

Internal distribution code:

- (A) [] Publication in OJ
(B) [] To Chairmen and Members
(C) [X] To Chairmen
(D) [] No distribution

**Datasheet for the decision
of 26 July 2011**

Case Number: T 0319/08 - 3.3.05

Application Number: 00114857.6

Publication Number: 1052224

IPC: C01G 23/047, C01G 23/053

Language of the proceedings: EN

Title of invention:

Particles, aqueous dispersion and film of titanium oxide, and preparation thereof

Patent Proprietor:

SHOWA DENKO K.K.

Opponent:

KRONOS INTERNATIONAL, INC.

Headword:

Titanium oxide sol/SHOWA DENKO

Relevant legal provisions:

EPC Art. 52(1), 54(1)(2), 56

Keyword:

"Novelty, main request (no): implicit novelty-destroying disclosure"

"Novelty, auxiliary request (yes): implicit disclosure not convincingly demonstrated"

"Inventive step, auxiliary request (yes): product with optimised balance of properties not suggested by prior art"

Decisions cited:

-

Catchword:

-



Case Number: T 0319/08 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 26 July 2011

Appellant: KRONOS INTERNATIONAL, INC.
(Opponent) Postfach 10 07 20
D-51307 Leverkusen (DE)

Respondent: SHOWA DENKO K.K.
(Patent Proprietor) 13-9, Shiba Daimon 1-chome
Minato-ku
Tokyo (JP)

Representative: Strehl Schübel-Hopf & Partner
Maximilianstrasse 54
D-80538 München (DE)

Decision under appeal: Interlocutory decision of the Opposition
Division of the European Patent Office posted
12 December 2007 concerning maintenance of
European patent No. 1052224 in amended form.

Composition of the Board:

Chairman: E. Waeckerlin
Members: B. Czech
C. Vallet

Summary of Facts and Submissions

- I. The appeal is from of the interlocutory decision of the opposition division concerning maintenance of the European Patent No. 1 052 224 in amended form.
- II. In the contested decision the opposition division concluded that the patent as amended according to the auxiliary request 1 then on file, met the requirements of the EPC.

The independent claims 1, 7 and 11 according to said auxiliary request 1 read as follows (features added to the claims as granted emphasised by the board):

*"1. An aqueous titanium oxide-dispersed sol comprising **crystalline** titanium oxide particles dispersed in water, said sol comprising chloride ions in an amount of 50 to 10,000 ppm by weight as the chlorine element."*

"7. A titanium oxide film which is formed on a substrate using the aqueous titanium oxide-dispersed sol as set forth in any of claims 1 to 6."

*"11. A process for preparing an aqueous titanium oxide-dispersed sol, comprising the steps of:
forming an aqueous titanium oxide-dispersed sol **comprising crystalline titanium oxide particles** by hydrolysis of titanium tetrachloride, and controlling the amount of chloride ions in said aqueous titanium oxide-dispersed sol to 50 to 10,000 ppm by weight as the chlorine element."*

III. In the opposition proceedings, the parties relied *inter alia* on the following prior art:

D1: EP 0 444 798 B1;

D2: US 2 448 683 A;

D3: EP 0 261 560 B1; and

P1: Römpp Chemie Lexikon, 9. Auflage, 1992, Band 5
"Pi - S", page 4199, keyword "Sol".

IV. In the decision under appeal, the opposition found *inter alia*

- that the subject-matter of claim 1 as granted (main request then on file) was novel over document D1;
- that novelty was lacking, however, over document D2;
- that the amendments to the claims according to auxiliary request 1 then on file were not objectionable under Articles 123(2), 83 and 84 EPC;
- that the subject-matter of claim 1 according to said auxiliary request 1 was novel over documents D1 and D2 and involved an inventive *step*, taking document D3 as the closest prior art; and
- that claims 7 and 11 relating, respectively, to a titanium oxide film formed on a substrate using the sol of claim 1, and to a process for preparing said sol, also met the requirements of the EPC.

V. In its statement of grounds of appeal and in a letter dated 21 June 2011, the appellant (opponent) raised various objections concerning claims 1 to 4 and 7 as allowed by the opposition division.

In particular, the appellant argued that the subject-matter of claims 1, 2 and 3 lacked novelty over document D1. In this respect, the appellant also referred to the newly filed document:

ER1: A declaration ("Eidesstattliche Versicherung") by Mr L. Elfenthal, dated 4 April 2008, comprising an experimental report relating to a reproduction of example 1 of D1 and, annexed thereto, an X-ray diffraction spectrum and a TEM photograph.

Furthermore, the appellant held that the subject-matter of claims 4 and 7 according to said main request did not involve an inventive step in view of D3 representing the closest prior art. In this context, it also referred to document D2.

VI. Under cover of its reply dated 13 November 2008, the respondent (proprietor of the patent) filed seven sets of amended claims as main request and auxiliary requests 1 to 6, respectively.

In said letter, the respondent also stated that it considered the filing of "counter-experimental data".

Concerning the main request, it argued that the alleged lack of novelty of claim 1 over D1 had not been conclusively demonstrated, and that the claimed invention was inventive over the disclosure of documents D3 and D2.

VII. The respondent filed the previously announced "counter-experimental data" under cover of its letter dated 17 December 2008 in form of document

ER2: An experimental Report concerning reproduction of example 1 of document D1.

Under cover of said letter the respondent also filed two sets of amended claims as auxiliary requests 0 and 1a.

VIII. In response to the summons to oral proceedings, the respondent filed the further document

ER3: A declaration by Masayuki Sanbayashi dated 20 May 2011 comprising a revised version of the experimental report comprised in document ER2.

IX. In a communication issued by the board in preparation of the oral proceedings, the board addressed *inter alia* the question of whether or not a "slurry" as disclosed in D1 could be considered as a sol within the meaning of claim 1. Furthermore, the board raised various questions concerning the ambit and the patentability of claims 7 and 10 relating to a "*titanium oxide film...*".

X. Under cover of a reply to said communication, the appellant filed the two further documents

B1: Römpps Chemie Lexikon, 8. Auflage, 1983, Band 3 "H - L", pages 2169-2170, keyword "Kolloidchemie";

and

B2: Ullmann's Encyclopedia of Industrial Chemistry, 5th edition, 1992, Vol. A20; page 271 and pages 276 to 279.

XI. Oral proceedings were held on 26 July 2011, during which the board expressed concerns regarding claims 6 to 9 of auxiliary request 0 then on file, which are directed to titanium oxide films. In response, the respondent filed a new auxiliary request 0 consisting of claims 1 to 8 and no longer comprising claims directed to such films.

Independent claims 1 and 6 according to the new auxiliary request 0 read as follows (features added to the claims as granted highlighted by the board):

*"1. An aqueous titanium oxide-dispersed sol comprising **crystalline** titanium oxide particles dispersed in water, said sol comprising chloride ions in an amount of 50 to 10,000 ppm by weight as the chlorine element, wherein said titanium oxide particles have an average particle size of 0.01 to 0.1 μm ."*

*"6. A process for preparing an aqueous titanium oxide-dispersed sol, comprising the steps of:
forming an aqueous titanium oxide-dispersed sol **comprising crystalline titanium oxide particles having an average particle size of 0.01 to 0.1 μm** by hydrolysis of titanium tetrachloride, and controlling the amount of chloride ions in said aqueous titanium-oxide dispersed sol to 50 to 10,000 ppm by weight as the chlorine element."*

XII. As far as they concern the respondent's main request and the auxiliary request 0 submitted during the oral proceedings, the arguments of the parties may be summarised as follows.

The **appellant** considered that the subject-matter of claim 1 (both requests) lacked novelty over the disclosure of document D1. D1 was concerned with the preparation of crystalline titanium dioxide having a crystal size of less than 100 nm. Considering the definition of the term "sol" as given e.g. in document B1, the "slurry" obtained as intermediate product, at the end of step b of the process according to D1, before the final neutralisation, was a sol of crystalline titanium dioxide particles. Moreover, according to D1, the "slurry" according to D1 contained HCl in a concentration of 8 to 25 g/l, preferably 10 g/l, and hence contained an amount of chloride ions falling within the range specified in claim 1. The reproduction of this example 1 of D1 (see document ER1) showed that the "slurry" was a sol of microcrystalline TiO₂ and met the definition of a sol as indicated in document B1. Said sol contained 10 g/l HCl, and thus chloride ions in an amount of less than 10,000 but more than 50 ppm by weight. Moreover, the experimental reproduction of example 1 of D1 showed that a sol of microcrystalline anatase-type titanium dioxide, having an average crystallite size of about 5 nm and agglomerate sizes of from 40 to 90 nm was obtained. The experimental report ER3 submitted by the respondent did not convincingly demonstrate the contrary, since the experiment described therein was not a strict reproduction of example 1 of D1, which related to the preparation of titanium dioxide by the so-called "sulfate method". With respect to the latter, the appellant referred to document B2. In view of the differences between the method of example 1 of D1 and the method used in ER3, it was not surprising that according to ER3 coarser particles were obtained.

Moreover, since claim 1 at issue did not require a specific transparency or stability of the sol, the findings in ER3 relating to these properties were immaterial.

With regard to inventive step, the **appellant** argued that taking the disclosure of document D3, and more particularly example A thereof as closest prior art, the preparation of a TiO_2 sol falling within the terms of claim 1 at issue, i.e. having a chloride ion concentration in the range of from 50 to 10,000 ppm, was obvious in view of the information contained in D3 alone. Example A of document D3 disclosed an acidic sol of pH 1.5 containing about 14,600 ppm chloride. According to table 1 of the contested patent, a pH value of 5 corresponded to a chloride content of 50 ppm. Since document D3 referred to known TiO_2 sols stabilised at a pH of 3, which value corresponded to a chloride content within the range specified by claim 1 at issue, it was obvious, when starting from example A, to provide further sols having all the features of claim 1 at issue. Moreover, document D3 taught the preparation of stable sols having a pH of 6 to 8 and mentioned that stable sols having a lower pH, i.e. a higher chloride content, were known e.g. from document D2. Since no discontinuity in terms of the stability of the sol were to be expected in the range from very low chloride concentrations at a pH of 6 to 8 to the chloride concentration of 14,600 ppm indicated in example A of D3, it was obvious to the skilled person in view of document D3, taken alone or in combination with document D2, to provide and use further stable sols having a chloride content in the range of from 50 to 10,000 ppm.

The **respondent** argued that document D1 disclosed neither explicitly nor implicitly the preparation of a sol of titanium dioxide. Compared to a sol, the "slurry" of a "precipitate" obtained according to example 1 of D1 was a suspension of coarser particles or of agglomerated particles, which were visible to the eye and easier to filter off. The experimental report ER1 of the appellant did not convincingly establish that the slurry obtained according to example 1 of D1 was *de facto* a sol as defined in claim 1. According to ER1 the titanium dioxide obtained was of the anatase type and had a different crystallite size distribution than the product of example 1 of D1, which was of the rutile type. Moreover, the particle size of the titanium dioxide were measured after having being filtered off and dried under conditions not specified in the report ER1. Referring to its own experimental report ER3, the respondent submitted that a reproduction of example 1 of D1 did not lead to a sol, but to a slurry of precipitated material. This was confirmed by the measured average particle size and its increase over time, as well as by the cloudiness and lack of transparency of a film formed on a substrate, using said slurry.

Having regard to inventive step, the respondent pointed out that the sols described in D3 were not necessarily crystalline and could be sols of hydrous titanium oxide. Starting from the disclosure of document D3 as the closest prior art, the technical problem consisted in providing TiO₂ sols which, when formed into a film on a substrate, exhibited a good combination of photocatalytic activity, transparency and adhesiveness to the substrate. The examples of the patent showed in

particular the criticality of the upper and lower boundaries for the chloride ion content of the sol in terms of the transparency and adhesiveness achieved.

Neither D3 nor D2 addressed this technical problem or gave a hint to provide a sol according to present claim 1.

XIII. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

The respondent requested that the appeal be dismissed (main request) or, in the alternative, that the patent be maintained on the basis of the claims according to one of the auxiliary requests 0, 1, 1a, 2 to 6, taken in that order - auxiliary requests 1 and 2 to 6 were filed with letter dated 13 November 2008, auxiliary request 1a was filed with letter dated 17 December 2008, and auxiliary request 0 was filed at the oral proceedings.

Reasons for the Decision

Procedural matters

1. Admissibility of the new auxiliary request 0
 - 1.1 The claims according to said request filed at the oral proceedings are identical with the claims according to auxiliary request 0 previously on file, except for the deletion of claims 6 to 9 directed to titanium oxide films and the renumbering of the remaining claims.

1.2 The request was filed in response to concerns expressed by the board in respect of said claims 6 to 9 in the course of the oral proceedings and in its communication. The deletion of these claims did not raise any new, let alone complex issues, and the appellant did not object to the filing of said new request.

1.3 Under these circumstances, the board decided to admit the respondent's new auxiliary request 0 despite its late filing, in accordance with Article 13(1) and (3) RPBA.

2. Admissibility of late filed evidence

2.1 Both parties filed additional evidence after having been summoned to oral proceedings.

2.2 The experimental report ER3 submitted by the respondent is a revised version of the experimental report ER2 filed much earlier. Document ER3 was signed and dated by the author of the report and a translation error in the description of the experiment performed was corrected (viz. the term "sulfuric acid" used in ER2 was replaced by the term "hydrochloric acid" in ER3).

2.3 Documents B1 and B2 are short extracts from encyclopaedias illustrating common general knowledge in the concerned technical fields.

2.4 For the board, the filing of documents ER3, B1 and B2 did not raise any complex issues. Neither did the parties to the proceedings object to the late filing of said documents.

- 2.5 The board thus decided to admit the late filed evidence in accordance with Article 13(1) and (3) RPBA.

Main request

3. Amendments

- 3.1 The amendments restrict the independent claims 1 and 11 to sols comprising crystalline titanium oxide particles and to processes for preparing such sols, respectively.

- 3.2 The amendments narrow down the ambit of claims 1 and 11 as granted and find a basis in the parent European application No. 97115028.9 as filed (see e.g. claims 1, 2 and 15) and in the divisional application as filed (see e.g. claims 1, 2 and 11 of the latter) that lead to the grant of the patent in suit. This was not in dispute.

- 3.3 The amendments thus meet the requirements of Articles 76(1), 123(2) and 123(3) EPC.

4. Novelty

- 4.1 Document D1 discloses the preparation of microcrystalline titanium dioxide having rutile structure and having a crystal size of less than 100 nm (D1: page 1, lines 3 to 7; page 3, lines 6 to 7; claims 1 and 13). The preparation process comprises treating solid titanium dioxide hydrate with a base (step a), treating the precipitate treated with the base with hydrochloric acid (step b), and neutralising the so-treated precipitate (step c). In step b, the final hydrochloric acid content is adjusted to 8 to 25 g/l,

preferably about 10 g/l (D1: claims 1 and 13; page 3, lines 53 to 56).

More specifically, example 1 of D1 discloses the treatment of titanium dioxide hydrate precipitated by hydrolysis. The treatment comprises the steps of adding a base, followed by adding hydrochloric acid. As an intermediate product, a slurry of titanium dioxide is obtained, which is further acidified to set the HCl content of the slurry to 10 g/l. The slurry obtained contained titanium dioxide in the form of rutile crystals (D1: page 4, lines 37 to 51) It was not in dispute that the chloride ion concentration in said slurry falls within the range of 50 to 10,000 ppm specified in present claim 1.

4.2 In document D1 it is stated that the dispersion obtained as (intermediate) product in step b of the method is a "precipitate" (see e.g. claim 1 of D1) or a "slurry" (see e.g. example 1 of D1). D1 contains no express reference to a "sol". Hence, the crucial question to be decided regarding the issue of novelty is whether or not the slurry of rutile crystals obtained in step b of the method disclosed in D1 (as illustrated by example 1 thereof) is, nevertheless, to be considered as a "sol" within the meaning of present claim 1 in view of its implicit properties.

4.3 The board notes that the term "sol" usually designates a very fine dispersion of a solid in a liquid, which may also be called "colloidal solution". (see P1, keyword "sol"). However, there exists no sharp distinction between colloidal solutions and sedimenting suspension in terms of a generally accepted physico-

chemical definition, as pointed out in document B1 (page 2169, right-hand column, last paragraph), which is an extract from a chemical encyclopaedia illustrating common general knowledge. Despite the lack of a precise, generally applicable definition of a "sol" in terms of the maximum size of the dispersed particles, colloidal particles are conventionally considered to have sizes in the range of from 10^{-7} to 10^{-5} cm, i.e. of from 0.01 to 0.1 μm (B1: loc. cit.)

4.4 Against this background, the board considers that the mere use of the term "sol" in claim 1 at issue is not sufficient to delimit its subject-matter from "slurries" or "precipitates". Therefore, it has to be investigated whether a micro-crystalline, HCl-containing rutile "slurry" or "precipitate" described as intermediate product of step b in document D1 qualify as "sol" within the meaning of present claim 1. In other words, the question arises whether D1 implicitly discloses a dispersion of titanium dioxide to be considered as a sol in accordance with the explanations given in documents P1 and B1.

4.4.1 In this respect, it can be gathered from the respondent's experimental report ER3 that the average particle size of the aqueous dispersion freshly prepared along the lines of example 1 of D1 is about 0.125 μm . This value is greater than, but relatively close to, the value of 0.1 μm which is conventionally considered to represent the upper limit of the particle size of sols (see B1, loc. cit.).

The board notes that in claim 1 at issue no upper limit is specified for the particle size of the dispersed

titanium oxide particles of the sol. An upper limit of 0.1 μm is only indicated in present dependent claim 2, and can thus not be considered as mandatory, but only as a preferred feature of the sol referred to in independent claim 1. For the board, the measured average particle size of 0.125 μm reported in ER3 is thus not, as such, sufficient to establish that the freshly prepared dispersion according to Example 1 of D1 is distinct from a sol.

- 4.4.2 In the report ER3, the stability of the dispersion so-obtained was evaluated by measuring the average particles size after one day. It was found that the size had increased to 0.153 μm . Moreover, the slurry was used to coat a film having a thickness of 0.15 μm on a quartz plate and the light transmittance was measured at 550 nm. A value of 86% was found (ER3: page 2, section "results"). The author of the report ER3 states that "the stability of the slurry was low and the slurry was not a stable sol with an excellent dispersion", and that "the film coated on the quartz plate was clouded and the light transmittance was 86%, indicating that the particles in the slurry were agglomerated". Therefore, the author concludes that "the slurry obtained in Example 1 of D1 is not a sol".

The board observes, however, that a specific stability over time of the sol, let alone in the absence of a stabiliser, is not required by present claim 1. Thus, the observed increase of the average particle size after one day is not sufficient to establish that the dispersion freshly formed according to Example 1 of D1 is distinct from a sol. Likewise, the board is not convinced that the observed "cloudiness" and the

measured value of the transmittance (86% at 550 nm) of the film deposited on a substrate, which has to be compared to a value of 90% considered acceptable according to example 6 of the patent in suit (see table 1), demonstrates in a conclusive manner that the particles of the freshly prepared dispersion are agglomerated to such an extent that said dispersion is distinct from a sol.

- 4.4.3 The appellant also held that the reference, in example 1 of D1, to the filtration of the slurry implied that the latter was not a sol but a dispersion of relatively coarser particles.

However, considering that it was not in dispute that sols can be filtrated using e.g. semi-permeable membranes (see e.g. document B1, page 2170, left-hand column, second full sentence), the board does not accept this argument.

- 4.4.4 For the above reasons, the board arrives at the conclusion that the slurry of crystalline TiO_2 obtained in step b of the process according to Example 1 of D1 can be considered as a "sol" within the broadest meaning of the term.

- 4.5 In the board's judgment, the subject-matter of claim 1 thus lacks novelty over the disclosure of document D1 (Articles 52(1) and 54(1)(2) EPC).

5. Consequently, the respondent's main request is not allowable.

Auxiliary request 0

6. Amendments

6.1 The independent product and process claims 1 and 6 were amended by incorporating a feature further characterising the dispersed titanium oxide particles, namely the "*average particle size of 0.01 to 0.1 µm*".

6.2 Compared to the claims according to the main request, the amendments narrow down the ambit of the independent product and process claims 1 and 6. The amendments find a basis in the parent application as filed (see e.g. claim 2) and in the divisional application as filed (see e.g. claim 2) that lead to the grant of the patent in suit. This was not disputed by the appellant.

6.3 The amendment consisting in the deletion of independent claim 7 and of dependent claims 9 to 10 as granted (directed to "*titanium oxide films...*") and the consequential renumbering of the remaining claims cannot, by its very nature, give rise to objections under Articles 76(1), 123(2) or 123(3) EPC.

6.4 The amendments thus meet the requirements of Articles 76(1), 123(2) and 123(3) EPC.

7. Novelty

7.1 The appellant held that the experimental evidence ER1 proved that when carrying out the method described in D1, and more particularly the method used in example 1 thereof, a sol within the meaning of claim 1 was inevitably obtained, i.e. a dispersion wherein the

crystalline TiO_2 particles had an average size within the range of "0.01 to 0.1 μm " as specified in claim 1 at issue.

- 7.1.1 The board observes, however, that according to example 1 of D1 rutile crystals are formed in step b of the process described, whereas anatase crystals were obtained in the experiment reported in ER1, which in the appellant's view was supposed to reproduce example 1 of D1.

For the board, this is an indication that the appellant, when reproducing example 1 of D1, may have carried out the process under different conditions than the authors of D1.

- 7.1.2 Moreover, according to ER1, the reported average crystal size (5 +/- 0.5 nm) and agglomerate size range (40 to 90 nm) was not measured directly within the dispersion formed, but only after filtering off and drying the TiO_2 contained therein under conditions not specified in ER1.

Therefore, the board considers that the results reported in ER1 do not conclusively prove that TiO_2 micro-crystals in the form of aggregates having an average particle size of less than 0.1 μm must inevitably prevail in the solid/liquid dispersion before filtering off and drying the TiO_2 so-prepared.

- 7.1.3 The appellant criticised the experimental evidence ER3 submitted by the respondent. It argued that the measured average particle size was above the upper limit of 0.1 μm specified by claim 1 at issue because

example 1 of D1 was not properly reproduced. The measured average particles size of 0.153 μm was due to the fact that metatitanic acid, i.e. an iron-free material, had been used as raw material instead of titanium dioxide hydrate obtained by subjecting ilmenite to the sulfate method. It was well-known, as illustrated by e.g. document B2, that residual iron was one of the factors affecting the particle size and the degree of flocculation of the hydrolysates, and that "high concentrations" of salts such as FeSO_4 "lead to finely divided hydrolysate" (B2: page 278, right-hand column; page 279, left-hand column, lines 1 to 3). Hence, in the appellant's view, the absence of iron led to coarser particles and agglomerates than the ones observed according to ER1.

The board observes, however, that it is expressly indicated in example 1 of D1 that before the precipitation of the titanium dioxide hydrate "impurities are removed and the iron sulfate is crystallised away" (D1: page 4, line 39). Hence, there is no reason to assume the presence of "high concentrations" of iron salts in the titanium solution subjected to hydrolysis according to example 1 of D1. Moreover, the passages of document B2 quoted by the appellant do not address the particle and/or agglomerate size distribution of dispersions obtained when subjecting the hydrolysate to a peptisation with hydrochloric acid.

Therefore, the board does not accept the conclusion of the appellant, according to which the particle size reported in ER3 was too high due to the absence of iron in the starting material.

- 7.1.4 In view of the above, the board concludes that the appellant has not conclusively demonstrated that example 1 of D1 constitutes a direct and unambiguous disclosure of a sol of crystalline titanium oxide particles within the range of from 0.01 to 0.1 μm specified in claim 1. Hence, the appellant has not discharged the burden of proof resting on him in this respect.
- 7.2 The board is also satisfied that none of the other documents referred to by the parties in the course of the appeal proceedings discloses a sol according to claim 1 at issue. Since this was not in dispute between the parties, detailed reasons need not be given in this respect.
- 7.3 The subject-matter of claim 1 and, consequently, of claim 6 directed to a method for preparing a sol with the features of claim 1, and of dependent claims 2 to 5, 7 and 8, is thus novel (Articles 52(1) and 54(1)(2) EPC).
8. Inventive step
- 8.1 The patent in suit is concerned with aqueous titanium oxide sols and their preparation.
- 8.2 The parties concurred that document D3, which is concerned with the preparation stable titania sols having a pH in the neutral range (D3: page 2, lines 5 to 6), represents the closest prior art. More specifically, the board accepts that a sol prepared in accordance with example A of D3 constitutes a reasonable starting point for the assessment of

inventive step, since said example comprises a clear indication of the amount of HCl, and hence of the amount of chloride ions, comprised in the sol (D3: page 4, lines 39 to 41).

8.2.1 The sols according to are D3 are prepared by peptizing hydrous titanium oxides with a monobasic acid or a salt thereof to obtain an acidic titania sol, from which anions are subsequently removed using e.g. anion exchange resins (D3: claims 1, 8 and 14; page 3, lines 39 to 56). The sols so-prepared have a pH in the neutral range of from 6 to 8 and they may comprise a dispersion stabiliser (D3: claims 2 and 8). According to D3, the finely divided titania in the sol may be "amorphous oxides such as metatitanic acid, orthotitanic acid, partially-crystallized amorphous oxides containing a rutile or anatase type, or a mixture of both these amorphous oxides". Preferably, more than 80% of the dispersed titania particles have a size of less than 0.1 μm , to avoid increased "light-scattering effects" reducing transparency (page 3, lines 12 to 15).

8.2.2 The only example of D3 comprising an explicit indication of the chloride content of the titania sol is example A. The sol obtained in said example has a pH of 1.5 and contains 1.5 % by weight of HCl. It was not in dispute between the parties that this concentration of HCl corresponds to a chloride ion concentration outside and above the concentration range of 50 to 10,000 ppm specified in claim 1 at issue. Examples B and C of D2 describe the removal of anions from the sol obtained according to example A using an anion exchange resin. Both examples are, however, silent about the

residual chloride content of the neutral titania sols obtained.

8.3 According to the patent in suit, the technical problem underlying the invention consists providing a titanium oxide sol, which when formed into a film on a substrate exhibits excellent photocatalytic activity, transparency and adhesion to the substrate (see e.g. section [0016] of the granted patent).

8.4 As a solution to the stated technical problem, the patent proposes the provision of a sol according to claim 1 of the auxiliary request 0, which sol is characterised in particular

- in that it comprises "*crystalline titanium oxide particles dispersed in water*", "*wherein said titanium dioxide particles have an average particle size of 0.01 to 0.1 μm* ", and
- in that comprises "*chloride ions in an amount of 50 to 50,000 ppm by weight as chlorine element*".

8.5 In view of the experimental results reported in the patent in suit (see examples 1 to 8 and comparative examples 1 to 4), the board accepts that the stated technical problem is successfully solved by the claimed solution. The appellant has not provided arguments or evidence to the contrary.

8.5.1 More particularly, the examples of the patent in suit (see Tables 1 and 2) show that a sol meeting the criteria recited in claim 1 may indeed be formed into a film exhibiting an optimised balance of properties. The films obtained are photocatalytic ("decomposition of oxalic acid" of from 43 to 45 %), exhibit a high

transparency ("light permeability" of from 90 to 96 %), and adhere strongly to the substrate ("peeling strength" values from 90 to 100 %).

8.5.2 Comparative example 1 (chloride ion concentration of 30 ppm) and comparative example 2 (chloride ion concentration of 15,000 ppm) illustrate the importance of keeping the chloride ion concentration within the range specified in claim 1 at issue. They show that a sol with a chloride ion concentration below the lower limit indicated in claim 1 (comparative example 1) leads to films with an unsatisfactory adhesion to the substrate ("peeling strength" value of only 70 % in comparative examples 1 and 3) and that a sol with a chloride ion concentration above the upper limit indicated in claim 1 (comparative examples 2 and 4) leads to an unsatisfactory transparency of the film ("light permeability of only 55 %).

8.6 Hence, it remains to be decided whether, starting from the closest prior art as disclosed in document D3, the claimed solution to the technical problem was obvious in view of the state of the art.

8.6.1 The titania sols produced according to D3 are foreseen to be used, in view of their "ultraviolet ray screening effects", as raw material in applications such as cosmetic formulations, e.g. sunscreen products, or food packaging materials, e.g. in form of resin compositions coated onto the material (D3: page 2, lines 8 to 10; page 3, lines 2 to 8; page 4, lines 24 to 27; page 5, Test examples 1 and 2). A catalytic activity of the titanium oxide is neither aimed for nor addressed, and fully or partly amorphous titanium oxide sols are

considered to be useful for the intended purpose (D3: page 3, lines 9 to 11). The degree of crystallinity of the "titania sol" obtained according to said example A is thus not addressed in D3. In document D3, the focus lies, on the one hand, on the stability of the sol, which is achieved by the removing anions, such as chloride ions, therefrom. On the other hand, the focus lies on the UV radiation screening effect achievable.

The board considers that document D3 encourages the skilled person to perform a far-reaching removal of the chloride anions (as in examples B and C of D3) in order to achieve the desired high stability of the sol. Hence, although a preference is expressed in document D3 for sols having a particle size of less than 0.1 μm in order to achieve a high transparency (D3: page 3, lines 12 to 15), this document taken alone does not suggest to stop the removal of anions from the titania sol obtained according to example A at a chloride level in the range of from 50 to 10,000 ppm. More particularly, D3 does not suggest setting the chloride level within said range for the purpose of providing sols suitable for achieving an optimised balance of properties (crystallinity, catalytic activity, transparency and adhesion to the substrate) of films formed therefrom on a substrate.

- 8.6.2 The appellant pointed out that document D3 (page 2, lines 16 to 22) referred to known stable titania sols having a pH of 3 or less, and in particular to document D2. At the oral proceedings, it held that the skilled person would thus consider increasing the pH of 1.5 of the sol described in example A of D3 to a higher value, corresponding to a chloride ion concentration within

the range according to claim 1 at issue, as an obvious manner of providing a titania sol of reduced acidity but comparable stability, which may be useful in other applications.

The board does not accept this argument. D3 (see claim 1) expressly aims at providing sols "having a neutral pH range". Hence, the skilled person unaware of the present invention would not, when looking for a solution to the stated technical problem, consider stopping the anion removal from the sol such as to obtain a sol having an intermediate pH value between 1.5 (sol of example A of D3) and a neutral value higher than 6 (see the pH range in claim 1 of D3; the pH values 7.8 in example B and of 7.2 in example C of D3), let alone in order to set the chloride content in the range of 50 to 10,000 ppm.

8.6.3 The appellant also relied on a combination of the technical teachings of documents D3 and D2.

Document D2 (see claim 1; column 1, lines 45 to 56) relates to the preparation of soft-textured, friable, anhydrous TiO_2 , which can readily be dispersed in organic vehicles and in the form of particles having a diameter of 0.02 to 0.50 μm , i.e. an uniform particle size corresponding to the colloidal TiO_2 from which it is prepared, which comprises neutralising a peptised colloidal TiO_2 dispersion by means of an alkaline agent and subsequently calcining the coagulated TiO_2 product at a temperature ranging from 300 to 700 $^\circ\text{C}$ in the presence of the salt formed by the reaction between the alkaline agent and the peptising agent. D2 is focused on the use of the anhydrous TiO_2 as reinforcement agent

in rubber (column 4, lines 31 to 38; column 5, lines 55 to 73). Applications thereof as "solvent absorbent, catalyst, etc" are mentioned only in general terms (D2: column 3, lines 28 to 32). At the oral proceedings, the appellant submitted that example I of document D2 described a sol with a particles having a size of 25 nm and containing about 9000 ppm chloride ions. Starting from document D3, and considering the teaching of D2, the provision of a sol according to claim 1 at issue thus constituted an "obvious variation".

Document D2 is, however, not concerned with the preparation of aqueous sols to be used for forming catalytic TiO₂ films on substrates. Hence, this document neither addresses the crystallinity of the titanium dioxide aqueous sols prepared in intermediate steps, nor the transparency or adhesiveness of films that would be obtainable by depositing such sols on a substrate.

Therefore, even assuming that the person skilled in the art, starting from document D3 and seeking a solution for the stated technical problem would nevertheless consider document D2, he would not be induced by the content of D2 to modify the teaching of D3 by reducing the chloride concentration of the sol obtained according to example A of D3 (i.e. 14,600 ppm) to the chloride concentration of example I of D2 (i.e. 9,000 ppm according to the appellant) whilst making sure that the sol particles be crystalline, let alone for the purpose of providing sols suitable for achieving an optimised balance of properties (crystallinity, catalytic activity, transparency and adhesion to the substrate) of films formed therefrom on a substrate.

8.6.4 Summarising, the board concludes that, considering the disclosures of both D3 and D2, the provision of a sol according to claim 1 at issue was not obvious to the skilled person.

8.6.5 The board is also satisfied that none of the other prior documents referred to by the parties in the appeal proceedings contains any relevant information that could render the subject-matter of claim 1 obvious.

8.6.6 The same conclusion applies to the subject-matter of claim 6, since the latter relates to a process for the preparation of a sol having all the features of claim 1.

8.7 In the board's judgement, the subject-matter of independent claims 1 and 6 and, consequently, of claims 2 to 5, 7 and 8 dependent thereon, thus involves an inventive step (Articles 52(1) and 56 EPC).

9. Hence, the respondent's auxiliary request 0 is allowable.

10. Consequently, the respondent's lower-ranking auxiliary requests 1, 1a and 2 to 6 need not be given further consideration in the present decision.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the department of first instance with the order to maintain the patent on the basis of claims 1 to 8 according to the auxiliary request 0 filed at the oral proceedings and a description to be adapted.

The Registrar

The Chairman

C. Vodz

E. Wäckerlin