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Datasheet for the decision of 21 January 2016

Case Number: T 1612/13 - 3.3.06

Application Number: 02721170.5

Publication Number: 1381652

IPC: C09C1/36

Language of the proceedings: EN

Title of invention:
Titanium dioxide pigments with improved gloss and/or durability

Patent Proprietor:
Cristal USA Inc.

Opponent:
KRONOS INTERNATIONAL, INC.

Headword:
Titanium dioxide pigments / CRISTAL USA

Relevant legal provisions:
EPC Art. 52(1), 54, 56

Keyword:
Novelty (yes)
Inventive step (yes) - improvement convincingly proven

Decisions cited:

Catchword:

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Case Number: T 1612/13 - 3.3.06

D E C I S I O N
of Technical Board of Appeal 3.3.06
of 21 January 2016

Appellant: (Patent Proprietor)	Cristal USA Inc. 20 Wight Avenue Suite 150 Hunt Valley, MD 21030 (US)
Representative:	Cockerton, Bruce Roger Carpmaels & Ransford LLP One Southampton Row London WC1B 5HA (GB)
Respondent: (Opponent)	KRONOS INTERNATIONAL, INC. Postfach 10 07 20 51307 Leverkusen (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 13 May 2013 revoking European patent No. 1381652 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	B. Czech
Members:	L. Li Voti
	S. Fernández de Córdoba

Summary of Facts and Submissions

I. The present appeal is from the decision of the Opposition Division to revoke the European patent no. 1 381 652.

II. The patent was granted with a set of thirty claims, the independent claims thereof reading as follows:

"*1. A method for preparing titanium dioxide pigment having improved gloss and/or durability comprising: a) treating titanium dioxide with a sulfate-free zirconium compound to form a first layer on the titanium dioxide consisting of sulfate-free zirconia; and b) treating the zirconium treated titanium dioxide with a sulfate-free aluminum compound to form a second layer comprising sulfate-free alumina to form the titanium dioxide pigment having improved gloss and/or durability.*"

"*10. A method of preparing titanium dioxide pigment, having improved gloss and/or durability, comprising: wet treating titanium dioxide with a sulfate-free aluminum compound to form a sulfate-free alumina layer on the titanium dioxide, wet treating the aluminum treated titanium dioxide with a sulfate-free zirconium compound to form a sulfate-free zirconia layer, thereby forming the titanium dioxide pigment having improved gloss and/or durability.*"

"*23. A sulfate-free titanium dioxide pigment having improved gloss and/or durability comprising: titanium dioxide base, surface treated with a sulfate-free aluminum compound, a sulfate-free zirconium compound on the aluminum compound and a phosphate compound on the zirconium compound.*"

"*30. A sulfate-free titanium dioxide pigment having improved gloss and/or durability comprising: titanium dioxide base, surface treated with a compound consisting of sulfate-free zirconium, a sulfate-free alumina compound on the sulfate-free zirconium compound, and a phosphate compound on the sulfate-free aluminum compound.*"

The dependent claims concern specific embodiments of the preparation methods according to claims 1 and 10 or of the titanium dioxide pigment according to claim 23.

III. Opposition was filed on the grounds of lack of novelty, lack of inventive step (Articles 100(a) EPC) and insufficiency of disclosure (Articles 100(b) EPC).

The evidence relied upon by the parties include the following documents:

D1: EP 0 565 342 B1;
D2: US 4 328 040 A;
D4: Test report ("Vergleichsversuche") submitted with the notice of opposition dated 10 February 2010;
D5: Declaration by Neil Burniston dated 24 November 2010; Reworking of the D4 experiments
D6: Joint Declaration by Neil Burniston and David Salter dated 24 November 2010 - Statistical analysis of experimental data;
D7: "DATA SET 1", Annex A1 to Opponent's letter of 17 August 2011;
D9: Joint Declaration by Neil Burniston and David Salter dated 21 January 2013 - Graphical representation of experimental data;
D10: Joint Declaration by Neil Burniston and David Salter dated 21 January 2013, supplementing D5;

D11: Deutsche Norm DIN EN ISO 2813 (June 1999), cover page and page 7; and
D12: Deutsche Norm (Entwurf) DIN EN ISO 2813 (October 2012), cover page and pages 13, 24 and 25.

IV. The Opposition Division came to, *inter alia*, the following conclusions:

- The invention was sufficiently disclosed.

- The subject-matters of granted claim 1 and of claim 1 according to the then pending auxiliary request III lacked novelty over examples 6 and/or 7 of document D2.

- Example 7 of document D2 represented the closest prior art and the subject-matter of claim 1 according to the then pending auxiliary request III-b lacked an inventive step over D2.

- None of the other pending claim requests complied with the requirements of Article 123(2) EPC.

V. In its statement of grounds of appeal, the Appellant (Patent Proprietor) contested the reasoning given in the the appealed decision and defended the patent in its granted version. With the statement, it nevertheless submitted six sets of amended claims as auxiliary requests I to VI, as well as the following three declarations by Neil Burniston dated 19 September 2013:

D13: Technical evaluation of Example 7 of document D2;
D14: Further experimental data (reworking of example 7 of D2 and experimental comparison with example 1 of the patent in suit); and
D15: Statistical analysis of experimental data on file.

VI. In its reply, the Respondent (Opponent) rebutted the arguments submitted by the Appellant and maintained that the claimed subject-matter lacked novelty over example 7 of D2. It also argued that example 7 of D2 represented the closest prior art for the assessment of inventive step and that the claimed subject-matter (all requests) did not involve an inventive step for the reasons given in the decision under appeal and in its written submission made during the opposition proceedings. In countering the Appellant's arguments based on D13 and D14, it also referred to the following further documents:

D16: "Crystal Chemistry of Boehmite" by R. Tettenhorst et al.; Clays and Clay Minerals, 1980, Vol.28, No. 5, pages 373 to 380;
D17: "TiO_2 Pigment Structure and Kinetics of PVC Weathering" by U. Gesenhues et al.; Journal of Vinyl & Additive technology, June 2000, Vol.6, No. 2, pages 80 to 87; and
D18: EP 0 993 494 B1.

VII. With its reply thereto, the Appellant filed two further documents endorsing declaration D13, namely

D19: Declaration by Michael Eklund, dated 2 September 2014, and
D20: Declaration by Mark Watson, dated 5 September 2014.

VIII. Oral proceedings before the Board were held on 21 January 2016. The parties were heard regarding the Appellant's main request (patent as granted). As regards novelty, the Respondent only maintained an objection based on D2. As regards inventive step, it only maintained and substantiated an objection based on D2 as

the closest prior art and the experimental data on file, and being directed in particular against claim 1.

IX. Requests

The Appellant requested that the decision under appeal be set aside and the patent be maintained as granted (main request) or, in the alternative, that the patent be maintained on the basis of any of the sets of claims labelled auxiliary requests I to VI, submitted with the statement of grounds of appeal of 23 September 2013.

The Respondent requested that the appeal be dismissed.

X. The Appellant's arguments of relevance here, can be summarised as follows:

Novelty

- None of examples 6 and 7 of document D2 mentioned whether or not the aqueous solution of zirconium carbonate complex used according to these examples for coating the titanium dioxide contained sulfate ions. Moreover, the description of D2 disclosed, as the only information regarding ways of preparing the aqueous coating solution of zirconium carbonate complex, the reaction of zirconium **sulfate** with an alkali metal or ammonium carbonate solution. This reaction resulted necessarily in the presence of sulfate ions in the solution. Therefore, the cited examples did not disclose directly and unambiguously that the aqueous solution of zirconium carbonate complex used did **not** contain sulfate ions, or that the zirconia layer formed on the titanium dioxide was sulfate-free, i.e. did **not** contain any sulfate ions at all. Therefore, the subject-matter of claim 1 was novel over D2.

- Novelty of the subject-matter of independent claims 10, 23 and 30 was not contested by the Respondent.

Inventive step

- Considering example 7 of D2 as closest prior art, the technical problem underlying the invention was to be seen in the provision of a method for preparing coated titanium dioxide pigments having improved gloss and/or durability.

- The examples of the patent in suit, as well as the experimental evidence and declarations submitted as documents D5, D6, D9, D10 and D15, showed that the gloss and the durability of a titanium dioxide pigment coated according to the method of preparation of claim 1 as granted, i.e. of a titanium dioxide pigment containing a first layer of sulfate-free zirconia on the titanium dioxide and a second layer of sulfate-free alumina on the zirconia layer, were better than those of a similar titanium dioxide pigment containing a first layer of sulfate-containing zirconia. Moreover, the statistical analyses carried out in respect of the experimental data reported in documents D5, D6 and D10 confirmed with greater than 95% confidence that the improvements shown were significant. Considering the wealth of significant data provided by the Appellant, the contradictory results reported in the single experimental report D4 provided by Respondent, which also did not indicate the number of measurements carried out for any tested sample, were less meaningful and could not disprove that the effects invoked were actually achieved.

- Therefore, the technical problem underlying the claimed invention was convincingly solved by the method of preparation according to claim 1 as granted.

- The cited prior art did not contain any suggestion that the use of sulfate-free zirconium and aluminum compounds for forming sulfate-free zirconia and alumina coating layers onto a titanium dioxide pigment could bring about an advantage in terms of gloss and/or durability as compared to the use of sulfate-containing compounds leading to the formation of sulfate-containing coating layers. Hence, the skilled person would not have had any motivation to try using a sulfate-free zirconium compound in combination with the sulfate-free aluminum compound in the coating method disclosed in example 7 with the expectation of solving the technical problem posed.

- Therefore, the subject-matter of claim 1 as granted involved an inventive step.

- The arguments put forward with respect to the process claim 1 also applied to the subject-matter of the other independent process claim 10 as well as to the independent product claims.

- All the granted claims thus were thus novel and involved an inventive step over the cited prior art.

The **Respondent**'s counter-arguments can be summarised as follows:

Novelty

- Examples 6 and 7 of document D2 disclosed the use of an aqueous solution of zirconium carbonate complex in

order to provide a zirconia layer onto a titanium dioxide pigment. These examples neither mentioned that the aqueous solution of zirconium carbonate complex used was prepared or had to be prepared by reacting zirconium sulfate with an alkali metal or ammonium carbonate solution, as described in column 3 of document D2, nor that it contained sulfate ions. On the contrary, the skilled person would understand that the solution of zirconium carbonate complex used in the example was a conventionally purified product and did thus not contain sulfate ions as impurities. Therefore, examples 6 and 7 of D2 disclosed the use of a sulfate-free zirconium compound to coat a titanium dioxide pigment with a sulfate-free zirconia layer and were thus novelty-destroying for the subject-matter of claim 1 as granted.

Inventive step

- Experimental report D4 showed that a titanium dioxide pigment, prepared by a method similar to that used in example 1 of the patent in suit, and containing a first layer of sulfate-free zirconia and a second layer of sulfate-free alumina did not show an improved gloss under the indicated test conditions compared to a similar pigment, prepared in a similar way but comprising a sulfate-containing zirconia or alumina coating layer. Furthermore, the experiment described in D4 involving a 40% PVC alkyd paint had not been reworked by the Appellant according to its experimental report D5.

- The alleged improvements, supposedly to be corroborated by the experimental data contained in the patent in suit (Table 3) and in documents D5, D6, D9 and D10 submitted by the Patent Proprietor, had not been convincingly demonstrated. In fact, taking into account

the standard deviation to be applied in this kind of measurements (see e.g. also norms D11 and D12), quantitative improvements allegedly shown by the Appellant's tests, if any, were not significant, as shown, for example, in the Data Set 1 filed as document D7. In this respect, the consideration of the standard deviation had to be considered more relevant than the statistical analysis carried out by the Appellant in respect of its experiments.

- Therefore, the technical problem solved by the claimed invention had to be reformulated in a less ambitious way and could only be seen in the provision of a further method for providing coated titanium dioxide pigments.

- Since it was known from the cited prior art, for example from document D1, that sulfate-free zirconium or aluminum compounds could be used for coating a titanium dioxide pigment, it would have been obvious for the skilled person to use such sulfate-free compounds, for example a sulfate-free zirconium compound instead of the aqueous zirconium carbonate complex used in example 7 of document D2, in order to provide further coated titanium dioxide pigments with all the features of the pigments prepared according to the patent in suit.

- Therefore, the claimed subject-matter lacked an inventive step.

Reasons for the Decision

Admissibility of evidence filed in the appeal proceedings

1. In the course of the appeal proceedings, both parties to these appeal proceedings filed various further items of evidence.

1.1 The new documents were filed to further corroborate their respective positions as regards the pending issue of novelty over D2 (D13, D16, D19 and D20) or regarding the technical effects invoked in connection with the pending issue of inventive step (D14, D15, D17 and D18). No objections were raised as to the admissibility of these documents into the proceedings.

1.2 The Board thus decided to admit and consider these documents (Article 114(2) EPC and Articles 12 and 13 RPBA).

Main request (claims as granted)

2. Novelty

The only novelty objection maintained and substantiated by the Respondent at the oral proceedings was based on document D2 and directed against claim 1 (full wording under II, *supra*).

2.1 The method of claim 1 *inter alia* comprises (emphasis added) "*treating titanium dioxide with a* ***sulfate-free*** *zirconium compound to form a first layer on the titanium dioxide consisting of* ***sulfate-free zirconia***".

2.2 The disclosure of document D2

2.2.1 According to the methods disclosed in examples 6 and 7 of D2, a rutile (titanium oxide) pigment is treated in a first step with 1% by weight of ZrO_2, based on the pigment used, in the form of an aqueous solution of the

zirconium carbonate complex described in example 5, i.e. an aqueous solution of a zirconium carbonate complex of sodium containing 71 g of ZrO_2 per liter and having a pH-value of 9 (see column 7, lines 23 to 31 and 44 to 52, in combination with column 6, lines 1 to 5). The pigment of example 6 is then treated with a sodium aluminate solution whilst that of example 7 is treated with a waterglass solution and then with a sodium aluminate solution.

2.2.2 It is undisputed that the methods of preparation according to examples 6 and 7 both lead to the formation of a first layer consisting of zirconia on the titanium dioxide.

2.2.3 However, the cited examples do not expressly disclose i) whether or not the aqueous solution of zirconium carbonate complex used contains sulfate ions, and ii) whether or not the resulting zirconia layer is then sulfate-free, i.e. it does not contain any sulfate ions.

2.2.4 Moreover, there is no indication in D2 that the zirconium compound solution used or the zirconia layer obtained should be sulfate-free.

Document D2 (column 3, lines 20 to 28) teaches, however, that "*The alkaline aqueous aftertreatment solutions of zirconium carbonate complexes of the alkali metals or ammonium, which are suitable for the production of weather-resistant titanium dioxide pigments, may be produced for example by introducing zirconium* ***sulphate*** *solutions into alkali metal or ammonium carbonate solutions. Thus, it is possible, for example, to obtain zirconium carbonate complex solutions containing about 50 to 100 g of ZrO_2 per liter and having pH-values of around 9.*" (emphasis added).

2.2.5 As already indicated above, examples 6 and 7 do not mention the origin of the zirconium carbonate or the way in which the zirconium carbonate solution used was prepared. Taking the express teaching of document D2 (2.2.1 and 2.2.4, *supra*) into account, the Board therefore holds that the aqueous solutions of zirconium carbonate complex of sodium used according to examples 6 and 7 could actually contain sulfate ions.

2.2.6 In this connection, the Respondent also argued that the skilled person, in the absence of any specific indication to the contrary, would understand that the aqueous solution of zirconium carbonate complex used according to these examples was a conventionally purified product, not containing sulfate ions as impurities.

However, D2 does not suggest either that the aqueous solution of zirconium carbonate complex used in the examples had been or should be purified. Quite to the contrary, the passage of D2 cited under 2.2.4, *supra*, rather appears to indicate that an aqueous solution directly obtained by the zirconium sulfate reaction, i.e. without further purification, would be suitable for use in the process of examples 6 and 7.

2.2.7 Hence, in the Board's judgement, these examples do not even implicitly disclose in an unambiguous manner, and beyond any reasonable doubts, that the used aqueous solution of zirconium carbonate complex and the zirconia layer formed on the titanium dioxide are sulfate-free.

2.3 The subject-matter of claim 1 is, therefore, novel over D2, if only for this reason (Article 52(1) and 54 EPC).

2.4 Hence, there is no need to assess the issue (controversially debated) of whether or not a sulfate-free **alumina** layer is indeed formed on the (first) zirconia layer in the specific cases of examples 6 and 7 of D2; therefore, the further documents invoked in this connection (D13, D16, D19 and D20) do not need to be dealt with here.

2.5 Novelty of the subject-matter of the other independent claims 10, 23 and 30 was not disputed by the Respondent. The Board also sees no reason for calling it into question.

2.6 The Board thus concludes that the objections maintained with respect to novelty do not prejudice maintenance of the patent as granted.

Inventive step

3. At the oral proceedings, the Respondent essentially presented an objection and arguments with respect to method claim 1, which are thus addressed in detail first (points 8 to 9.5, *infra*).

4. The invention

4.1 The present invention concerns methods for preparing coated titanium dioxide pigments (see independent claim 1). In particular, claim 1 concerns a method for preparing a titanium dioxide pigment coated with a first layer consisting of sulfate-free zirconia and with a second layer comprising sulfate-free alumina.

4.2 The following is indicated in the description of the patent in suit (paragraphs [0011] and [0012]):

"*In general, inorganic coatings are known to improve durability, dispersibility and/or gloss of the titanium dioxide pigment. However, some coatings cause the formation of agglomerates that tend to cause difficulties in dispersing pigments into various compositions thereby reducing gloss and durability. Based on the foregoing, there is still a need for titanium dioxide pigments that have improved gloss and/or durability.*"

5. Closest prior art

5.1 Both parties agreed that document D2 and, in particular, example 7 thereof represents an appropriate starting point for the evaluation of inventive step. Considering the similarities between D2 and the patent in suit in terms of subject-matter concerned and goals to be achieved, respectively, the Board sees no reason to take a different stance in this respect.

5.2 Indeed, document D2 (column 2, lines 15 to 17; claim 1) is concerned, like the patent in suit, with processes for the production of titanium dioxide pigments with improved chalking resistance and gloss retention, i.e. durability, comprising coating the titanium dioxide with metal oxide(s). In particular, example 7 describes a process comprising the steps of treating a titanium dioxide pigment with a zirconium compound to form a first layer of zirconia onto titanium dioxide and then further treating the titanium dioxide pigment with waterglass and sodium aluminate.

6. The technical problem

According to the Appellant, the technical problem to be solved in the light of the closest prior art,

represented by example 7 of document D2, consists in the provision of a method for preparing coated titanium dioxide pigments having improved gloss and/or durability (as indicated in paragraph [0012] of the patent).

7. The solution

As the solution to this technical problem the patent in suit proposes the preparation method according to claim 1 which is characterised in particular in that

"*the titanium dioxide pigment is treated with a sulfate-free zirconium compound to form a first layer on titanium dioxide consisting of sulfate-free zirconia and then the zirconium treated titanium dioxide is treated with a sulfate-free aluminum compound to form a second layer comprising sulfate-free alumina*."

8. Success of the solution

Various items of evidence were submitted by the parties with regard to the question of whether or not the improvements invoked by the Appellant are actually achieved when performing the claimed subject-matter. As regards the evidence on file the Board observes the following.

8.1 The experimental data in the patent in suit

8.1.1 According to the patent in suit (paragraph [0049]; emphasis added) "*[t]he examples demonstrate improved gloss and/or durability of titanium dioxide pigments produced having essentially* ***no polyvalent anions (i.e. sulfate)*** *present during the surface coating process.*"

8.1.2 In particular, example 1 (paragraph [0050]) describes a process according to claim 1 as granted wherein a titanium dioxide pigment is treated with zirconyl chloride to form a first layer on titanium dioxide consisting of zirconia and then the zirconium treated titanium dioxide is treated with sodium aluminate to form a second layer comprising alumina. Although this is not expressly mentioned again in paragraph [0050] describing the exemplified process, there is no doubt that the compounds used and the layers so formed according to the invention are sulfate-free, as generally indicated in paragraph [0049].

This is also underlined once more in paragraph [0052], according to which (emphasis added) "[*v*]*arious commercially available sources of zirconia salts were used in the wet treatment process and the properties of the product produced using* ***$ZrOCl_2$*** *are compared (Table 2) with that from the* $ZrOSO_4$ *sources, i.e., the* ***sulfate-free*** *and sulfate containing versions.*".

8.1.3 Pigments A, B and C obtained using the sulfate-free zirconium compound were then compared with pigments D and E obtained using the sulfate-containing zirconium compound. What was compared was the measured gloss of an acrylic based paint at 40% PVC (pigment volume concentration) according to the most preferred method for determining gloss indicated in the patent in suit (see paragraph [0042]).

According to example 1, **Table 2**, the pigments A, B and C, prepared by using $ZrOCl_2$ (sulfate-free) show gloss values of 58, 58 and 59, respectively, whilst pigments D and E, prepared using $ZrOSO_4$ (containing sulfate), i.e. according to a method not falling within the ambit of claim 1 as granted, both show a gloss value of only 51.

This table thus shows a clear improvement in gloss for the pigments prepared according to the method of claim 1 as granted. These specific results were not contested by the Respondent.

8.1.4 In **Table 3** of example 1, a comparison is made between the gloss retention values, reflecting durability, of pigments A, B and C, and comparative pigment D, in an alkyd paint but under accelerated weathering conditions. The "*total unwashed gloss*" values for samples A, B and C are of 1016, 1009 and 1029, respectively, whilst the value for sample D is of only 993. The "*total washed gloss*" values for samples A, B and C are of 1065, 1048 and 1064, respectively, whilst the value for sample D is of 1047.

It can thus be gathered from these data that the unwashed gloss values for the samples containing pigments A, B and C prepared by a method according to claim 1 as granted are significantly better than the gloss value of comparative sample D. It was observed that the sample comprising comparative pigment D shows a total washed gloss value comparable with that of the sample comprising pigment B (1047 vs. 1048). However, for the Board, this isolated pair of comparative data is not sufficient to call into question that the coated pigments prepared according to claim 1 as granted, i.e. using sulfate-free zirconium and aluminum compounds show a better durability than sulfate-containing coated pigments.

For the sake of completeness, the Board remarks also that **Table 4** of example 2 shows further measurements performed using pigment A and two further pigments "F" and "F (second sample)", prepared by a method according

to claim 1 as granted described in example 1, and comparative pigment D. Pigments A, F and F (second sample) also showed better 40% PVC Acrylic Gloss values (52, 51 and 50, respectively) than pigment D (46), prepared using a sulfate-containing salt.

8.1.5 Therefore, for the Board, the comparative tests contained in the patent in suit show that pigments prepared by a method according to claim 1 and thus comprising a sulfate-free first zirconia layer and a second sulfate-free alumina layer have better gloss and durability than pigments prepared using a zirconia source comprising sulfate ions, i.e. by a method not falling under claim 1.

8.2 The Respondent's experimental report D4

8.2.1 According to D4, pigment samples I and II, prepared in a similar way as those of example 1 of the patent in suit, i.e. using a $ZrOCl_2$-solution, were compared with a sample III, prepared by a method using $ZrOSO_4$ instead of $ZrOCl_2$, and a sample IV, prepared by adding a sodium sulfate solution after the addition of sodium aluminate, the latter method apparently leading to the formation of coating layer comprising sulfate-containing alumina.

The so-obtained pigments were tested for gloss in a 40% PVC alkyd paint (Glanz HMG40) and in a 19% PVC acrylic paint.

8.2.2 However, this experimental report neither refers to measurements concerning the durability (gloss retention) of the pigments, nor to measurements of gloss in a 40% PVC acrylic paint, the latter being the preferred method for measuring gloss according to the patent in suit and having been used in obtaining the results shown in

Tables 2 and 4 of the patent.

Therefore, the results presented in D4 are not fully comparable and, hence, not suitable for calling into question the validity of the results shown in the patent in suit for durability and for gloss in a 40% PVC acrylic paint.

8.2.3 In the absence of evidence to the contrary, the Board thus holds that the experimental data in the patent in suit show convincingly that

- titanium dioxide pigments with sulfate-free zirconia and alumina coatings prepared by a method according to granted claim 1 have different characteristics than similar pigments prepared by a different method leading to the formation of a sulfate-containing layer(s) and

- titanium dioxide pigments prepared by a method according to granted claim 1 show improved gloss, at least in the paints used in the tests described in the patent in suit, i.e. using a 40% PVC acrylic paint, as well as improved durability.

8.2.4 Therefore, even accepting, *arguendo* but in the Respondent's favour, that the tests described in experimental report D4 do not show any improvement in gloss for pigments prepared by a method according to claim 1 as granted in the specific paints used in this report (40% PVC alkyd paint and 19% PVC acrylic paint), it remains undisputed that pigments prepared by a method according to claim 1 as granted display improved gloss and/or durability in the paints used in the tests described the patent in suit.

8.3 Appellant's experimental gloss data in D5, D6 and D10

8.3.1 D5 describes a re-working of the test described by the Respondent in D4 and carried out using a 19% PVC acrylic paint. The test for gloss on a 40% PVC alkyd paint (Glanz HMG40) was not repeated and was instead replaced by a test carried out using the preferred method of measurement according to the patent in suit, i.e. a test on a 40% PVC acrylic paint.

It is undisputed that all numerical values of gloss measured for samples I and II ("sulfate-free"), prepared by a method according to claim 1 as granted, are better than the values obtained for samples III and IV ("sulfate-containing"), prepared by a different method outside the invention (see Tables 1, 3 and 4).

Moreover, D5 reports, beside the standard deviation calculated for the single experiments, also a statistical analysis carried out in order to establish the statistical significance of these improvements in gloss values with greater than 95% confidence. According to this statistical analysis, pigments I and II, prepared by the method according to claim 1 as granted, show - with greater than 95% confidence - a significant improvement in gloss over comparative samples III and IV (see points 3.3 and 4.3 of D5).

8.3.2 D6 comprises a similar statistical analysis of (*inter alia*) the test results reported in Tables 2 and 4 of the patent in suit (see points 5 and 6). Also in this case, it was concluded that the samples prepared by the method according to claim 1 as granted show with greater than 95% confidence a significant improvement in gloss (points 4.3 to 4.6, 7 and 8).

8.3.3 In document D10 (points 7.3.1 and 7.3.2) the Appellant indicates that the measurement of gloss in a 40% PVC alkyd resin vehicle, like the test (Glanz HMG40) carried out by the Respondent in D4, was less reliable and thus less meaningful than the test carried out on a 40% acrylic paint as according to D5 and the patent in suit. This statement was not disputed by the Respondent that nevertheless maintained that its own tests were valid.

In D10 the Appellant also repeated the tests presented in Table 3 of D5 (20°Gloss reading in a 19% PVC acrylic paint) by increasing the number of readings for each sample (see point 11). Even though the values obtained on these samples more than two years after the tests of document D5 are quite different from those reported in Table 3 of D5, they still show that samples I and II prepared according to the method of claim 1 as granted have a statistically significant improvement in gloss compared to sulfate-containing samples III and IV (see points 12.1 to 12.6). The significance of the difference in gloss was confirmed by the further declaration D15. For the Board, even though the values obtained in D10 are different from those of D5, there is no reason to assume that they concern pigments having different coating layers than those tested in D5. Therefore, there is no reason for disregarding the values reported in D10. D10 clearly shows again the superiority in gloss of samples I and II over comparative, sulfate-containing samples III and IV.

8.4 Probative force of documents D4, D5 and D10

8.4.1 The Board remarks that

- whereas the Appellant, in carrying out the tests as described in D5, measured each paint sample at three

separate times (see points 3.1 and 4.1), and even increased the number of readings in D10 (point 11),
- the Respondent did not even indicate the number of readings for each sample tested according to D4.

8.4.2 Moreover, the statistical evaluation of the readings carried out by the Appellant in its experimental reports was not reworked by the Respondent, which only stated that the consideration of the standard deviation would be more significant for the evaluation of the experimental results than the statistical analysis of the Appellant. Even though the consideration of the standard deviation is undoubtedly of importance in the measurements of gloss, as confirmed by norms D11 (point 11) and D12 (point 10 and Table D.1), the Respondent's statement is, however, not supported by further evidence. Moreover, it is directly apparent that the statistical analysis carried out by the Appellant takes also into consideration standard deviation values (see, for example, D5, Tables 2, 5 and 6 and D10, point 12.5.

8.4.3 For the Board, considering the wealth of experiments and the more complete information provided, the Appellant's test reports have a higher probative force and are thus more convincing than those of the Respondent.
The Respondent's evidence is thus not sufficient for diminishing substantially the cogency of the experimental evidence and statistical analyses submitted by the Appellant/Patent Proprietor.

Consequently, the Board is satisfied that the Appellant has convincingly demonstrated that pigments prepared by a method according to claim 1 as granted have improved gloss.

8.5 Appellant's experimental reports D6 and D9 regarding durability and Respondent's document D7

8.5.1 In document D6 (see points 3.1 to 3.3) the Appellant listed as Data Set 1 the complete set of readings for the durability values of samples A, B, C and D reported in Table 3 of the patent in suit. Moreover, it provided further Data Sets 2, 3 and 4 (points 3.4).
A statistical analysis of the obtained values show that with greater than 95% confidence the pigments prepared according to claim 1 as granted have improved durability, expressed as total washed and unwashed gloss (see points 4.4 to 4.6). This is also apparent from the graphical representation of the tests filed as D9.

8.5.2 The Respondent filed as document D7 an overview of the durability test results presented in D6 for Data Set 1 arguing that considering the standard deviations, no improvement in durability could be seen for the samples prepared according to claim 1 as granted.

8.5.3 Also in this case, the Board notes that the statistical evaluation of the readings carried out by the Appellant was not reworked or contested as such by the Respondent and that the Respondent's statement that the consideration of the standard deviation would be more significant for the evaluation of the experimental results than the statistical analysis of the Appellant is not supported by further evidence.

It was noted by the Respondent that a few of the individual gloss values measured during the durability test do not show any apparent difference in gloss between samples prepared according to the invention and comparative samples. This can be derived, for example, from the crossings of the respective curves shown in

document D9.

However, for the Board, such isolated data points do not diminish substantially the probative value and cogency of the results reported in documents D6 and D9, taken as a whole, which show that the sulfate-free coated pigments prepared according to claim 1 as granted have generally - with greater than 95% confidence - improved durability than comparative sulfate-containing coated pigments.

8.6 Further evidence filed by the Appellant

Since claim 1 as granted does not require the titanium dioxide pigment base to be sulfate-free, the additional documents D14, D17 and D18, referred to on this point in writing by the parties, do not need to be dealt with here and in the following.

8.7 Conclusion regarding the success of the solution

Considering the overall experimental evidence available the Board concludes that it was credibly shown that pigments prepared according to granted claim 1 indeed display improved gloss and/or durability compared to coated titanium oxide pigments wherein the zirconia layer is not sulfate-free.

Therefore, for the Board, the technical problem posed (see point 6, *supra*) is successfully solved by the method of claim 1 as granted.

9. (Non-)obviousness of the solution

9.1 As already emphasised above (points 2.1, 2.2.4 and 2.2.7), the method of claim 1 as granted requires that a

titanium dioxide pigment is coated with a first layer consisting of sulfate-free zirconia by treatment with a sulfate-free zirconium compound, whereas example 7 of D2 fails to disclose that the zirconium compound used in the treatment of the titanium dioxide is sulfate-free or that the zirconia layer obtained according to that process is sulfate-free.

The following considerations regarding inventive step are based, for the sake of argument only, but in favour of the Respondent, on the assumption that the second layer formed following the method of example 7 of D2 indeed comprises sulfate-free alumina (see point 2.4 above). Accordingly, the method of example 7 of document D2 is considered to differ from the method of claim 1 as granted only in that it does not disclose the treatment of a titanium dioxide pigment with a sulfate-free zirconium compound to form a first layer of sulfate-free zirconia on titanium dioxide.

9.2 It is undisputed that the use of zirconium chloride for coating titanium dioxide pigments was known from the prior art (see, for example, document D1, page 4, lines 6 to 13).

9.3 However, it is also undisputed that the prior art does not contain any suggestion or teaching that the use of sulfate-free zirconium compounds (for example, sulfate-free zirconium chloride), or sulfate-free compounds in general, for coating titanium dioxide could lead to improved gloss and/or durability of titanium dioxide pigments so-obtained.

9.4 Therefore, the skilled person would not have found in the prior art any motivation or hint for implementing the process disclosed in the closest prior art,

represented by example 7 of document D2, by using a sulfate-free zirconium compound to form a first layer consisting of sulfate-free zirconia, before treating the pigment with a sulfate-free aluminum compound to form a sulfate-free alumina containing layer, with the expectation of obtaining a titanium dioxide pigment having improved gloss and/or durability.

9.5 In the Board's judgement, the subject-matter of claim 1 thus involves an inventive step (Articles 52(1) and 56 EPC).

10. Inventive step - Independent claims 10, 23 and 30

10.1 At the oral proceedings, the Respondent did not maintain or substantiate further inventive step objections regarding, more specifically, the subject-matter of the other independent claims. The Board's findings in respect are as follows.

10.2 Claim 10 (wording under II, *supra*) concerns a method of preparing coated titanium dioxide pigments differing from that of claim 1 in that titanium dioxide is first wet treated with a sulfate-free aluminum compound to form a first layer of sulfate-free alumina and is then treated with a sulfate-free zirconium compound to form a sulfate-free zirconia layer. Compared to the method of claim 1, the order of the layers coated onto the titanium dioxide base pigment is thus reversed.

10.2.1 For the Board, also for this alternative method of the invention, the closest prior art and the technical problem posed remain those indicated in points 5 and 6, *supra*.

10.2.2 Absent any argument, let alone evidence, to the contrary, the Board has no reason to doubt that the improvements in gloss and durability convincingly shown with respect to the pigments prepared according to the method of claim 1 (8.4.3 and 8.5.3, *supra*) will also be displayed by pigments prepared according to the method of claim 10, which also contains a sulfate-free zirconia layer, as indicated more generally on page 4, lines 55 to 56 and page 5, lines 27 to 28 of the patent in suit.

Therefore, the Board accepts that the technical problem posed is also convincingly solved by the method of claim 10.

10.2.3 Moreover, in order to arrive at the subject-matter of claim 10, the skilled person would not only have to use a sulfate-free zirconium compound but would additionally have to reverse the order of addition of the zirconium and aluminum compounds in the method described in example 7 of document D2.

The Board's rationale leading the Board to the conclusion that the method according to claim 1 (point 9.4, *supra*) is not obvious thus applies analogously (and even more so) to the subject-matter of claim 10. In the Board's judgement, the subject-matter of claim 10 is is thus not obvious either.

11. Independent product claims 23 and 30

11.1 Claims 23 and 30 (see point II) both concern a "*sulfate-free titanium dioxide pigment*" comprising a titanium dioxide base treated *inter alia* with sulfate-free aluminium and zirconium compounds.

11.2 The Board holds that, absent specific objections or arguments, let alone evidence, to the contrary, the rationale leading the Board to acknowledging an inventive step in respect of claims 1 and 10 also applies analogously to the subject-matters of independent claim 23 and 30, which are thus not obvious either.

12. Conclusion

The Board thus concludes that the subject-matter claimed in the patent as granted involves an inventive step (Article 52(1) and 56 EPC).

Order

For these reasons it is decided that:

The decision under appeal is set aside.

The patent is maintained as granted.

The Registrar: The Chairman:

D. Magliano B. Czech

Decision electronically authenticated