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Bezeichnung der Erfindung: Detergent compositions

Title of invention:

Titre de l'invention :

Klassifikation / Classification / Classement : C11D 1/62

ENTSCHEIDUNG / DECISION

vom / of / du 19 October 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /

Titulaire du brevet :

The Procter & Gamble Company et al

Einsprechender / Opponent / Opposant :

Unilever PLC/NV

Stichwort / Headword / Référence : STW-detergents/PROCTER

EPÜ / EPC / CBE Article 56

Schlagwort / Keyword / Mot clé :

"inventive step - (yes, after amendment of claims)"

Leitsatz / Headnote / Sommaire



Case Number : T 100/88 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 19 October 1990

Appellant :
(Proprietor of the
patent 01)

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.../...

Decision under appeal : Decision of the Opposition Division of the European Patent Office of 15 October 1987 posted on 15 December 1987 revoking European patent No. 0 026 528 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn

Members : P. Krasa

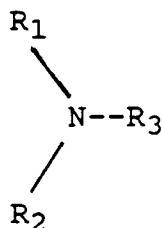
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Summary of Facts and Submissions

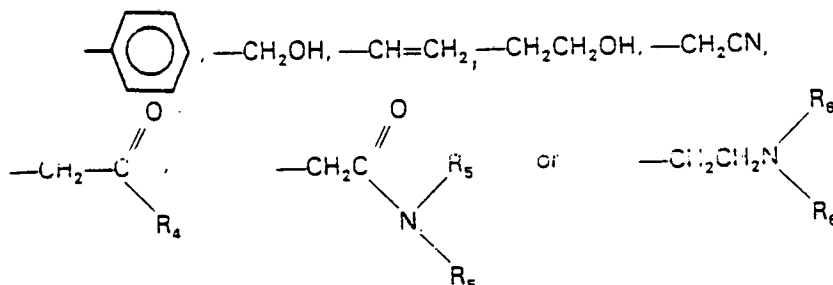
I. European patent No. 26 528 was granted to Procter and Gamble Company et al. with six claims. Claim 1 read:

"A particulate textile softening detergent composition comprising by weight of the composition:

- (a) 3%-30% of an anionic surfactant;
- (b) 1%-25% of a water insoluble tertiary amine having the general formula:-



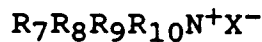
wherein R_1 is a C_{10} - C_{26} alkyl or alkenyl group R_2 is as R_1 provided that, where R_1 is a C_{20} - C_{26} alkyl or alkenyl group, R_2 is either a C_{20} - C_{26} alkyl or alkenyl group or is a C_1 - C_7 alkyl group, and R_3 has the formula $-CH_2-Y$ wherein Y is H, C_1 - C_6 alkyl,



wherein R_4 is a C_1-C_4 alkyl group, each R_5 is independently H or C_1-C_4 alkyl and each R_6 is independently H or C_1-C_{20} alkyl;

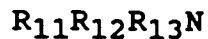
- (c) from 10% to 80% of a detergent builder salt, such that the pH of a 0.5% by weight aqueous solution of the composition is in the range from 8.5-11, said composition optionally comprising from 1.5% to 35% of an impalpable smectite-type clay having an ion exchange capacity of at least 50 meq. per 100 g, together with other components that are usually found in laundry detergents, characterised in that the composition also comprises
- (d) from 0.5% to 10% of a water soluble cationic compound selected from:-

- (i) quaternary ammonium compounds of formula:-



wherein R_7 is a C_8-C_{16} alkyl, each of R_8 , R_9 and R_{10} is independently C_1-C_4 alkyl or hydroxy alkyl, benzyl or $-(C_2H_4O)_xH$ where x has a value from 2 to 5, not more than one of R_8 , R_9 or R_{10} being benzyl and X^- is an anion; and

- (ii) aliphatic amines of general formula:-



wherein R_{11} is C_8-C_{14} alkyl, R_{12} and R_{13} are independently hydrogen, C_1-C_4 alkyl or benzyl, and water soluble salts thereof;

provided that the molar ratio of component (d) to component (a) does not exceed 1:1. The percentage weight of components (a), (b), (c) & (d) together with that of any said optional components totalling 100% by weight of the composition."

- II. A notice of opposition was duly filed by Unilever Plc. N.V. within the prescribed period (Art. 99 EPC).

The grounds of opposition were that all the claims lacked inventive step (Article 100(a) EPC) and, furthermore, that the patent did not sufficiently disclose the method of performing the invention (Article 100(b) EPC).

The opposition was based, *inter alia*, on

- (1) GB-A-1 514 276 and
- (3) DE-A-2 747 726.

After expiry of the opposition period

- (2) EP-A-225

was filed.

- III. The Opposition Division revoked the patent by a decision delivered orally on 15 October 1987, with written reasons posted on 15 December 1987.

In its decision, it held that it was the aim of the patent in suit to improve the softening properties of a detergent composition as described in document (1) without impairing its cleaning properties and that this problem was solved by the addition of a water-soluble quaternary ammonium compound or a water-soluble salt of an aliphatic amine to

the detergent compositions already known from (1). This solution was held to be obvious for a skilled person in view of (2), because it was known from that document that water-soluble quaternary ammonium compounds - in contrast to water-insoluble ones - did not depress the cleaning performance of the anionic surfactants present. The water-soluble quaternary ammonium surfactants of (2), being known softeners, were considered available to the skilled person for addition in amounts falling within the scope of Claim 1 of the impugned patent, to the detergent compositions known from (1) with the expectation of better softening effects without concomitant loss of cleaning performance. The use of a clay component, at that time only an optional feature of Claim 1, was held to be an obvious modification for a man skilled in the art.

The objection under Article 100(b) EPC was dismissed as the description contained sufficient information enabling a skilled man to prepare the compositions claimed.

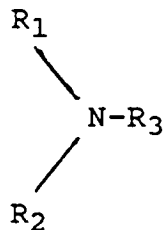
- IV. An appeal was lodged against this decision on 19 February 1988 and the prescribed fee was paid. A Statement of Grounds of Appeal was filed on 18 April 1988 together with amended claims making the optional clay ingredient an essential feature. In a Communication the Board indicated, *inter alia*, that these claims could be in conflict with Article 123(3) EPC. The Appellant filed a revised Claim 1 in response to this communication.

The current set of Claims 1 to 4 was submitted by the Appellant in the course of the Oral Proceedings held on 19 October 1990, in response to comments made by the Board

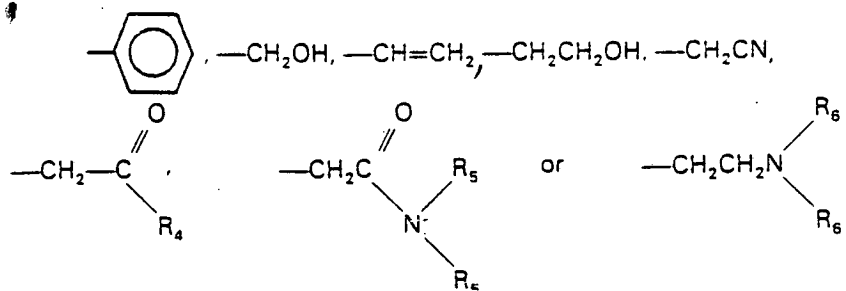
in respect to the previous draft. Claim 1 is the only independent claim and reads (after amendment of an obvious clerical error at the end of paragraph (a)):

"A particulate textile softening detergent composition comprising by weight of the composition:

- (a) 5%-15% of an anionic surfactant selected from water soluble salts of alkyl benzene sulfonates, alkyl sulfates, paraffin sulfonates, alpha olefin sulfonates and soaps;
- (b) 1%-6% of a water insoluble tertiary amine having the general formula:



wherein R_1 represents a C_{12} - C_{22} alkyl group, R_2 is the same as R_1 and R_3 has the formula $-CH_2-Y$ wherein Y is H, C_1 - C_6 alkyl,



wherein R_4 is a C_1 - C_4 alkyl group, each R_5 is independently H or C_1 - C_4 alkyl and each R_6 is independently H or C_1 - C_{20} alkyl;

- (c) from 10% to 80% of a detergent builder salt such that the pH of a 0.5% by weight aqueous solution of the composition is in the range from 8.5-11, said composition optionally comprising other components that are usually found in laundry detergents, characterised in that the composition also comprises
- (d) from 0.5% to 3% of a water soluble quaternary ammonium compound of formula:



wherein R_7 is a C_8-C_{14} alkyl, each of R_8 , R_9 and R_{10} is independently C_1-C_4 alkyl or hydroxyethyl and X^- is an anion; and

- (e) from 4% to 12% of an impalpable smectite-type clay mineral of particle size less than $50 \mu m$ and having an ion exchange capacity of at least 50 meq per 100g, said clay mineral being selected from sodium and calcium montmorillonite, sodium saponite and sodium hectorite

provided that the molar ratio of component (d) to component (a) does not exceed 1:1, the percentage weight of components (a), (b), (c), (d) and (e), together with that of any said optional components totalling 100% by weight of the composition, wherein any optional components comprise nonionic and zwitterionic surfactants, bleaching agents and organic precursors therefor, suds suppression agents, soil suspending and anti-redeposition agents, enzymes, optical brighteners, colouring agents and perfumes."

The Appellant's arguments can be summarised as follows:

The Opposition Division's finding that the quaternary ammonium compounds of the patent in suit were known softeners was wrong.

The use of a clay component, which is now an essential feature, is neither disclosed in (1) nor in (2). Thus any conceivable combination of the disclosures of (1) and (2) would, of necessity, still fail to disclose or hint at this particular feature. Document (3) is the only document that mentions a smectite clay, but this is concerned with the incorporation of perfumed organic carrier substance particles into liquid compositions and, thus, with a product totally different from that of the patent in suit. Therefore, the Opposition Division's decision that the clay component was an obvious modification was unsupported by the documents cited, and was, therefore, ill-founded.

The compositions claimed also show an improvement of the fabric softening effect which, according to the Appellant, is demonstrated by the comparative experiments submitted with the Statement of Grounds of Appeal.

V. The Respondent submitted additional citations, inter alia:

(4) GB-A-1 418 720

(5) van Olphen "An Introduction to Clay Colloid Chemistry", Interscience Publishers, 1963, pages 165, 166, and

(6) Schertzer, Kernan, JMRS 27, 261-282 (1985),

and argued as essentially follows:

It is known from (5) that clay adsorbs cationic surfactants. While at low levels of adsorption hydrophobic particles result therefrom with a tendency to flocculate in aqueous dispersion, positively charged particles are formed in the presence of excess cationic surfactants which are hydrophilic and dispersible in water. In the Examples of the patent in suit and in the experimental evidence submitted by the Appellant, the cationic surfactant was used in excess of the exchange capacity of the clay and, thus, the enhanced deposition of clay on the negatively charged fabric in the presence of excess cationic surfactant was to be expected and was also already suggested in (4).

The experimental data submitted by the Appellant did not establish inventive step as they were not conclusive, in that the assessment of the softening effects achieved by them was on a Scheffe-scale, which, being subjective, produces variable and hence inconclusive results.

- VI. In a written reply, the Appellant conceded that these data per se did not demonstrate a statistically significant effect. At the Oral Proceedings, the Appellant pointed out that the compositions of (1) cannot be commercialised since they are extremely sticky owing to the high amount of tertiary amine present.
- VII. The Appellant requested maintenance of the patent on the basis of the set of claims filed in the course of the Oral Proceedings on 19 October 1990; the Respondent requested the dismissal of the appeal.
- VIII. At the end of the Oral Proceedings, at which the duly summoned Respondent was not represented, the Chairman announced the Decision of the Board to allow the appeal.

Reasons for the Decision

1. The appeal is procedurally admissible.
2. There are no objections under Article 123(2) EPC to any of the claims because they are supported by the application documents as originally filed:
 - 2.1 - component (a): The anionic surfactants, which can be present in the composition, are limited to five particular groups of anionic surfactants. Support can be taken from page 6, lines 9 to 17 and page 8, lines 8 to 10 of the application documents as originally filed, corresponding to page 4, lines 3 to 7 and lines 40 to 41 of the patent in suit;
 - component (b): The present definition of the water insoluble tertiary amines is supported by page 8, lines 11 to 30 together with page 9, lines 31 to 33 of the application documents originally filed, corresponding to page 4, line 44 to page 5, line 14 together with page 5, lines 52 and 53 of the disputed patent;
 - component (c): The detergent builder salt and its amount are the same as in Claim 1 as granted and were disclosed as an optional feature already in the original application documents, page 11, line 32 together with page 19, lines 10 to 13;
 - component (d): The numerical range and scope of water soluble quaternary ammonium compounds is limited in as far as R₇ is now C₈-C₁₄ alkyl instead of C₈-C₁₈ alkyl (R₈, R₉, R₁₀ remaining unchanged) and was disclosed on

original page 10, lines 7 to 14 together with page 11, lines 22 to 26, corresponding to page 6, lines 41 to 48 and page 7, lines 12 to 15 of the patent in suit; aliphatic $R_{11}R_{12}R_{13}N$ amines were deleted;

- component (e): The impalpable smectite-type clay mineral was disclosed on page 12, lines 24 to page 13, line 6 together with page 17, lines 6 to 10 of the application documents as originally filed, corresponding to page 7, lines 35 to 46 together with page 9, lines 9 to 11 of the patent;
- the specification of the optional components under heading (c)) finds its support in the paragraph bridging pages 11 and 12 of the originally filed application documents, corresponding to page 7, lines 20 to 22 of the patent in suit.

2.2 In present Claim 1 the definitions of the components anionic surfactants (a), water insoluble tertiary amines (b), and water soluble quaternary ammonium compounds (d) are restricted as compared with Claim 1 as granted, both in respect to their concentration ranges and to the range of chemical compounds falling within the scope of the respective definitions. Such limitations were already present in the claims submitted by the Appellant together with the Statement of Grounds of Appeal. The Respondent objected, under Article 123(3) EPC, against the claim thus amended and it may be assumed that the present version of Claim 1 would not overcome his objections. In his submission, compounds which were no longer encompassed by the limited definition of component (a), (b) or (c) fall within the scope of the expression "other components that are usually found in laundry detergents" and, thus, were no longer subject to the concentration limits for the respective components in the claims as granted. Therefore,

compositions were now covered by the amended claims which were not within the scope of the claims as granted, thereby contravening Article 123(3) EPC.

However, this argument has to be rejected by the Board because the expression "other components that are usually found in laundry detergents" was defined as meaning optional ingredients in the application documents as originally filed (see page 11, lines 27 to 30). Optional ingredients of a composition are normally components which are different from and may be present in addition to the essential active ingredients of such a composition. In the present case, the definition of these optional ingredients remained unchanged (see EP-B-26 528, page 7, lines 17 to 19), apart from the omission of smectite-type clay. These clays are now an essential component of the composition of Claim 1. This amendment, however, has no bearing on the issue. Therefore, the "other components that are usually found in laundry detergents" of the current claims cannot be construed as encompassing compounds which were originally never within the scope of this definition simply because these compounds were deleted from the essential components (a), (b) and (d) by way of limitation.

- 2.3. Dependent Claim 2 is identical with Claim 2 as granted. Dependent Claim 3 differs from Claim 3 as granted by a linguistic adaptation to Claim 1 and the amendment of a clerical error: "wherein the water-soluble cationic compound (d) is selected from quaternary ammonium salts in which" was changed to "wherein the water soluble quaternary ammonium compound is selected from salts in which". Dependent Claim 4 corresponds to Claim 5 as granted and is limited to a range which was given as preferred range in said former Claim 5.

Thus, all Claims 1 to 4 satisfy the requirements of Article 123 EPC.

3. The patent in suit concerns a particulate textile softening detergent composition which imparts softness to the fabrics laundered with it. The Board considers, as did the Opposition Division, that document (1) represents the closest prior art. This document is concerned with laundering compositions comprising water-insoluble tertiary amines which exert softening effects for the fabrics treated. These tertiary amines have preferably two alkyl groups with 12 to 22 carbon atoms and are used in amounts of 6.5 to 35% by weight of the total laundry composition. See page 2, lines 121 to 123, Examples 33 to 35 on pages 7 and 8 and Claim 21.

The compositions of (1) are, according to the Appellant's uncontested and credible submission, sticky; this causes the disadvantage that granular compositions cannot be achieved with compositions comprising 6.5% by weight or more of tertiary amine.

In the light of this closest prior art, the technical problem underlying the patent in suit can be seen to be the provision of laundry detergent compositions which overcome the above disadvantage of stickiness maintaining simultaneously about the same cleansing and softening performance as the respective compositions known from (1).

According to the patent in suit, this technical problem is solved by reducing the contents of tertiary amine and simultaneously adding smectite-type clay and quaternary ammonium compounds as specified in Claim 1.

The Board is satisfied that the above-defined technical problem is effectively solved (cf. also the Examples).

4. After examination of the cited documents, the Board has reached the conclusion that the subject-matter of the disputed patent is novel and since this issue was not contested, there is no need to give detailed reasons for this finding.

5. It still remains to decide whether the statutory requirement of inventive step is met by the claims as they now stand amended.

- 5.1 It is clear from (1) that tertiary amines, especially those with two alkyl groups having 12 to 22 carbon atoms can be utilised advantageously in so-called STW-laundry detergent compositions (STW = softening through wash) which confer a softening effect on fabrics without the need to add a softening agent to the water with which the fabric is rinsed after washing (see (1), page 1, paragraph bridging both columns).

For solid laundry detergent compositions comprising the tertiary amine and an anionic detergent, an amount of 6.5% by weight to 35% by weight is disclosed in Claim 21 of (1); in the examples 33 to 35 of (1), the only examples concerned with solid STW laundry detergent compositions, 10% by weight and 7.5% by weight (twice) are disclosed for the tertiary amine. On page 4 of (1), lines 5 to 7, it is said: "Preferably the solid amine composition contains at least 6.5 and especially at least 7% of the amine." This, in principle, does not exclude lower amounts of the tertiary amine. However, according to the existing circumstances of the case, especially the lack of any tangible figure lower than 6.5% in this context, the Board finds that (1) gives no incentive for the skilled man seriously to contemplate a reduction of the tertiary amine contents below 6.5% by weight.

However, even if a skilled person had found out that the high content of tertiary amines caused stickiness, he would not have been led by the cited art to the solution of this problem as suggested by the patent in suit.

The laundry detergent compositions disclosed in document (2) are, quite apart from the presence of a detergent builder, three-component systems comprising water soluble quaternary ammonium compounds and anionic surfactants together with nonionic surfactants (see (2), page 3, lines 5 to 21 and page 7, lines 20 to 35). Several groups of quaternary ammonium compounds are given, including those as defined in Claim 1 of the patent in suit side-by-side with groups of compounds outside this range. These compositions are said to exhibit improved greasy soil removal capabilities - the technical problem with which document (2) is concerned.

It is stated on page 12, lines 22 to 24 of this citation (without any experimental support) that "... detergent compositions ... soften the fabrics laundered therewith, ...". This has to be read in the context of the whole section starting on page 11, last paragraph. A particular type of cationic compounds is disclosed there, not covered by Claim 1 of the disputed patent and comprising at least two oxa-groups and, optionally, other functional groups. The document goes on to state: "These surfactants, when used in the compositions of the present invention, yield excellent ... soil removal. In addition, the detergent compositions control static and soften the fabrics laundered therewith, and ... Further, these novel cationic surfactants are environmentally desirable ..." (page 12, lines 20 to 27). Thus, the Board considers that even if a skilled person would pay attention to the softening effect mentioned, he would not connect it either to the quaternary ammonium compounds used in the compositions of

the disputed patent, or to water soluble quaternary ammonium compounds in general, but rather to the particular group of quaternary ammonium compounds disclosed in the said section of document (2).

Consequently, the Board holds that the skilled person would deduce nothing at all from (2) with respect to the softening properties of the present compositions, let alone that the quaternary ammonium compounds of the patent in suit could be useful for solving the technical problem as defined.

- 5.3 Document (3) discloses fabric conditioning compositions comprising cationic surfactants together with a smectite clay and a perfume, such compositions to be incorporated into conventional, preferably aqueous, rinse-added fabric softening compositions (cf. (3), page 3, first paragraph, page 4, second paragraph, page 5, second paragraph together with page 12, first paragraph and example 8 on pages 24, 25).

There is no hint at all in (3) that these fabric conditioning compositions could be useful in STW-compositions, let alone that they would be useful to solve the above problem.

The Respondent argued that the amounts of clay, cationic detergent and nonionic detergent of example 8 of (2) would be in the same order of magnitude as in the patent in suit as soon as all the water was removed. However, no reason can be seen, nor was any given by the Respondent why the skilled person should follow such a recipe for solving the problem underlying the disputed patent.

- 5.4 When the Opposition Division stated in the appealed decision that "... the quaternary ammonium surfactant of document (2) are known softeners ..." they did not cite any reference in support, but obviously relied on relevant

common general knowledge. The term "softener" is a very broad one, and is not limited to agents which confer softness to fabric in a laundry process. Softeners are, e.g. also required in spinning industry and an agent useful in this area is not necessarily a proper softener for laundered fabric. In this connection, the disclosure of document (3) gains particular importance: Numerous groups of surfactants are disclosed in (3) with the only requirement that they have at least one alkyl group with about 10 to 24 carbon-atoms (see pages 12 to 16, especially page 12, last paragraph). A preferred group of these cationic surfactants has the formula $R_1R_2R_3R_4N^+A^-$ with R_1 through R_4 being alkyl whereby one or two of these alkyl groups have 12 to 30 carbon atoms (see page 13, last paragraph).

The second paragraph on page 14 of (3) states (in the English translation):

"Quaternary ammonium compounds, which are conventional softeners are preferred components of the compositions according to the invention. These softeners have the above formula, where R_1 and R_2 are each alkyl groups ... with 12 to 22 carbon atoms ... Such softeners can be used in the compositions of the invention together with the preferred cationic surfactants with only one long chain."

This disclosure is in contradiction with the Opposition Division's finding relating to common general knowledge. It suggests strongly that the quaternary ammonium compounds of the present Claim 1 with only one C_8 to C_{14} alkyl group are not commonly known softeners for fabric laundry processes, in contrast to those quaternary ammonium compounds with two longer chains. Thus, the Board holds that, while the Opposition Division's finding might

be correct in a broader sense, it is not true in respect to the present issue.

- 5.5 None of the citations (1), (2) or (3) comprise any information on the properties of a system containing anionic detergent, water soluble cationic detergent and smectite clay. The only information in this respect can be found in (4) which is concerned with granular laundering compositions providing simultaneously laundering, softening and anti-static benefits on textiles. On page 3, lines 32 to 36 it is stated:

"... water-soluble quaternary salts become chemically affixed to the surface of the clay, or react with the soap or curd dispersant. When the quaternary anti-static agent is affixed to the surface of the clay, or has reacted with the soap or curd dispersant, it does not perform the desired anti-static function"

and on the same page, lines 45 to 47 it is stated:

"Quaternary ammonium-compounds are not generally considered to be useful in combination with anionic materials such as soaps since the opposite charges on these two types of materials cause them to react and precipitate from solution."

While these statements in one patent document do not amount to a prejudice against the combined use of such a system comprising the three components, they clearly provide no incentive for a skilled person to try such a system for solving the technical problem as defined above, since obviously, competing reactions will take place in such a system with unpredictable implications.

- 5.6 It follows from the above that the cited references, neither on their own nor in combination with each other, are such as to render obvious the subject-matter of Claim 1.
- 5.7 Documents (5), (6) and the Appellant's letters in connection with European patent applications 79 200 656.1 (letter dated 29 September 1981) and 80 200 878.9 (letter dated 13 April 1988) were submitted only on 17 August 1988, i.e. after expiry of the opposition period.

According to the Respondent, the late filing of (5) was due to the fact that the clay component did not previously constitute an essential component of the compositions of the invention. However, this is true only for Claims 1 to 3 as granted, and cannot be accepted as an excuse since the compositions of dependent Claim 4 as granted already comprise, as an essential feature, "4%-15% of a sodium or calcium montmorillonite".

The Board has already decided that Article 99(1) and Rule 55(c) EPC considered together require that an Opponent's case against an opposed patent should be presented fully and completely in the Notice of Opposition, and should not be developed in piecemeal fashion (T 117/86, Costs/FILMTEC, Reasons for the Decision No. 4, OJ EPO 1989, 401, 405, confirmed by T 416/87, Block copolymer/ISR, Reasons for the Decision No. 10, OJ EPO 1990, 415, 424). This means that already in the Notice of Opposition, full arguments as well as supporting evidence must be submitted, showing why features which are only mentioned in dependent claims cannot render patentable the respective subject-matter of those claims.

For the above reasons, the Board finds that the late submission of (5) was not justified.

Furthermore, and regardless of the above considerations, and having studied all these late-filed documents and letters, the Board decides to disregard them as being irrelevant for the following reasons: Firstly, document (5) and the two letters relate to compositions different from those claimed. Furthermore, document (5) deals with adsorption phenomena in systems comprising clay and cationic surfactants but no anionic surfactants.

Secondly, document (6) refers to perception scales, which have no relevance to the technical problem underlying the patent in suit.

5.8 Claims 2 to 4 cover particular embodiments of the subject-matter of Claim 1 and derive their patentability from that of Claim 1.

6. In respect to the lack of sufficient disclosure asserted in the Notice of Opposition, the Opposition Division held that this objection was unfounded since the description of the disputed patent contained information enabling the skilled man to prepare the claimed composition. This finding was not challenged in the appeal proceedings and the Board sees no reason not to concur with it.

Order

For these reasons, it is decided that:

1. The appeal is allowed.
2. The decision of the Opposition Division is set aside.

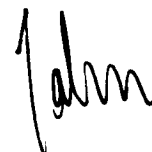
3. The case is remitted to the Opposition Division with the order to maintain the patent on the basis of the claims submitted in the course of Oral Proceedings.

The Registrar:



P. Martorana

The Chairman:



K. Jahn