide is up to 50% of the entire hydrogen peroxide required for the reaction**"

VIII. In the annex to the summons to oral proceedings, the board *inter alia* commented on the issue of the admissibility of the appeal. It also noted that S18 was a European patent published after the priority and filing dates of the patent in suit and identified the corresponding European application S18' (EP-A-0 636 106), which refers to the corresponding published PCT application S18'': WO-A-93/21105.

- IX. With its last written submission dated 28 July 2005, respondent 2 submitted copies of the two US applications from which S18' claims priority.
- X. Oral proceedings took place on 25 August 2005. During these oral proceedings, respondent 2 explicitly dropped its objection concerning the admissibility of the appeal.
- XI. The appellant requested - as main request - that the decision under appeal be set aside and the patent be maintained on the basis of the set of claims according to the main request filed with letter dated 13 June 2002, or, in the alternative, on the basis of one of the sets of claims filed
- as auxiliary request by letter of 13 June 2002,
  - as 2nd auxiliary requests by letter of 25 July 2005, or
  - as 3rd auxiliary requests by letter of 25 July 2005.

The respondents both requested that the appeal be dismissed.

- XII. The essential arguments of the parties can be summarised as follows:

*Concerning the main request*

The appellant was of the opinion that the amendments to claim 1 were of a clarifying and restricting nature and

were based on the content of the application as filed. The description was not in contradiction with the claims. The description provided a clear basis for the interpretation of the expression "*substantial absence of added chloride ions*" and the maximum level of chloride was now defined in claim 1. In the appellant's view, a non-crystallising process combining all the features of claim 1 was not clearly and unambiguously disclosed in S18', despite the reference to Mathieson reactors. Only single vessel processes were explicitly described. S18' was thus not novelty destroying. At the oral proceedings, the appellant accepted that S8 could be considered as the closest prior art. One element of the teaching of S8 was to keep a certain chloride ion concentration in the reaction mixture. This was achieved by adding 0.5 to 10 mole-% sodium chloride to the chlorate fed to the reactor, i.e. a relative amount of chloride much higher than that prescribed by present claim 1. Starting from the process of S8 the problem to be solved could be seen in further lowering the chlorine content in the chlorine dioxide produced. Trying to lower the chlorine content by lowering the amount of chloride added would go against the teaching of S8. A1 related to a different process relying mainly on the use of methanol as reducing agent. A1 had to be read in the light of what was generally accepted at the time the invention was made, namely that the presence of a significant amount of chloride ions was necessary to run the reaction using hydrogen peroxide as the reducing agent, as illustrated e.g. by S8 itself, A6 and S11 (R2P process). In the Solvay process based on the use of methanol, chloride was also added. Referring also to D1, it emphasised that commercial alkali chlorate always contained some chloride. Hence, when A1



spoke about "no chloride", this could thus not mean "zero" chloride, and there was no clear recommendation in A1 to operate the process with chlorate that contains very little, i.e. less than 0.05 percent chloride. The term "reduced" used to describe the amount of by-product chlorine was merely of a relative nature. Generally speaking, it was not possible to obtain a chlorine dioxide product containing no chlorine at all without a subsequent scrubbing step as in the R2P process referred to in S11. Moreover, the unclear reference, in comparative example 1 of A1, to "*chlorine ions as a medium*" (page 12) could be considered as a pointer to the presence of chloride or chlorine in the reaction with hydrogen peroxide. Interpreted in a proper manner, A1 thus did not give the skilled person an incentive to go against the clear teaching of S8 having regard to the chloride concentration to thereby solve the stated problem whilst still keeping the process efficient. Document S2 related to a process involving reaction at reduced pressure in a single vessel with crystallisation. It was not obvious from S2 that it was even possible to produce chlorine dioxide in a non-crystallising process in the absence of significant amounts of chloride ions. S2 could thus not motivate the skilled person to go against the clear teaching of S8, which related to a different process. Moreover, in the process of S2 there was no liquid effluent. Hence, the skilled person knew that under steady state conditions, even the use of chlorate containing very little chloride would lead to an accumulation of chloride to higher levels in the reaction medium. The appellant also argued that the claimed process was a commercial success. Had the

authors of S2 thought that such a process was possible, they would have disclosed and claimed it.

The respondents maintained objections under Articles 123(2) and (2) and 84 EPC. Respondent 2 held that the claimed process lacked novelty in view of the "overall teaching" of S18'. Referring to the disclosures of A1 and S2, both respondents contested that a prejudice still existed upon filing of the patent in suit with respect to the necessity of using chloride. In their view, A1 gave a clear teaching to avoid the presence of chloride in order to lower the amount of chlorine by-product. Since chlorates with very low chloride contents were known and available, the skilled person, starting from S8 as closest prior art, would consider using them as an obvious measure. Respondent 2 also argued that the claimed process lacked an inventive step in view of a combination of S8 with S2, the latter showing the possibility of carrying out the reaction using chlorates with a very low chloride content. It emphasised that S2 did not mention any accumulation of chloride, and that no evidence demonstrating such an accumulation had been provided by the appellant. It argued that even if an accumulation of chloride occurred, the chloride would react to chlorine according to the known side-reaction and would then leave the reactor together with the chlorine dioxide formed.

*Concerning the 1st and 2nd auxiliary requests*

The appellant considered the respective claims 1 of these two requests to be based on the application as filed. Processes with all the features of these

claims 1 were not disclosed by S18'. The amendments provided additional distinctions versus the disclosure of document S8. S8 and S2 were silent about the temperature and pressure conditions prevailing in a second reaction vessel, whereas documents A1 and A6 suggested using higher temperatures in the second reaction vessel. Operating at the same temperature in both vessels meant that it was not necessary to heat the second vessel and that no extra heating means were required for this purpose. The reaction being exothermic, turning off the cooling would normally suffice to keep the temperature at the same level in the second reactor vessel. The claimed process was advantageous in that it was simple and energy saving. However, to arrive at the claimed process, the skilled person had to go against the teachings of A1 and A6. Since the prior art gave no incentive to operate the two vessels at the same temperature, the claimed process was non-obvious even in the absence of an improvement attributable to the said additional features.

The respondents upheld the objections under Articles 123 and 84 EPC as already raised with respect to the main request. Respondent 2 also considered claim 1 according to the 1st auxiliary request to lack clarity since the expression "*the second reactor*" had no antecedent. Respondent 2 considered the method according to the respective claims 1 of these two requests to also lack novelty over S18'. It argued that carrying out the reaction in a Mathieson reactor inherently meant that both vessels were operated at about atmospheric pressure and at the same temperature. According to respondent 1, the operating conditions

would be the same in the two reactors mentioned in S8, except for the chlorate concentration. Concerning the latter, it was indicated that the disclosed value was the one in the first reactor. In the absence of any further indications concerning differing operating conditions, i.e. temperature and pressure in the two reactors, the skilled person would thus understand that they were the same. Moreover, no technical effect could be attributed to these additional features of claim 1. Respondent 2 argued that S8 related to a process run at atmospheric pressure in both vessels, and that the indications given in S8 (page 6, second paragraph) concerning the keeping of the reaction solution at a certain temperature applied to both vessels. It also pointed out that since the patent in suit expressly referred to the heating of the second reactor to maintain the temperature there was no simplification as alleged by the appellant. Running the process described in S8 in two cascaded vessels at atmospheric pressure and at the same controlled temperature was the simplest thing to do and therefore obvious. Since A1 and A6 both concerned processes differing in terms of the reducing agents and thus had different optimum temperatures they did not suggest departing from what was generally disclosed in S8.

*Concerning the 3rd auxiliary request*

According to the appellant claim 1 had been reformulated in response to objections concerning the feature "*substantial absence of added chloride ions*". The passage on page 9, lines 14 to 20, of the application as filed formed a basis for this amendment. In view of this passage, it was clear that the only

chloride added was the one present in the chlorate as an impurity. He considered that the wording of present claim 1 was not in contradiction with the said passage. Moreover, it did not agree to discuss the objection under Article 123(2) EPC against present claim 1, the wording of which, as far as "*added chloride ions*" were concerned, stemmed from claims 1 and 8 as granted. It considered this objection to be based on a new ground of opposition. Concerning inventive step, it merely stated at the oral proceedings that there was no teaching in the prior art to perform the process as presently claimed, and that it did not have any further comments to add.

Respondent 1 argued that present claim 1 was not a simple combination of claims 1 and 8 as granted and lacked clarity with respect to the meaning of the expression "*... in the substantial absence of added chloride ions, the amount of chloride ions added being ...*". Respondent 2 additionally raised an objection under Article 123(2) EPC, arguing that the particular wording used in claim 8 as granted and now being present in claim 1 had no basis in the application as originally filed. The respondents did not object to the novelty of the claimed process. However, they considered that everything said in connection with claim 1 according to the preceding requests also applied in the case of the present request. The additional feature concerning the amount of hydrogen peroxide to be added to the second vessel was also disclosed in S8 and could thus not make the claimed process inventive.

## Reasons for the Decision

1. The appeal is admissible.

### *Main Request*

2. *Allowability of the amendments*

- 2.1 A chlorate concentration of at least 0.09 moles/l is disclosed on page 8, lines 14 and 15 and in claim 5 of the application as filed. The possibility of carrying out a reaction step in two or more reaction vessels is addressed in the application as filed on page 6, lines 15 to 24 and finds further support in claim 1 and dependent claim 8 thereof. Said claim 8, which also refers back to claim 5, did not contain further ranges concerning the conditions prevailing in the two reaction vessels. In the board's view, since both vessels mentioned in said claim 8 belong to the same reaction step, the reaction conditions, i.e. the pressure, temperature, acidity, and chlorate concentration ranges recited in claims 1 and 5 of the application as filed implicitly apply to both of them. However, claim 8 of the application as filed was not formulated so as to prescribe that the actual temperature, pressure and acidity values, while lying within the ranges of claim 1, had to be the same in both reactors. In the case of the chlorate concentration, said claim 8 even indicated that the solution leaving the first vessel and being fed to the second vessel was depleted in chlorate, and was thus implicitly of a lower concentration. Concerning the conditions prevailing in the two vessels belonging to the reaction step, and in particular the conditions

prevailing in the second vessel, if present, it is true that more specific reaction conditions are mentioned in the application as filed and in the patent in suit than in present claim 1 (see the detailed description of the first reaction step in column 6, line 18 to column 7, line 3, and column 7, lines 28 to 38 of the patent in suit). However, this fact does not as such make the present claim objectionable under Article 123(2) or (3) EPC. The amendments in question therefore meet the requirements of Article 123(2) and (3) EPC.

2.2 On page 9, lines 9 to 13 of the application as filed, it is indicated that no substantial amount of chloride ions is added since their presence had a detrimental influence on the process. The feature "*chlorate that contains not more than about 0.05 weight percent chloride*" incorporated into present claim 1 restricts the claim and finds a basis in the next sentence on page 9, lines 13 to 18, of the application as filed. This amendment thus also complies with the requirements of Article 123(2) and (3) EPC.

2.3 The board is also satisfied that the other minor amendments carried out in claim 1 according to the main request comply with the requirements of Article 123(2) and (3) EPC.

3. *Clarity of amended claim 1 - "added chloride"*

3.1 The expression "*in the substantial absence of added chloride ions*" was already present in claim 1 of the patent as granted (and in claim 1 of the application as filed). Although it does not impose a precise upper limit on the amount ("*substantially no ...*") or origin

("added") of any chloride ions that may be present during the reaction, it does not absolutely exclude the presence of chloride ions. Having regard to the amount and origin of chloride that may be present according to the said relative expression claim 1 is thus to be construed as far as necessary.

3.2 The present amended claim 1 additionally specifies that the alkali metal chlorate fed to the first reaction vessel "*contains not more than about 0.05 weight percent of chloride*". Feeding such a chloride containing chlorate to the reactor implies that the reaction is carried out not necessarily in the total absence but in the presence of only a small amount of chloride. Whether the quoted feature is in contradiction with the requirement of a "*substantial absence of added chloride ions*" initially present in claim 1, and whether a lack of clarity thus arises from the amendment, i.e. the incorporation of the additional feature, depends on the meaning to be given to the expression "*substantial absence of added chloride ions*".

3.2.1 In one and the same paragraph (column 7, 2<sup>nd</sup> paragraph) of the granted patent, it is indicated that "*no substantial amount of chloride ions is added*", that by way of its manufacturing, the chlorate used in the reaction "*always contains a small amount of chloride*", this amount being "*not more than about 0.5, often not more than about 0.05, preferably not more than about 0.02, most preferably not more than about 0.01 weight per cent of the alkali metal chlorate*", and that "*beside this amount of chloride being an impurity of the chlorate, no further chloride is added*".



Considering these indications, it is apparent that the expression "*substantial absence of added chloride ions*" was and is supposed to also cover embodiments wherein the chlorate fed to the reaction vessel contains a small amount of chloride as an impurity, e.g. up to about 0.5 weight percent.

3.2.2 In particular in view of the indications in the description, the additional specification of an upper limit of about 0.05 weight-% for the chloride content of the chlorate is not in contradiction with the remainder of the claim but amounts to a further limitation of the claim, as already mentioned under point 2.2. above). This amendment does not, therefore, give rise to a clarity objection in connection with the expression that was already present in claim 1.

#### 4. *Novelty*

4.1 The board has no reason to question that the disclosure of S18', the sole document cited against novelty at the oral proceedings, belongs to the state of the art pursuant to Article 54(3) and (4) EPC for the designated states FR, PT and SE. This was not in dispute. S18' is identical in content with S18'', from which the passages quoted in this decision are taken.

4.2 S18' generally relates to processes for the production of chlorine dioxide by reacting chloric acid and/or an alkali metal chlorate with hydrogen peroxide at temperatures of from 30 to 100°C and at acidities of from 0.5 to about 12 N, wherein a compound selected from urea, a phosphonic acid based complexing agent, or mixtures thereof is added to the reaction medium to

increase the chlorine dioxide formation rate, see claim 1 and page 2, line 26 to page 3, line 16. The following is also stated on page 5, 2<sup>nd</sup> paragraph: *"Therefore the present invention can be used with all known chlorine dioxide processes using hydrogen peroxide as the reducing agent. Processes run at a normal pressure as well as reduced pressure processes are equally suitable. The invention can be used in batch processes or in continuous processes. All known types of reactors can be used e.g. SVP<sup>(R)</sup> reactors or Mathieson reactors and others. The chlorine dioxide production is run in a conventional manner."* Claim 8, dependent on claim 1 only, mentions a minimum chlorate concentration of 0.25M. Claim 9, dependent on claim 1 only, refers to the use of *"conventional alkali metal chlorate without extra added alkali metal chloride"*. *"Conventional, commercially available chlorate (without extra added alkali metal chloride)"* is stated to normally contain not more than about 0.5, often not more than about 0.05, weight percent alkali metal chloride. However, it is also indicated in S18' that additional alkali metal chloride can be added to obtain a chloride concentration of 0.001 to 0.08 moles per litre in the reactor, see page 6, lines 6 to 21.

- 4.3 S18' does not expressly mention a two vessel process, let alone a process with all the features of present claim 1. In its attempt to establish a lack of novelty, respondent 2 combined several elements of information taken from different parts of the document (i.e. from claims 1, 8 and 9, from the more general second paragraph on page 5, and from the description of a quite different specific embodiment extending from page 5, line 11 to page 6, line 21). Moreover,

concerning allegedly implicit features of the use of "Mathieson reactors" mentioned on page 5, line 9, it also relied on common general knowledge.

- 4.4 The board strongly doubts that the skilled person can clearly and unambiguously derive from S18' a process specifically combining all the features of claim 1 of the patent in suit. However, since the process of present claim 1 lacks the required inventive step in view of the other prior art cited, the issue of novelty over S18' need not be dealt with in more detail and can be left open.

5. *Inventive step*

5.1 Document S8 - Closest prior art

- 5.1.1 S8 discloses a non-crystallising process for the continuous production of highly pure chlorine dioxide (i.e. with a low chlorine content) gas by reduction of alkali metal chlorate with hydrogen peroxide in an aqueous sulphuric acid solution in the presence of chloride ions (see the claim on page 1). It remained undisputed at the oral proceedings that the feature distinguishing the claimed process from the disclosure of S8 was the chloride concentration of the chlorate fed to the reactor (see S8, page 7, lines 16 to 19). The board also accepts that the other features of claim 1 are explicitly or implicitly disclosed in S8, see the claim on page 1, page 6, second sentence, page 7, the last two sentences, and page 8, example 1.

- 5.1.2 As a way of keeping the chloride content in the reaction medium within a required concentration range,

S8 suggests "*adding 0.5 to 10 mol-% of sodium chloride to the chlorate*", see page 7, lines 16 to 19. As also acknowledged by the appellant during the oral proceedings, the indicated minimum value of 0.5 mol-% corresponds to sodium chlorate containing about 0.276 weight percent sodium chloride. The chlorate to be used according to S8 thus contains significantly more chloride than permitted in present claim 1 ("*not more than about 0.05 weight percent of chloride*").

5.1.3 Considering the similarities of the process of S8 and of the process claimed, and considering that S8 also aims at "*preparing highly pure chlorine dioxide without byproducing chlorine*" in an efficient manner and without requiring any complicated reactor (see page 3, 3<sup>rd</sup> paragraph, and page 12, last paragraph), the board can also accept that S8 represents the closest prior art.

## 5.2 Technical problem

5.2.1 In accordance with the submission of the appellant at the oral proceedings, the technical problem starting from a process according to S8 can be seen in the provision of a further efficient process for the production of chlorine dioxide, but which leads to a chlorine dioxide product containing even less chlorine as a by-product, see also column 3, lines 4 to 8 of the patent in suit.

5.3 In the process according to present claim 1, the maximum chloride content of the sodium chlorate fed to the reactor is much lower than foreseen by S8. For a given sodium chlorate concentration, the mixture in the

- reactor will therefore contain less chloride ions available for the unwanted reactions leading to the formation of chlorine. Therefore, it is plausible that for a given set of reaction conditions, the claimed process leads to the production of chlorine dioxide containing less chlorine than in the case of S8. The additional examples submitted by the appellant with letter dated 16 September 1999 appear to confirm that the claimed solution is effective in solving the stated technical problem: compare the respective values for the NaCl concentration and the purity of the product ("GAE") in Trial Nos. 1 to 3.
6. What remains to be seen is whether the claimed solution of the stated problem was obvious in view of the cited prior art. In particular it remains to be seen whether it was obvious to modify the process of S8 by using a chlorate of much lower chloride content than prescribed by S8 as the only source of chloride in the reaction mixture.
  7. Document S2 relates to another process for the production of chlorine dioxide by reducing alkali metal chlorate with hydrogen peroxide in an aqueous reaction medium containing sulphuric acid, which process "*is carried out in the substantial absence of added chloride ions*" in a single reaction vessel, see claim 1, and in particular the last two lines thereof. In the description of S2 (see column 3, lines 43 to 57), the following statement can be found: "*The present process is an essentially chlorine free process. No substantial amount of chloride ions are added. The chlorate used in the process is conventional, commercially available, chlorate. By way of manufacturing such chlorate always*

contains a small amount of chloride. That amount of chloride is not more than about 0.5, often not more than about 0.05, preferably not more than about 0.02, most preferably not more than about 0.01 weight percent alkali metal chloride. Beside this amount of chloride being an impurity in the chlorate no further chloride is added. There is also commercially available chlorate with higher amounts of chloride. That chlorate has been obtained by adding extra alkali metal chloride to the chlorate. Such a chlorate is not suitable for the present process." According to the sole example of S2, "0.15 g/h NaCl was also added together with the chlorate solution (emanating from chloride impurity of the conventional chlorate)". It was not disputed that the conventional NaClO<sub>3</sub>, fed (379 g/h) to the reactor according to this example thus contained roughly 0.04 weight percent sodium alkali chloride, which chloride content is below the upper limit of not more than 0.05 weight percent prescribed by present claim 1.

- 7.1 The authors of S2 were fully aware of the process of S8 (see S2, column 2, line 64 to column 3, line 21). They emphasise that, in contrast to the teaching of S8, the process of S2 permits the production of chlorine dioxide "without any substantial addition of chloride ions, thereby obtaining an essentially chlorine free process", see column 3, lines 21 to 24. Although a high efficiency and high production rate are achieved, "wherein little or no chlorine is formed as a by-product", see column 2, lines 32 to 47, the process of S2 requires no deliberate addition of chloride ions other than the ones contained as impurities in the commercially available chlorate starting material (see also claim 7). Chlorates containing an amount of not

- more than about 0.05, preferably not more than about 0.01 weight-% chloride as impurity are preferred.
- 7.2 As emphasised by the appellant, the continuous SVP<sup>(R)</sup> (single vessel process) technology of S2, i.e. the use of a single "generator-evaporator-crystalliser" reaction vessel, differs from the process according to present claim 1 in that it is carried out in a single reaction vessel under sub-atmospheric pressure of suitably 60 up to 400 mm Hg at the boiling point of the reaction medium. Water evaporates and the alkali metal salt of the mineral acid crystallises and is removed from the reaction vessel, see claim 1; column 1, lines 5 to 14; and column 3, line 58 to column 4, line 7.
- 7.3 Considering that S2 aims at providing a process of high efficiency and high production rate wherein little or no chlorine is formed as by-product (column 2, lines 33 to 36), the skilled person confronted with the stated technical problem would have taken S2 into consideration despite the differences mentioned. Irrespective of the said differences, it can be unambiguously gathered from S2 that chlorine dioxide containing only very small amounts of chlorine can be produced at a high efficiency and production rate by reacting alkali metal chlorate containing very little chloride with hydrogen peroxide in aqueous mineral acid.
- 7.4 Confronted with the stated technical problem and knowing from S2 that the reaction was possible without adding substantial amounts of chloride, the skilled person would thus obviously consider modifying the process of S8 by lowering the amount of chloride fed to the reactor together with the chlorate, e.g. by using

- commercially available chlorates containing as little as 0.04 (see example 1 of S2) or even less weight-% chloride, in order to reduce chlorine by-product formation.
- 7.4.1 The appellant has not contested the argument of respondent 2 that the pressure in the reactor has practically no influence on the chlorine dioxide generation reactions. Furthermore, the appellant has not indicated any technical reason for which a skilled person, aware of the content of S2, would still have expected that a high chlorine dioxide production rate with minimised chlorine formation would not be possible when the reaction is carried out at atmospheric pressure in a non-crystallising mode. S2 itself does not mention anything that could be considered as an indication that this was not feasible.
- 7.4.2 When modifying the process of S8, the skilled person would, as a matter of routine and taking into account the indications in both S8 and S2, adapt the other reaction conditions (concentrations and temperature) so as to optimise the efficiency and production rate. By doing so, it would inevitably arrive at a process according to present claim 1.
- 7.5 Even accepting the appellant's argument that page 4 of S8 reflected a long lasting prejudice and was in line with the theory generally accepted at the time, i.e. that significant amounts of chloride have to be fed to and to be present in the reactor, this theory or prejudice was no longer generally valid after the publication of S2. Even though S2 relates to a different process involving a different reactor, the



skilled person reading S2 would have recognised without difficulties that the use of chlorate containing chloride amounts as high as the ones recommended by S8 were not necessary. The absence, in S2, of a claim to using low chloride chlorate in a non-crystallising process is not a proof to the contrary.

8. Moreover, the feasibility of carrying out the reduction of sodium chlorate with hydrogen peroxide in aqueous sulphuric acid, without employing chloride and in a non-crystallising mode at about atmospheric pressure is confirmed by document A1.

8.1 The process of A1 differs from the processes according to present claim 1 and according to S8 in that a part of the sodium chlorate is reduced with methanol as the reducing agent in the first vessel (reaction zone A), and the remaining, unreacted chlorate is then reduced by feeding hydrogen peroxide to the depleted reaction mixture in the second vessel (reaction zone B), see claim 1 and example 1.

8.2 One of the objectives of A1 is to "*reduce the amount of by-product chlorine*" when compared to the method using hydrochloric acid as a reducing agent. More particularly, it is stated in A1 that "*the amount of by-product chlorine can be either reduced or made essentially zero*" since no "*hydrochloric acid or chloride forming a source for hydrogen chloride generation is employed*", see page 2, section "2. Scope of claim", page 6, second paragraph, page 9, third paragraph, first and second sentences, and Example 1. Therefore, the skilled person confronted with the

stated technical problem would also consider this document.

- 8.3 The quoted passages of A1 disclose that both reducing steps, hence also the one using hydrogen peroxide in reaction zone B, can be carried out at atmospheric pressure, in a non-crystallising mode, and without employing chloride. The skilled person would also understand that in order to minimise the formation of chlorine, the presence of chloride in the materials fed to the reactor is to be avoided or at least reduced. According to A1, the reduction with methanol according to the Solvay method is "*extremely sluggish*", and the unit consumption of chlorate "*unfavourable*". However, excellent overall unit chlorate consumption can be achieved with the process of A1 by using the efficient but more expensive hydrogen peroxide in the second vessel, see page 8, section [Action]; page 9, lines 1 to 10 and the last two lines; page 10, the first two paragraphs. It can thus be gathered from A1 that methanol, despite its lower reactivity, is mainly used for reducing a part of the chlorate because of its lower price, whilst the more expensive hydrogen peroxide is very efficient and hence very suitable for reducing the remainder of the chlorate. It is also indicated in A1 that "*the percentage decomposition of the sodium chlorate in reaction zones A and B respectively can be freely adjusted according to the amounts of methanol, sulphuric acid and hydrogen peroxide added*" (page 7, third sentence from the bottom). In particular in view of this passage, despite the use of methanol in the first reaction zone, A1 cannot be considered to represent a disincentive for

- using hydrogen peroxide as sole and efficient reducing agent in a two-vessel process.
- 8.4 It was common ground that commercially available sodium chlorates may contain chloride in varying amounts stemming from the chlorate preparation process (see e.g. S2 and post-published D1, example 1). Although A1 is silent about the purity of the chlorates to be used, the quoted passages of A1 would definitely further encourage the skilled person, confronted with the stated technical problem, to use those available chlorates having particularly low chloride contents.
9. The appellant's further arguments presented in support of inventive step are not convincing for the following reasons.
- 9.1 The appellant has submitted no evidence demonstrating the allegedly inevitable accumulation of soluble chloride within the SVP<sup>(R)</sup>-reactor of S2. On the other hand, in view of the reactions involved (see e.g. S2, column 1, lines 21 to 26) and in the absence of any evidence to the contrary, it cannot be excluded that the chloride leaves the reactor as chlorine, as alleged by respondent 2. At the oral proceedings, the appellant moreover confirmed that the chlorine formed as by-product would leave the reactor with the generated chlorine dioxide. Concerning the alleged accumulation of chloride, the burden of proof therefore rested with the appellant who failed to discharge this burden. Therefore, the appellant's arguments based on the alleged accumulation of chloride are not taken into consideration.

9.2 A1 was published in 1991, i.e. after the publication date of S8 (1988). Moreover, the authors of A1 were aware of the content of S8 (see A1, page 9, line 5). Hence, the disclosure of A1 that "*no chloride forming a source for hydrogen chloride generation is employed*" is not to be interpreted in a different or less stringent manner merely because of some statements made in S8 concerning the reaction mechanisms involved, or because the process of S8 as well as some other known processes comprise the addition of significant amounts of chloride; see the R2P process referred to in S11 (sheets 3 and 5), and the Mathieson and Solvay processes referred to in A6 (page 632, second paragraph and page 633, first paragraph).

9.3 The board also notes that the R2P process described in S11 comprises the addition of NaCl and HCl (see "Flow Diagram"). Moreover, it is indicated in S11 (sheet 5) that chlorine is "*coproduced in a small amount*". The ClO<sub>2</sub> generated is then absorbed in water, and most of the co-produced Cl<sub>2</sub> "*is also absorbed in the ClO<sub>2</sub> solution*". Traces of Cl<sub>2</sub> are removed from the vent gas in a tail gas scrubber. The indication in S11 that "**no Cl<sub>2</sub> or NaClO byproduct emanated**" (emphasis added) obviously relates to the overall process including the absorption and scrubbing steps and not to the actual chlorine dioxide generation step, whereas the value of 0.03 ton/tonClO<sub>2</sub> (i.e. 3%) indicated in the table "chemical consumption" on sheet 3 appears to refer to the total chlorine co-produced. S11 is thus not suitable for establishing that a skilled person would not understand from A1 that in order to arrive at a process wherein the amount of by-product chlorine is "essentially zero" in reaction zones A and B (page 9,

paragraph "1)") it had to use chlorates containing very little chloride.

9.4 Comparative example 1 of A1, illustrating the reduction of chlorate with hydrogen peroxide only, contains a reference to "*chlorine ions as a medium*" (page 12, line 4). Considering that this expression is unclear and does not correspond to the language used in the other quoted passages of A1, it cannot give a different meaning to the latter, in the sense that chloride or chlorine needs to be present.

10. The appellant has not provided any evidence in connection with the alleged commercial success of the patented process it relied on at the oral proceedings. In the absence of such evidence, it is not possible to rule out that the alleged commercial success was the result of particular marketing efforts and/or of a specific implementation of the process requiring optimised reaction conditions and engineering measures more specific than those indicated in claim 1. Therefore, this argument cannot be taken into consideration.

11. The subject-matter of claim 1 is thus not based on an inventive step.

*1st and 2nd auxiliary requests*

12. Amendments

12.1 Claim 1 of the first auxiliary request comprises an additional reference to the addition of "*inert gas*" to the second reaction vessel and has been further

modified by the incorporation of the feature "*wherein more chlorate in the depleted medium is converted to chlorine dioxide and whereby the pressure in the second reactor is maintained at about atmospheric pressure and the temperature is the same as in the first reaction vessel*". These amendments find a literal basis on page 9, lines 25 to 36 of the application as filed.

- 12.2 Claim 1 according to the second auxiliary request has the same wording except for the replacement of the expression "*second reactor*" by "*second reaction vessel*". In the passage quoted above (and in the corresponding passage of the patent as granted, see column 7, lines 22 to 34) the expression "*this second reactor*" is used referring to the "*second reaction vessel*" mentioned earlier in the same paragraph. The description thus supports the view of the board that the skilled person would clearly understand that the antecedent of "*the second reactor*" mentioned in claim 1 according to the 1st auxiliary request is the "*second reaction vessel*" mentioned two lines above.
- 12.3 The other objections raised by the respondents with respect to these two auxiliary requests are not affected by these additional amendments and were already dealt with in connection with the main request.
- 12.4 For the reasons given above, the amendments carried out in the respective claims 1 according to the 1<sup>st</sup> and 2<sup>nd</sup> auxiliary requests are not objectionable under Articles 123(2)(3) and 84 EPC.
13. The issue of novelty over S18' can be left open considering that the processes of the respective

claims 1 according to the 1<sup>st</sup> and 2<sup>nd</sup> auxiliary requests lack the required inventive step in view of the other prior art cited for the reasons given below.

14. *Inventive step*

14.1 The patent in suit indicates that "*it is suitable to add heat to the second reactor to maintain the temperature at the stated value*" and that "*this heat may be added by an external heater or by adding additional sulphuric acid to the second reactor*", see column 7, lines 34 to 38. In view of these statements, the board does not accept the appellant's argument that the process according to claim 1 of the 1<sup>st</sup> and 2<sup>nd</sup> auxiliary requests was generally simpler than the one of S8 because it did not require an external heater.

14.2 In the patent in suit no specific effect is associated with the choice of the same temperatures in both vessels. The appellant has not shown that the efficiency and production rate achieved with the claimed process would be higher when operating both vessels at the same temperature rather than at different temperatures. At the oral proceedings, the appellant merely argued that energy could be saved by not heating the second vessel to a higher temperature than the first one. However, S8 does not indicate the operating conditions that should prevail in the second vessel and therefore does not teach that the temperature in the second vessel should be higher than in the first vessel. In these circumstances, it cannot be accepted that an improvement in terms of energy savings was actually achieved with respect to the process of S8.

14.3 S8 discloses a process carried out at atmospheric pressure and with introduction of inert gas (air), see e.g. example 1, and is silent about measures or equipment for increasing or reducing the pressure in the reactor. Hence, carrying out the reaction in the second vessel at atmospheric pressure and with introduction of inert gas as well would be the most straightforward manner of reducing to practice the two vessel process generally mentioned in S8 (page 6, first paragraph).

14.4 Similarly, the skilled person, when reducing to practice the said two vessel process, would first try to carry out the reaction in the preferred temperature range of 30 to 50°C of S8 (see claim) in both vessels. Bearing in mind that the reaction rate depends on the temperature and considering also the disadvantages of operating at higher temperatures (rising peroxide consumption) or lower temperatures (special cooling device required) as indicated in the second paragraph of page 6 of S8, the skilled person would choose an appropriate operating temperature for each of the two vessels. Doing this, it would try different temperatures in both vessels as well as the same temperature in both vessels, thereby arriving at the claimed process. The choice of using the same temperature in both vessels lies within the competence of the skilled person. In the absence of any unexpected effect that may be attributed to this feature, the latter cannot establish the presence of an inventive step.



14.5 There is nothing in A1 or A6 supporting the appellant's allegation that these documents would keep the skilled person trying to put the process of S8 into practice from considering carrying out the reaction at the same temperature in both vessels.

14.5.1 A1 contains no recommendation as to the relative levels of temperatures that should prevail in the two vessels. In example 1 of A1, the first vessel is operated at 40°C, and the second one at a higher temperature of 50 to 60°C. However, according to A1 methanol is used as reducing agent in the first vessel, and hydrogen peroxide is used in the second vessel. There is no reason to infer from this teaching that in the case of using hydrogen peroxide as the reducing agent in both vessels the temperatures would also have to be different.

14.5.2 The cited passage of A6 (page 632, second and third paragraphs from the bottom) refers to the Solvay process which differs from the claimed one in that it relies on the use of methanol as the reducing agent. As pointed out by respondent 2 during the oral proceedings, operating both vessels at the same temperature (135°F) is one option actually disclosed in A6.

14.6 Therefore, the additional features contained in the respective claims 1 according to the 1<sup>st</sup> and 2<sup>nd</sup> auxiliary requests are not considered to render the claimed process inventive.

3<sup>rd</sup> auxiliary request

15. Allowability and clarity of the amendments

15.1 No objection was raised by the respondents with respect to a first additional amendment consisting in the introduction of the feature "*whereby the amount of added hydrogen peroxide is up to 50% of the entire hydrogen peroxide required for the reaction*" into claim 1. This finds a basis on page 9, lines 30 to 33 of the application as filed.

15.2 The second additional amendment is the insertion into claim 1 of the feature "*the amount of chloride added being not more than about 0.05 weight percent of the alkali metal chlorate*". At the oral proceedings, the respondents objected to both the clarity of this amendment and its allowability under Article 123(2) EPC.

15.2.1 Concerning clarity, the respondents argued that claim 1 amended in this manner could be understood to cover embodiments wherein further chloride is deliberately added in addition to the chloride fed to the process in form of an impurity of the chlorate. This would, however, be in contradiction with the passage in column 7, lines 15 to 18 of the patent in suit which reads: "*Beside this amount of chloride being an impurity in the chlorate no further chloride is added*". In reply to the respondents' objection, the appellant stated that the added feature meant that the total amount of chloride added, including the amount of impurities in the chlorate, was not more than about 0.05 weight-% of the alkali metal chlorate.

Even accepting in favour of the appellant that claim 1 as amended had the meaning suggested by the appellant, claim 1 would fail since it covers embodiments wherein chloride is only introduced into the reaction medium as an impurity amounting to not more than 0.05 weight-% of the chlorate starting material and since such embodiments lack the required inventive step for the reasons given under point 17.

15.2.2 The question of whether or not the amendment complies with the requirements of Article 123(2) EPC can be left open since, as indicated above, amended claim 1 must be refused for lack of inventive step of its subject-matter, see point 17. below.

16. Present claim 1 contains an additional, restricting feature not disclosed in S18', namely an indication concerning the maximum amount of hydrogen peroxide fed to a second reactor. The subject-matter of claim 1 is thus novel over S18'. This was not disputed by the respondents at the oral proceedings.

17. *Inventive step*

The previous S8 considerations concerning inventive step also apply to present claim 1 (see point 15.2.1). Furthermore, S8 itself discloses the possibility of feeding a small amount of H<sub>2</sub>O<sub>2</sub> to the second reactor. It specifically mentions feeding to the second reactor 3 to 5% of the amount of H<sub>2</sub>O<sub>2</sub> added to the main reactor, see page 7, the last two sentences, i.e. an amount which falls within the range defined in claim 1. At the oral proceedings, this was acknowledged but not commented on further by the appellant. In the quoted

passage, S8 teaches to feed the said amount of  $H_2O_2$  to the second reactor for "*raising the utilisation factor*" of chlorate, i.e. for converting more of the remaining chlorate to chlorine dioxide by reaction with  $H_2O_2$ . According to the patent in suit, the depleted chlorate solution from the first reaction vessel  $H_2O_2$  is also fed to the second reaction vessel "*for further reaction*" (see claim 12). Besides this indication, the patent in suit contains no specific information concerning the importance of the upper limit of 50%. Hence the additional feature incorporated into present claim 1 cannot render the claimed method inventive.

18. Summarising, since the respective claims 1 according to all requests presented by the appellant are all directed at subject-matter which is not based on an inventive step, none of these requests can be granted.

## **Order**

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

The appeal is dismissed.

The Registrar

The Chairman

A. Wallrodt

M. Eberhard