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D E C I S I O N
of 4 April 2000

Case Number: T 0743/96 - 3.2.2

Application Number: 86306622.1

Publication Number: 0228151

IPC: C23C 22/36

Language of the proceedings: EN

Title of invention:

Acidic, aqueous phosphate-coating solutions for use in a process for phosphate-coating metal surfaces

Patentee:

HENKEL CORPORATION (a Delaware Corp.)

Opponent:

Metallgesellschaft AG

Headword:

-

Relevant legal provisions:

EPC Art. 54

Keyword:

"Novelty (no)"

Decisions cited:

T 0279/86, T 0026/85

Catchword:

-



Case Number: T 0743/96 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 4 April 2000

Appellant: Metallgesellschaft AG
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Representative: -

Respondent: HENKEL CORPORATION (a Delaware Corp.)
(Proprietor of the patent) The Triad, Suite 200, 2200 Renaissance
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 13 June 1996
rejecting the opposition filed against European
patent No. 0 228 151 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: W. D. Weiß
Members: R. Ries
J. C. M. De Preter

Summary of Facts and Submissions

- I. European patent No. 0 228 151 was granted on 22 December 1993 on the basis of European patent application No. 86 306 622.1.
- II. The granted patent was opposed by the present appellant on the grounds that its subject matter lacked novelty and inventive step with respect to the state of the art (Article 100(a) EPC).
- III. With its decision posted on 13 June 1996 the Opposition Division held that the grounds of opposition did not prejudice the maintenance of the patent in the form as granted and rejected the opposition.
- IV. On 8 August 1996 the appellant (opponent) lodged an appeal against the decision of the Opposition Division and the appeal fee was paid the same day. The notice of appeal was followed by the statement of grounds submitted with letter (by telefax) of 11 October 1996. Of the prepublished documents cited during the opposition and appeal proceedings, the following documents are considered as being pertinent:
- D1: EP-A-0 106 459
- D7: Amtsblatt der Europäischen Gemeinschaften
30.08.80, No. L 229, pages L229/11 to L229/24
- D8: Letter of 31 October 1996 Magistrat der Stadt Wien
(two pages)
- D9: Letter of 18 October 1996 Brusselse Intercommunale
Watermaatschappij, (two pages)

D10: Letter of 15 October 1996 AB YTTEKNIK, Sweden,
(five pages)

D11: Schweizerisches Lebensmittelbuch, Zweiter Band-
Spezieller Teil, fünfte Auflage, Kapitel 27A,
(five pages)

D12: Ost-Rassow, Lehrbuch der Chemischen Technologie,
volume 1, Joh. Ambrosius Barth Verlag, Leipzig
1965, page 139

D13: K. Höll, Wasser, Untersuchung, Beurteilung,
Aufbereitung, W. De Gruyter Verlag, Berlin, 1960,
pages 69, 71

V. Oral proceedings before the Board were held on 4 April
2000.

- The appellants requested that the decision under
appeal be set aside and that the patent be
revoked.

- The respondent requested that the appeal be
dismissed and the patent be maintained as granted,
or, as an auxiliary request, that the description
be amended as submitted at the oral proceedings.

VI. Claim 1 of the patent as granted reads as follows:

"1. An acidic aqueous phosphate solution, for use in
phosphating a metal surface, said solution containing
at most 0.2 g/l and preferably no chlorate ion as well
as the smallest practical concentration of chloride ion
never exceeding 0.5 g/l thereof, said solution
otherwise containing:

- (a) from 0.1 to 1.5 g/l of zinc ion,
- (b) from 5 to 50 g/l of phosphate ion,
- (c) from at least 0.8 to 4 g/l of manganese ion,
- (d) at least 0.05 g/l of a fluoride ion,
- (e) at least one of the following phosphating accelerators within the following concentrations:
 - (i) from 0.01 to 0.2 g/l of nitrite ion,
 - (ii) from 1 to 15 g/l of nitrate ion,
 - (iii) from 0.5 to 5 g/l of hydrogen peroxide (based on 100% H₂O₂);
 - (iv) from 0.05 to 2 g/l of m-nitrobenzene-sulfonate ion,
 - (v) from 0.05 to 2 g/l of m-nitrobenzoate ion, and
 - (vi) from 0.05 to 2 g/l of p-nitrophenol."

VII. The appellant argued as follows:

The ingredients of the phosphate solution given in document D1 on pages 3 and 4 correspond - apart from item e(i): the optional presence of 0.05 to 3 g/l chlorate ion - to those listed in claim 1 of the patent. The chlorate in document D1, however, represents merely one optional phosphating accelerator

among seven alternatives. As an example for a chlorate-free and chloride-free phosphating solution, document D1 discloses on page 13 a "concentrate" which fully complies with the "concentrate" described in the patent. It comprises nickel carbonate (II) and manganese nitrate rather than nickel chloride or manganese chloride. Having regard to the purity level of the ingredients making up the phosphating solution, generally "pure" raw materials are chosen which exhibit at most 0.1% chloride ions as an impurity. After diluting the "concentrate" with tap water and after adding 20% sodium nitrite as accelerator, the resulting phosphating solution falls within the claimed ranges, except for the amount of Mn-ion which is calculated to be 0.7 g/l and which is slightly outside the claimed range of 0.8 g/l to 4 g/l Mn-ion. Using this chlorate- and chloride-free "concentrate" in the same way as a basis composition for preparing the phosphating solutions No. 5 and 11, the water used to dilute the concentrate would be the only possible source for chloride ions. It is, however, standard practice to use tap water or deionised water in the phosphatization process, e.g. for washing and rinsing or making up the phosphating solution, as can be seen from document D1. Given that the chloride ion concentration in tap water is generally far below 500 mg/l, a fact that has been amply proven by documents D7 to D13, the compositional requirements of claim 1 are also fully met by examples No. 5 and 11 given in document D1. In this context, the respondent agreed in a letter submitted during the opposition proceedings concerning European patent No. 0 544 650 that the make-up water would have been selected from deionized water or tap water rather than "dirty" water and that chlorate and/or chloride are unlikely to be introduced in that way. The subject

matter of claim 1, therefore, lacks novelty with respect to the technical teaching given in document D1.

The respondent argued as follows:

Document D1 does not address the problem of "white spots" or "seediness". Although this phenomenon had been observed in the art for a long time, the origin of these excessively thick spots has been unknown. The present inventors have found that "white spotting" or "snow" can be avoided and low coating weights can be achieved provided that the amounts of chloride and chlorate ions are restricted to specific tolerable levels and provided that manganese and fluoride are both present in the phosphating solution. There is no clear and unmistakable direction in document D1 or any of the remaining documents to use narrowly restricted amounts of chloride and an essentially chlorate-free phosphating solution in order to avoid "white spotting", as proposed by claim 1 of the patent.

Even if some of the solutions claimed by the patent were generically encompassed by the teaching given in document D1, the technical features of claim 1 would not be specifically disclosed in D1. In particular, examples 5 and 11 of D1 fail to disclose the exact chloride amount present, since the ionic partner for manganese and/or nickel is not known. Even if demineralized water (i.e. chloride-free water) were used to prevent chloride contamination when making up the phosphatizing solution, chloride ions could be introduced into the solution for example by using manganese chloride or nickel chloride, as for instance proposed in document D1, or by decomposing chlorate. Given that there is no implicit or explicit teaching of

the chloride range in examples 5 and 11, the subject matter of claim 1 is not anticipated by the composition of these phosphating solutions.

Regarding the exemplifying chloride- and chlorate-free "concentrate" given on page 13 of document D1, the manganese content of 0.7 g/l falls outside the range specified in claim 1 of the patent and, therefore, the claimed phosphating solution is unambiguously distinguished from that given in document D1. The subject matter of claim 1 is, therefore, novel with respect to document D1.

Reasons for the Decision

1. The appeal is admissible.
2. *Novelty*

Like the patent in suit, document D1 is concerned with an acidic aqueous phosphating solution for treating e.g. car body steel components which include both an iron based and a zinc-based surface (cf. page 3, third paragraph bridging page 4, line 26), the solution containing:

- (a) from about 0.1 to about 2 g/l of zinc ion,
- (b) from about 5 to about 50 g/l of phosphate ion,
- (c) from about **0.2** to about 4 g/l of manganese ion,
(preferably 0.6 to 3 g/l manganese ion)

- (d) at least about 0.05 g/l of a fluoride ion, and
- (e) at least one of the following phosphating accelerators (conversion coating accelerator):
 - (i) from 0.05 to **3 g/l** (preferably 0.05-**1.9 g/l**, more preferably **0.2-1.5 g/l**) of **chlorate ion**;
 - (ii) from about 0.01 to about 0.2 g/l of nitrite ion,
 - (iii) from about 1 to about 10 g/l of nitrate ion,
 - (iv) from about 0.5 to about 5 g/l of hydrogen peroxide (based on 100% H₂O₂);,
 - (v) from about 0.05 to about 2 g/l of m-nitrobenzene-sulfonate ion,
 - (vi) from about 0.05 to about 2 g/l of m-nitrobenzoate ion, and
 - (vii) from about 0.05 to about 2 g/l of p-nitrophenol.

The phosphating solution claimed in the patent in suit differs from the known solution cited above only in the items emphasized by using bold letters and numbers.

Consequently, the phosphating solution disclosed in document D1 permits higher amounts of chlorate ion as an accelerator and is silent about the chloride ion concentration actually present to less than 0.5 g/l as does the patent at issue.

Notwithstanding that, document D1 specifically discloses phosphating solutions which are chlorate-free (examples 5 and 11) and additionally gives an exemplifying phosphating composition prepared by diluting a chlorate- and chloride-free "concentrate". It is noted in this context that the composition of the "concentrate" and the solution made up therefrom disclosed on page 13, paragraph 1 of document D1 fully complies with the one given in the patent in suit (see page 5, line 56 bridging page 6, line 6 and which is identified there to "be a solution of the invention". As regards the water quality used for diluting the "concentrate", the respondent no longer challenged that deionized water or tap water are used in the art for this purpose. Therefore, the diluting water has to be eliminated as a possible source for chlorate and chloride ions. In this context the appellant cited the respondent's submissions dated 25 March 1999 in the opposition proceedings against European patent No. 0 544 650. Thus, as agreed by the parties, the only distinction to the claimed solution lies in the manganese ion content of 0.7 g/l of the resulting aqueous phosphating solution which is outside the manganese ion range of 0.8 to 4 g/l claimed in the patent at issue.

Given this situation, the claimed phosphating solution could be regarded as being a "selection" from the broad variety of solutions disclosed in document D1. Such a selection of sub-ranges of numerical values from broader ranges is, however, only novel when each of the following criteria is satisfied (cf. Case Law of the Boards of Appeal of the European Patent Office, 3rd edition 1998, I-C, Chapter 5, in particular page 91, item 5.2):

- (i) the selected ranges are narrow in relation to those of the prior art;
- (ii) the selected sub-range should be sufficiently far removed from the preferred part of the known range (as illustrated for instance by the examples given in the prior art), and
- (iii) the selected sub-range should not be an arbitrarily chosen specimen from the prior art, i.e. not merely one way of carrying out the prior art teaching, but must provide a new invention (purposive selection).

A comparison between the composition of the claimed phosphating solution and the prior art D1 shows a high degree of overlap of the numerical ranges. Except for the restrictions to the amounts of chloride ions and chlorate ions, the claimed compositional limitations for the ions of zinc, phosphate, manganese, fluoride and for the various accelerators are the same as disclosed in document D1. The narrower lower limit of the manganese ion content (0.8 g/l) featuring in claim 1 of the patent at issue is also disclosed in claim 4 of document D1 as a preferred embodiment.

Although the appellant's argument is correct that the example given on page 13 of document D1 falls outside the claimed range by virtue of a manganese ion content of 0.7 g/l, this value nevertheless comes close to the claimed range of 0.8 g/l to 4 g/l Mn-ions. The sub-range claimed in the patent in suit is, therefore, not sufficiently far removed from the preferred embodiment of the prior art represented by the example on page 13 of document D1. Since according to the originally filed

documents of the application underlying the patent in suit manganese ion contents of down to 0.2 g/l are admitted, the Board is convinced that a phosphating treatment with the known solution containing 0.7 g/l can only result in a coating which is also free of "white spots" or "snow". No information whatsoever is found in the patent specification that a minimum of 0.8 g/l manganese ion has any effect on the white spot formation. Hence, the patent does not claim any effect which is not disclosed in the prior art.

In view of these considerations, neither of conditions (i) to (iii) which are the prerequisites for a "novel selection" is satisfied by the solution claimed in the patent in suit.

It is noted in this context that, according to the accepted jurisprudence of the EPO the attribute of novelty must not be given such a narrow interpretation that only what has been described already in identical terms is prejudicial to it. The teaching of a cited document is not confined to the detailed information given in the examples of how the invention is carried out, but embraces any information regarding the starting substances and the final products in the claims and the description enabling a person skilled in the art to carry out the invention (cf. T 0279/89). Although initially a skilled person focuses his attention on the claims and the examples of the relevant document, he would not restrict his study of the document solely to them, but would also have regard to the general description in his search for technical teaching relevant to the problem confronting him. Therefore, in assessing the novelty of an invention over the prior art in a case where overlapping ranges

of a certain parameter exist, it has to be considered whether a person skilled in the art would, in the light of the technical facts, seriously contemplate applying the technical teaching of the prior art document in the range of overlap. If it can be fairly assumed that he or she would do so, it must be concluded that no novelty exists (cf. T 0026/85).

This situation applies to the present case. Based on examples 5 and 11 disclosed in document D1, the skilled reader realizes that the phosphating films formed by chlorate-free solutions provide an excellent match in film weight, brine spraying test results, adhesion and resistance to spot rust and blisters on the coated surface, i.e. a combination of properties which is superior to that of phosphate films produced by the chlorate containing solutions 1 to 4, 6 to 10 and 12, 13. The assessment that chlorate-free solutions are more preferred in document D1 is confirmed by example 14 which specifically deals with the nature of the phosphate film formed by solutions 5 and 11 on galvanized steel plate and cold rolled steel plate. It is beyond doubt that the composition of the chlorate-free solutions 5 and 11 completely falls within the claimed ranges, although the ionic partner of Mn and Ni is not explicitly mentioned. Since the acidic aqueous phosphate solutions according to document D1 are usually prepared by diluting a "concentrate" and by adding further ingredients as needed (cf. D1, page 13, second paragraph), a skilled person would judiciously resort to the chloride- and chlorate-free "concentrate" disclosed on page 13 as a starting material for formulating the phosphating compositions Nos. 5 and 11. Based on these considerations, the list of anions and cations present in solution No. 5 and 11 has to be

taken as being exhaustive, and, consequently, the composition as being "essentially chloride-free". Moreover, those skilled in the field of phosphating technology appreciate that the presence of chloride ions in a phosphating solution promotes corrosion and, therefore, manganese nitrite and nickel carbonate are preferred in the "concentrate" rather than Mn- or Ni-chloride. Hence, the claimed aqueous phosphating solution given in claim 1 of the patent at issue is also anticipated by the solutions No. 5 and 11 disclosed in document D1.

In view of all these considerations, the subject matter of claim 1 of the main request and the auxiliary request lacks novelty with respect to the technical teaching disclosed in document D1.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar:

The Chairman:

V. Commare

W. D. Weiß