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Aktenzeichen / Case Number / N^o du recours : T 241/89 - 3.3.1

Anmeldenummer / Filing No / N^o de la demande : 83 201 058.1

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Bezeichnung der Erfindung: Detergent compositions

Title of invention:

Titre de l'invention :

Klassifikation / Classification / Classement : C11D 3/39

ENTSCHEIDUNG / DECISION

vom / of / du 14 August 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /

Titulaire du brevet :

Unilever NV

Unilever PLC

Einsprechender / Opponent / Opposant :

OI AKZO NV

OII Henkel KGaA

OIII Procter & Gamble (NTC) Ltd.

Stichwort / Headword / Référence : Detergent compositions/UNILEVER

EPÜ / EPC / CBE Article 83

Schlagwort / Keyword / Mot clé : "Insufficient disclosure - new parameter"

Leitsatz / Headnote / Sommaire



Case Number : T 241/89 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 14 August 1990

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.../...

Decision under appeal : **Decision of the Opposition Division of the European Patent Office dated 19 January 1988 posted on 23 February 1989 revoking European patent No. 0 101 113 pursuant to Article 102(1) EPC.**

Composition of the Board :

Chairman : K.J.A. Jahn

Members : R.W. Andrews

 G.D. Paterson

Summary of Facts and Submissions

- I. The mention of the grant of European patent No. 0 101 113, in respect of European patent application No. 83 201 058.1 filed on 18 July 1983, was announced on 28 May 1986 (cf. Bulletin 86/22).
- II. Notices of opposition, which were filed on 19 and 25 February 1987, requested the revocation of the patent on the grounds that its subject-matter lacked novelty and did not involve an inventive step. Opponents OII and OIII also alleged that the disclosure of the patent was insufficient.
- III. By a decision delivered orally on 19 January 1988, with written reasons posted on 23 February 1989, the Opposition Division revoked the patent. The Opposition Division held that the disclosure of the patent was insufficient since, from the information on page 2, lines 37 to 51, the skilled person would not understand the term "reactive Ti" to mean "distilled water-soluble Ti".
- IV. A notice of appeal was lodged against this decision on 7 April 1989 with payment of the prescribed fee.

In their statement of grounds filed on 12 June 1989, the Appellants contended that the expression "after removal of any non-reactive titanium present by ultra-centrifugation" can only be interpreted as meaning that the non-reactive titanium is separated and removed as and together with any other (in detergent terms "water insoluble") solids and that the reactive titanium (IV) analysis is carried out on the remaining liquid. Moreover, the fact that a solution is analysed is a requirement of the Plasma Emission Spectroscopy (PES) method.

The Appellants also argued that distilled water is the obvious and only suitable solvent in order to avoid the introduction of further trace metals and unnecessary changes in compound-forms.

According to the Appellants, one hour at 70 to 90°C is amply sufficient to dissolve the detergent and the amount of sample employed is governed by the correlation between ppm range of analysed metal and the detection limit capabilities of the analytical method. Ultra-centrifugation is used to avoid incomplete solid/liquid separation with the consequence of faulty results.

- V. The Respondents have contended that the disclosure is insufficient and that the arguments put forward by the Appellants do not overcome the objections of the Opposition Division.

- VI. The Appellants request that the decision under appeal be set aside and the patent maintained on the basis of the amended description and claims filed with the statement of grounds of appeal. The only independent claim of these amended claims reads as follows:

"Detergent composition comprising at least 3% by weight of a silicate, an inorganic persalt and an organic peracid precursor, characterised in that it contains not more than 3 mg/kg of reactive titanium (IV), based on the total weight of the composition."

The Respondents request that the appeal be dismissed.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. The disputed patent relates to a detergent composition comprising at least 3% by weight of a silicate, an inorganic persalt and an organic peracid precursor. In lieu of the inorganic persalt/peracid precursor combination, the composition may contain an organic peracid.

- 2.1 However, such low temperature bleaching detergent compositions are subject to variations in the stability of peracid system in solution, in spite of the presence of stabilising agents. The reduction in peracid stability causes a decrease in peracid concentration in the bleaching solution with consequent deterioration of bleach efficiency. According to the patent in suit, it has been discovered that this reduction in peracid stability is connected with the so-called "reactive titanium IV" present in the formulations. The expression "reactive titanium IV" is used to distinguish the titanium, which causes the problem, from the non-reactive titanium, which does not destabilise the peracid.

In the light of this finding, the present detergent compositions are characterised in that, based on the total weight of the composition, they contain not more than 3 mg/kg of reactive titanium (IV).

3. This appeal is solely concerned with the question of whether the disclosure of the disputed patent is sufficient to enable the skilled person to put the claimed invention into practice.

The only characterising feature of the claimed detergent compositions, which distinguish them from conventional low temperature detergent compositions, resides in the requirement that the amount of reactive titanium (IV) should be below a certain level.

- 3.1 Therefore, in the Board's judgement, in order that the disclosure could be considered to be sufficient, it is necessary that either the method used to determine the amount of reactive titanium (IV) present in a detergent composition is fully described in the disputed patent, or that the method should be so apparent to the skilled person that, in the light of his common general knowledge, a detailed description is unnecessary.

According to page 2, lines 46 to 51 of the disputed patent, the reactive titanium (IV) can be detected and its concentration can be measured by the use of the plasma emission spectroscopic (PES) method as described in Chemical Analysis, Volume 46, Trace Analysis, Spectroscopic Methods for Elements, edited by J.D. Winefordner, pages 142 ff. 1976. PES is a well-known technique of trace element analysis and, therefore, a detailed description of the apparatus or the method is totally unnecessary. However, it should be mentioned that, due to the high temperature of the plasma, this technique cannot distinguish between the various valency states of titanium. Therefore, the results obtained using this technique represent the total amount of the titanium in the sample. The expressions "reactive titanium" and "reactive titanium (IV)" are clearly synonymous (cf. page 2, line 44) and, therefore, Claim 1 should not be construed as being restricted to titanium in the tetravalent state, particularly since it is stated on page 2, lines 48 to 49 that the standard analytical method for reactive titanium analysis used is the PES method.

3.2 Before the amount of reactive titanium in a detergent composition can be determined, the non-reactive titanium must be removed by ultra-centrifugation (cf. page 2, lines 46 and 47). According to the Appellants' letter filed on 5 October 1987, any non-reactive titanium, which may be present in the detergent compositions, is separated by heating 25 g of the composition with 500 ml of distilled water at 90°C for one hour with stirring. The resulting mixture is allowed to cool and then transferred to a one litre measuring flask and made up to one litre with distilled water. A 20 ml sample of the well-mixed solution is centrifuged at a rotation of 13 000 rpm for ten minutes. The reactive titanium is determined by the PES method on a 5 ml sample removed from the clear upper layer resulting from the centrifugation.

Therefore, with respect to sufficiency, it is necessary to consider whether, in the absence of these specific details, the skilled person would be in a position to determine the amount of reactive titanium in any low temperature bleaching detergent composition falling within the terms of the preamble of the present Claim 1. In resolving this question, it should be borne in mind that the concept of reactive titanium in the context of the destabilisation of peracid in detergent formulations was introduced for the first time in the disputed patent.

According to the disputed patent, reactive and non-reactive titanium may be introduced into the formulation from various sources, the most important of which are enzyme encapsulates and impurities in silicates, including aluminosilicates (cf. page 2, lines 34 to 40). However, silicates are stated to be the major source of reactive titanium (cf. page 5, line 15). Furthermore, the non-reactive and reactive titanium are separated from each other by ultra-centrifugation (cf. page 2, lines 46 and 47).

- 3.3 Although the reference to centrifugal separation implies a solid-liquid separation, the disputed patent does not indicate either the solvent or the conditions used to prepare the solid-liquid mixture, which is to be subjected to this centrifugal separation. Furthermore, it is not clear from the disclosure of the patent whether the analysis for the reactive titanium is to be carried out on the resulting solid or supernatant liquid.
- 3.4 However, before deciding whether to subject the liquid or solid to analysis, the skilled person must decide how to treat the detergent composition to obtain the material from which the non-reactive titanium is to be removed by centrifugation. The disputed patent is completely silent on this point and the skilled person's common general knowledge would be of no assistance to him since he is encountering the concept of reactive and non-reactive titanium for the first time.

Although it is true that the present low-temperature bleaching detergent compositions would be used in the presence of water at temperatures up to about 60°C, this knowledge is not directly relevant to the skilled person who wishes to separate reactive and non-reactive titanium from each other in order to determine the amount of reactive titanium in the sample. Since the skilled person is aware that practically all titanium compounds are only soluble in a strongly acid medium, distilled water would not be the obvious and only suitable solvent for separating non-reactive and reactive titanium from each other.

- 3.5 The disputed patent is equally silent on the conditions employed to separate the two types of titanium. In the absence of any knowledge or disclosure with respect to any differences between non-reactive and reactive titanium, the skilled person would be unable to envisage those conditions

required to achieve a complete separation of the two forms of titanium.

In view of the fact that the skilled person would not be in a position to prepare the material from which the non-reactive titanium should be removed by ultra-centrifugation, it is immaterial whether, on the basis of the disclosure and his common general knowledge, he could deduce that the analysis for reactive titanium is carried out on the supernatant resulting from this operation.

4. Therefore, in the Board's judgement, the disclosure of the patent in suit is insufficient insofar as the method for determining the amount of reactive titanium (IV) present in a low temperature bleaching detergent composition is concerned, particularly since this new parameter is the only feature which serves to distinguish the present compositions from prior art ones. Under these circumstances, the drafter of a patent specification cannot rely on a reference to an analytical method and a vague reference to separation by ultra-centrifugation to satisfy the requirements of Article 83 EPC.

Order

For these reasons, it is decided that:

The appeal is dismissed.

The Registrar:



M. Beer

The Chairman:



K.J.A. Jahn