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D E C I S I O N
of 25 April 1996

Case Number: T 0692/93 - 3.3.2

Application Number: 85308142.0

Publication Number: 0181773

IPC: A61K 7/08

Language of the proceedings: EN

Title of invention:
Shampoo compositions

Patentee:
THE PROCTOR & GAMBLE COMPANY

Opponent:
01: HENKEL KGaA
02: Unilever PLC
03: L'OREAL

Headword:
Shampoo composition/PROCTER & GAMBLE

Relevant legal provisions:
EPC Art. 54, 56, 83, 123

Keyword:
"Novelty: range of values not disclosed in the prior art"
"Inventive step: second use (optimized) of a known agent"

Decisions cited:
-

Catchword:
-



Case Number: T 0692/93 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 25 April 1996

Appellant/Respondent:
(Opponent 01) HENKEL KGaA
TFP / Patentabteilung
D-40191 Düsseldorf (DE)

Representative: -

Appellant/Respondent:
(Opponent 02) Unilever PLC
Colworth House
Sharnbrook
Bedford MK441LQ (GB)

Representative: Hagemann, Heinrich, Dr.rer.nat., Dipl.-Chem
Patentanwälte
Hagemann & Kehl
Postfach 86 03 29
81630 München (DE)

Appellant/Respondent:
(Opponent 03) L'OREAL
14, rue Royale
75008 PARIS (FR)

Representative: Dossmann, Gérard
Bureau D.A. Casalonga-Josse
Morassistrasse 8
D-80469 München (DE)

Appellant/Respondent:
(Proprietor of the patent) THE PROCTER & GAMBLE COMPANY
One Procter & Gamble Plaza
Cincinnati
Ohio 45202 (US)

Representative: Lawrence, Peter Robin Broughton
GILL JENNINGS & EVERY
Broadgate House
7 Eldon Street
London EC2M 7LH (GB)

Decision under appeal: Interlocutory decision of the Opposition Division of the European Patent Office posted 14 July 1993 concerning maintenance of European patent No. 0 181 773 in amended form.

Composition of the Board:

Chairman: P. A. M. Lançon
Members: C. Germinario
C. Holtz

Summary of Facts and Submissions

- I. European patent No. 0 181 773 was granted in response to European patent application No. 85 308 142.0.
- II. Notice of opposition was filed by the Opponents I, II and III. Revocation of the patent in its entirety was requested on the ground of lack of novelty, lack of inventive step [Articles 100(a), 52, 54 and 56 EPC] and insufficiency of disclosure [Article 100 (b) EPC].

Among the documents cited, the following are relevant for the present decision with regard to the novelty and/or inventive step of the invention:

- 1: EP-A-0 152 194;
- 2: EP-A-0 166 232;
- 3: EP-A-0 180 464;
- 4: EP-A-0 141 593;
- 5a: EP-A-0 077 920;
- 6: US-A-3 964 500;
- 11: UA-A-2 826 551;
- 13: US-A-4 452 732;
- 14: EP-A-0 034 846;
- 17: Dow Corning Spec 4/7 82, "Liquid soap"
(enclosed: a) extracts from CFTA International Cosmetic Ingredient Dictionary and Handbook and b) Down Corning product Bulletin);
- 21: US-A-4 337 166;

The following further documents are also taken into account with regard to specific aspects of the invention:

- 23: US-A-4 364 837;
- 38: Cosmetics & Toiletries, Vol 96, July 1981, "Opacifiers and pearling agents in shampoos", pages 65 to 78;
- 39: Product specification of Empicol 0627 and an analytic report;
- 42: see E3 below;
- 45: Chemical Abstracts, 105(16): 139394m;
- 46: "Silicones for personal care" General Electric Silicones;
- 47: Cosmetics and Toiletries, Vol. 91, January 1976, "Volatile silicone fluids for cosmetic formulations", page 2932;
- 48: "Information about volatile silicone fluids", Dow Corning 1982;
- 49: "Volatile Silicones", General Electric Silicones;
- 50: "Silicon Compounds (Silicones), B.B. Hardman, A. Torkelson, General Electric Comp.;
- 54: Kosmetik, Zeitschrift für die Körperpflegemittel, Parfümerie-, Reichstoff- and Aerosol-Industrie, 43. Jg. N. 19, 16 September 1970 (& Seifen, Öle, Fette, Wachse, 96. Jg, 19/1970), pages 657 to 661;
- 56: Jp-A-5751799, (enclosed english translation);
- 57: Henkel Brochure "Cutina AGS" April 1980;
- 59: CIR, "Final Report of the Safety Assessment for glycol stearate, glycol distearate", The Expert Panel of the Cosmetic Ingredient Review, November 1980;
- 71: J. Am. Chem. Soc., Vol. 68, April 1946, D.F. Wilcock "Vapor Pressure-Viscosity Relations in Methyl polysiloxanes", pages 691 to 696;

The following evidences in the form of comparative tests or declarations are also considered:

- E2: Annex and Tables 1, 2 and 3 submitted by Appellants Opponents II on 12 November 1993 and resubmitted on 14 July 1994;
- E3: Two photographs and enclosed text submitted by Appellants Opponents III submitted on the 17 June 1993;
- E4: Declaration 1 by Cauwet of 19 March 1996;
- E6: Declaration in the USPTO by Bolich of 31 July 1987;
- E11: Comparative tests in the form of appendix 8, 9 and 10, submitted by the Appellants Proprietor on 19 November 1993;

III. In its interlocutory decision, the Opposition Division held, that the patent could be maintained in amended form, refusing the main, first and second auxiliary requests for lack of novelty over the content in (2) [Article 54(3) EPC] and (21) [Article 54(2) EPC].

With regard to the refused requests, the disputed point, as to (2), was whether the emulsifiable conditioning agent of example 1 was in the same crystallized form as the corresponding long chain acyl derivative of the opposed patent. The answer of the Division was in the affirmative since the agent of (2) is said to be either a liquid or a solid.

As to (21), the question was whether the cyclic siloxanes of example 1 and 3 were equivalent to the non-volatile silicone of the patent. Having considered that the features volatile or non-volatile are to be evaluated at the normal preparing conditions of a

shampoo and that some of the cyclic siloxanes of (21) exhibit a boiling point of 254°C, it was concluded that also the compositions of (21) comprise, at least in part, essentially non-volatile siloxanes.

All the amendments introduced into the text of claim 1 of the first and second auxiliary requests were regarded as unable to meet the objection of lack of novelty, at least against (21), already raised with regard to the main request.

The Opposition Division recognized the novelty of the third auxiliary request based on the specific size of the crystals of the long chain acyl derivative (10 μ or less) and its inventive merit both over the teaching in (21) when combined with (7) or in (11) when combined either with (13) or (14), basically for the following reasons:

None of the two documents (21) or (11), which alternatively were considered as the closest prior art, but which both failed to indicate to feature "crystals" or "crystal size" for the long chain acyl derivative, addressed the present problem of suspending insoluble non-volatile silicones.

Solutions to this problem could be found neither in (13) nor in (14) which described the use of long chain acyl derivative for stabilizing different materials. Nor was the solution of the aforementioned problem suggested by (7) which referred to compositions other than shampoos. Finally none of (13), (14) or (7) mentioned that the long chain acyl derivative must be in crystalline form having the claimed particles size.

IV. Appeals against this decision were lodged by the Proprietors (appellants and respondent) and by the Opponents I, II and III (also appellants and respondents).

Oral proceedings took place on 25 April 1996.

V. In their statement of ground of appeal and further submissions the proprietors reiterated that the subject-matter of claim 1 as granted was indeed novel over the content in (2) and (21) since a long chain acyl derivative, as comprised in the cited prior art compositions, was not necessarily in a crystalline form or not even in a solid form. As long as it was in a crystalline form, as in (21) and (17), the patentees submitted test-results (E11) providing evidence that the crystal size was not that suitable to achieve the intended purpose of stabilizing the silicone, which was a functional feature inherent in the qualification as "suspending agent" of the acyl derivative.

It was further pointed out that, unlike the silicone in the patent at issue, the amino-functionalized siloxanes of (1), (4) and (17) were soluble in the shampoo matrix, and the cyclic siloxanes of (21) were volatile as illustrated in (45), (46), (47), (48), (49), (50), and (71).

As to the inventive step involved in the claimed subject-matter, it was pointed out by the proprietors that document (6), when taken as the closest prior art, suggests the addition of a thickener to solve the problem of phase instability in compositions comprising insoluble silicones.

It was however maintained that one cannot find any technical motivation in (6) to replace the thickener, therein proposed, by a long chain acyl derivative as described in (7), (13) or (14).

Moreover the results reported in the Bolich in E6 provide evidence that long chain acyl derivatives were not able to suspend any type of materials.

While taking (21) as the closest prior art, the proprietors argued that the document addresses a different technical problem than that of suspending dispersed non-volatile silicones.

To corroborate these arguments, the results of a test of stability were submitted (E11) proving that the composition according to example 3 of (21) is indeed unstable upon centrifugation. This was because the long chain acyl derivative in Empicol 0627 is present as large agglomerates rather than as crystals.

Yet, since the presence of a long chain acyl derivative in the composition of (21) was merely optional and intended for a different purpose, the skilled reader would not find any motivation to decreasing the size of the crystals of said acyl derivative in order to improve the silicone stability.

- VI. Opponents I, II and III objected to the novelty of claim 1 of each of the requests on the basis of (1), (2), (3), (4), (5a), (6), (21) and (17), all documents describing shampoo or detergent compositions.

In so far as these documents did not expressly indicate that the long chain acyl derivative comprised in the prior compositions was in crystalline form [see (1) to (6)] the opponents argued that this feature, as the specific size of the crystals, were inherent in any pearling agents prepared according to the usual process of heating and cooling.

They also contended that the aminofunctional silicones of (1), (4) and (17) and cyclic siloxanes of (21) shared the same features of the silicone of the opposed patent, namely that they were insoluble, the former and non-volatile the latter.

In so far as claim 1 of the third auxiliary request could be recognized as novel, due to the specific particle size of the acyl derivative, the opponents objected to its inventive step based on (21) or alternatively (6), as the closest prior art and considering said prior art either alone or combined with documents (54), (38), (13), (14) or with the results of any of the comparative test (E2), (E3), (E4) or (E5).

Opponent I and III both argued that the definition of the crystal size could not endow the claimed composition with an inventive merit over (21) since the indicated size was the usual one for any pearling agent as proved by document (54). Moreover the claimed crystal size was the result of the method of routine for preparing compositions comprising pearling agents.

This opinion was corroborated by the data submitted as (E4) by Opponent III which provided evidence that a newly prepared shampoo composition as according to (21) contained crystals of ethylene glycol stearate of 5 μm in size.

Opponents II indicated document (11), later replaced by document (6), as the closest prior art and argued as follows: The problem of the stability of the silicone-containing compositions, originally disclosed in (11), was identified in (6), which suggested, as a solution, the addition of thickeners.

Since the thickeners bring about the drawback of a too high viscosity and consequent lost of silicone deposition, as known from (23), the skilled reader would solve the above cited stability problem according to the teaching in (13) or (14) which both suggest the use of long chain acyl derivative for purpose of stabilising suspensions.

Opponent II also had investigated whether the selection of the particle size 10 μ m or less is actually essential to achieve the pretended technical effect of the invention.

By way of test (E2) comparing the stability of two compositions comprising long chain acyl derivative in crystalline form having particles size of 14 μ m and 6.5 μ m no difference in stability could be detected. It was therefore concluded that the claimed crystal size was arbitrary and did not involve in itself an inventive merit.

VII. During the oral proceedings the proprietors requested that:

the decision under appeal be set aside;
the appeals lodged by the opponents be dismissed;

the patent be maintained on the basis of the patent as granted (main request), or alternatively on the basis of the first auxiliary request as filed on 25 March 1996 (first auxiliary request) or of one of the second or third auxiliary requests filed during the oral proceedings.

The opponents requested that:

the appeal lodged by the Proprietor be dismissed and the European patent 0 181 773 be revoked.

VIII. The independent claims on file read as follows:

Main request

A shampoo composition comprising:

- (a) from 5% to 70% of a synthetic surfactant;
- (b) from 0.01% to 10% of a dispersed, insoluble, non-volatile silicone;
- (c) from 0.5% to 5% of a suspending agent selected from long chain acyl derivatives and mixtures thereof, said acyl derivative being present in the shampoo composition in the form of crystals; and
- (d) water.

First auxiliary request

A shampoo composition comprising:

- (a) from 5% to 70% of a synthetic surfactant;
- (b) from 0.01% to 10% of a dispersed, insoluble, non-volatile silicone;

- (c) from 0.5% to 5% of a suspending agent selected from long chain acyl derivatives and mixtures thereof, said acyl derivative being present in the shampoo composition in the form of crystals; and
- (d) water,

wherein the acyl derivative has an average particle size in the shampoo composition of about 10 μm or less.

It is not necessary to consider the second and third auxiliary requests in the present decision.

Reasons for the Decision

- 1. The appeal is admissible.
- 2. *Main request*
- 2.1 Admissibility

The text of claim 1 requires that the long chain acyl derivative of point (c) be present in the shampoo composition in form of crystals.

The feature, which was not cited in claim 1 as filed, is disclosed in the original description, under the heading "Method of manufacture", in the sentence " In the cooling step the acyl derivative is preferably crystallized into particles having an average particle size of about 10 microns or less".

The amendment introduced into the wording of claim 1 as granted, namely the crystalline form of the suspending agent, without any further limitation as to the size of the crystals, does not contravene the requirements of

Article 123(2) EPC, since the word "preferably" underlines that the indicated particle size is only a preferred, but not binding feature.

The text of claim 1 under consideration is identical to that of the granted claim. Therefore the main request complies with the requirements of Article 123(3) and Article 84 EPC.

2.2 Novelty

Documents (1), (2) (3), (4), (5a), (6), (17) and (21) are regarded by Opponents I, II and II as prejudicial to the novelty of the subject matter of claim 1.

The Board does not see any other more relevant document cited during the procedure.

2.2.1 Among the aforementioned documents, (21) is regarded as the most pertinent.

The document describes conditioning-shampoos, containing, as a conditioning agent, cyclic siloxane.

The composition according to example 3 comprises:

sodium lauryl sulphate (28% in water)	40 parts
pearlising agent (Empicol 0627)	8 parts
coconut diethanolamide	3 parts
thickening agent	qs
citric acid and water	

To 9 parts of said matrix one part was added of a mixture of cyclic siloxanes consisting of about 70% by weight of decamethylcyclopentasiloxane and about 25% by weight of dodecamethylcyclohexasiloxane, the remaining being cyclotetra- and cyclotri-siloxanes.

As proved during the procedure and accepted by all the parties, the commercial product Empicol 0627, which is used in example 3 as pearling agent, is a mixture comprising ethylene glycol distearate, which is a long chain acyl derivative as covered by the wording of claim 1.

- 2.2.2 The controversial points discussed by the parties are whether the Empicol 0627 is present in the composition of the prior art in the form of crystals and whether the cyclic siloxanes share the same feature of the dispersed, insoluble, non-volatile silicone of claim 1.

Answer to the former question can be found in (38). This document relates to opacifiers and pearling (pearling) agents in shampoos and reports, on page 70, first paragraph of the lefthand-column, that "all pearling agents work on the principle that they are insoluble in the base material... and will crystallise out from this in the form of small platelets that act rather like miniaturised semi-silvered mirrors..". Actually Empicol 0627 is contained in the composition of (21) as a pearling agent; therefore it will be present in the crystalline form.

This interpretation is admitted by the proprietors, who, while discussing the composition of (21) (item 23 of the ground of appeal), recognise that Empicol 0627 contains preformed crystals of ethylene glycol distearate (EGDS) in a surfactant suspension. However, upon examination under the microscope, the EGDS was found to be present as large agglomerates rather than as individual crystals.

The Board however would like to stress that the physical organisation of the crystals into clusters does not change the intrinsic crystalline form of the EGDS. This was confirmed by photos, appendix 9 and 10 (E11).

It remains therefore undisputed that Empicol 0627 contains a long chain acyl derivative in crystalline form. The fact that this component of the prior shampoo is intended for a different purpose (pearlising effect) is irrelevant in assessing the novelty of a product claim as present claim 1.

It should moreover be stressed that no characterising functional feature or limiting effect can be recognized in the word "suspending" as an attribute of the "agent", as contended by the proprietor, since the wording of the claim does not require this agent actually to achieve the effect of suspending the dispersed silicone.

Hence it can be concluded that Empicol 0627 is in the same form as the agent covered by the wording of claim 1.

2.2.3 The second disputed aspect is whether the cyclic siloxanes of (21), which are insoluble and dispersed in the shampoo matrix as indicated in column 2, lines 24 and 25, are also non-volatile in the meaning given in the opposed patent.

The invention of (21) is based on the replacement of linear silicones, said to be non-volatile, by the relatively volatile cyclic siloxanes, which significantly reduce the drying time following the shampooing step.

The cyclic siloxanes are defined by the general formula in column 1 and further identified in example 1 as being a mixture of decamethylcyclopenta-siloxane (D5), dodecamethylcyclohexa-siloxane (D6), cyclotetra-siloxane (D4) and cyclotri-siloxanes (D3).

The opposition division expressed the opinion that the feature "non-volatile" is a relative term unable to make a distinction between the siloxanes of (21) and those of the opposed patent, and additionally that, among the siloxanes of (21) the term D6 (cyclohexa-siloxane) should be considered as non-volatile (or at least as relatively non-volatile) having a boiling temperature of 245°C.

The proprietors on the contrary contend that the skilled practitioner is well familiar with the expression "(non)-volatile" which was employed in the technical field since the first report of a shampoo composition containing silicones (H.C. Geen, document (11)) and that the term should be given an absolute rather than a relative meaning as evident from the documents (23), (45) to (50) and (71).

They also contest the criteria followed by the opposition division to infer the non-volatile nature of the cyclic siloxanes of (21) from their high boiling temperature and maintain that the correct parameters to assess whether a silicone compound is volatile are the evaporation rate, the evaporation heat, the vapour pressure, the surface tension and the viscosity, but not necessarily the boiling point.

- 2.2.4 The Board agrees that the expression "volatile" is cited in many prior art documents. The fact however remains that the authors of these pieces of prior art, while using this expression, felt the need to clarify it by

way of an additional chemical definitions of the concerned siloxanes. In other words, the authors recognized that the expressions "volatile" and "non-volatile", when taken alone, did not identify a clear threshold between the two categories. For instance, documents (23), (46), (47) and (48) indicate as volatile the polyalkyl tetra- or penta- (D4 or D5) cyclic siloxanes. Document (21) includes in a group said to be "relatively volatile" the term D6 (dodecamethylcyclohexa-siloxane), while documents (49) and (50) include the still higher terms D7 and D8.

Therefore the fact that the expression was known in the prior art does not automatically imply the existence of a generally recognized precise technical meaning.

It is also agreed, as maintained by the proprietors, that the boiling temperature alone is no sufficient parameter to decide whether or not the cyclic siloxanes of (21) can be regarded as non-volatile.

For this reason the Board relies instead on those parameters indicated by the proprietors and discussed in documents (71), specifically Table 3, Fig. 2, Fig. 3 and Fig. 4, document (49) and document (50), Table 5, namely vapour pressure, vaporisation heat and viscosity.

The undisputable teaching derivable from said documents is the **relative** nature of said parameters necessary to evaluate the volatility of a silicone compound.

It is indeed immediately evident that the vapour-pressure, the latent heat of vaporisation and the viscosity all depend on the number of "Si" units of the siloxane. Thus by changing this number, it is always possible to obtain compounds exhibiting higher or lower vapour-pressure, latent vaporisation heat and viscosity

and consequently being more or less volatile. This independently of whether the siloxane is cyclic or linear.

In conclusion, the binomial linear/cyclic is independent of and does not reflect the binomial non-volatile/volatile. Therefore the feature "non-volatile" in claim 1 does not permit identifying a group which is clearly distinct from the group "cyclic siloxanes" of (21).

Nor can the interpretation of said expression, as given in the description of the opposed patent (page 5), better support any alleged difference vis-a-vis the compounds of (21).

In fact, the silicone of the invention is said to be "essentially non-volatile". The word "essentially" cannot help in defining the meaning of non-volatile and does not allow a clear limitation against the group of cyclic siloxanes of (21), which are said to be "relatively volatile".

Furthermore, among the different classes of silicone, the patent cites the polyalkylsiloxanes having a viscosity ranging from 5 to 100000 centistokes at 25°C. Yet, according to (50), the dodecamethylcyclohexasiloxane comprised in the composition of (21) exhibits, at the same temperature, a viscosity of 6.62 centistokes, thus within the above indicated range.

Hence, the conclusion of the Board is that the feature "non-volatile", is a relative term, unclear in the context of the patent, which cannot substantiate a difference between the shampoo composition of claim 1 and the composition of example 3 of document (21).

Since no other point of difference may be envisaged in the claimed composition over the content in document (21), the subject matter of claim 1 of the main request lacks novelty in the meaning of Article 52 and 54 EPC.

2.3 Article 83 EPC

Since the main request is not acceptable under Article 54 EPC, it is not necessary to decide on this point.

3. *First auxiliary request*

3.1 *Admissibility*

Claim 1 of the first auxiliary request is limited as regard to claim 1 according to the main request in that the acyl derivative (c) has an average particle size in the shampoo composition of about 10µm or less.

As already discussed in relation to the main request, the particle size of the long chain acyl derivative is disclosed in the application as filed.

The first auxiliary request is thus formally allowable in the term of Article 123(2) and, due to the limitation in scope of the claim, 123(3) EPC.

3.2 Article 83 EPC and 84

3.2.1 The Board does not see any objection pursuant to Article 84, since the amended claims are clear, concise and supported by the description.

3.2.2 During the procedure, the opponents raised different objections as to lack of sufficiency of disclosure of the invention.

The first is based on the silicone feature of being "insoluble". The feature is interpreted in the description as "insoluble in the shampoo matrix".

In this regard the opponents contend that no clear definition of said shampoo matrix, in terms of components thereof, is given in the patent disclosure. Thus no support is provided to enable the skilled practitioner to select the silicones suitable for reducing the invention to practice.

The Board accepts that the insolubility in the "shampoo matrix" is not the same as insolubility in water or in any other liquid, and that this property is actually an essential feature of the invention.

But it is quite obvious to the Board that, for the skilled reader, "shampoo matrix" means the whole of the essential components which form the body of the shampoo, specifically surfactants, thickeners and all those agents which may influence the stability and the solubility of the different components. This is evident, for instance, from document (21): in example 3 a "shampoo matrix" is prepared containing all the surfactant-, thickening- and further agents of the shampoo; to this matrix, the mixture of silicone derivatives is thereafter added to obtain a dispersion.

The expression "shampoo matrix" is regarded as a synonymous of the expression "base material" used, for instance, in document (38), page 70, first paragraph of the lefthand column.

Therefore the skilled person must be familiar with the meaning of the expression "shampoo matrix", and is able to select, without undue experimentation, the suitable silicones insoluble in said shampoo matrix.

- 3.2.3 A further objection concerns the size of the particles of suspending agent since the patent allegedly fails to give any instruction as how to obtain said size.

It is undisputed by the parties that the long chain acyl derivatives of the invention are known in the prior art as pearling agents, and perform their activity when present in form of crystals.

Moreover, the crystals may be obtained by well known procedures of heating and cooling as disclosed in document (38), page 74 lefthand column and page 76. The same document, stressing the importance of the correct particle size (page 70), also offers instructions as how to influence said size, by experimentally changing the condition during the cooling step (page 74).

Having regard to this general knowledge it is concluded that the skilled person would be able to easily find, without undue experimentation, the suitable conditions to obtain crystals in the desired size.

- 3.2.4 Finally, the opponents contend that the pretended stability of the claimed shampoo composition depends on factors or features, which are not even cited in the patent disclosure, given that the shampoo composition of (21) proved to be instable (E11) and that said composition allegedly falls within the scope of claim 1. Therefore their conclusion was that the claim embraces embodiments which are not operable, or which do not bring about the pretended technical effect.

However, as it will be explained in more details later on in the present decision, when examining the novelty of claim 1, the Board does not recognize the validity of the assumption that the shampoo composition of example 3 of document (21) falls within the scope of claim 1 as amended. Therefore, these opponents' arguments are immaterial.

3.3 Novelty

3.3.1 The Invention

The shampoo composition according to claim 1 of the first auxiliary request comprises a long chain acyl derivative in the form of crystals, wherein said acyl derivative has an average particle size in the shampoo of about 10 μm or less.

The word "comprising" leaves the possibility open that the claimed composition also contains, in addition to long chain acyl derivatives in crystalline form and defined size, other similar derivatives exhibiting a different function as that of a suspending agent and that they may be present in a form different from the claimed one.

The Board is however convinced that this interpretation is excluded by the wording of the claim. In fact the last part of the text cites the acyl derivative - which has the specific particle size of 10 μm or less - without any further definition or reference to its function; therefore, should any additional long chain acyl derivative be present in the composition, it would, regardless of its function, necessarily be in the physical form of particles of size 10 μm or less.

3.3.2 From all the documents quoted for the purpose of novelty, **document (21)** was indicated by the opponents as the most relevant. The Board shares this opinion.

As already seen above, the pearlised shampoo composition of example 3 comprises the commercially available product Empicol 0627 in an amount of 8%.

Admittedly Empicol contains preformed ethylene glycol distearate (EGDS) crystals (cf. 2.2.2).

The proprietors contend that the EGDS crystals are not present as individual particles but rather as agglomerates exhibiting a size much higher than the size sought by claim 1. These arguments are corroborated by photos, submitted as appendix 9 and 10 (E11), of the microscope examination of Empicol 0627.

To evaluate the size of said agglomerates, photos of a standard dispersion of beads having controlled size of 10 μm were enclosed.

The agglomeration of the EGDS crystals in large clusters is also evidenced by the photos submitted by the opponents as document (42 & E3).

From this evidence it is undeniable that the commercially available Empicol 0627 used as pearling agent in the composition of (21) is not in the same physical form as covered by claim 1. Hence this prior composition cannot be not prejudicial to the novelty of the composition of claim 1.

The opponents contest the reliability of this evidence on the basis of Cauwet's declaration 1 (E4), which reports the results of a test, in which the author produced a composition similar to the composition of

document (21). The author, however, instead of using the commercially available Empicol 0627, manufactured it directly from its components.

By following the usual process for preparing pearlised compositions, namely heating at 70°C and cooling at room temperature, the author obtained a dispersion, which contained EGDS crystals having an average size of 5 µm, thus well within the range indicated in claim 1.

This apparent contradiction with the results submitted in (E11) was convincingly explained by the proprietors, who referred to document (39), which is a brochure, supplied by the producer, illustrating the properties, the use and the advantages of Empicol 0627.

In it, Empicol 0627 is said to be "a formulated pearling agent concentrate which allows the manufacture of lotion shampoos without the need for expensive heating equipment.." The document further reports the method of manufacture of pearlised formulations containing Empicol and stresses that the temperature should not rise above 35°C.

Accordingly no heating step is contemplated in the preparing-process of the pearlised shampoo composition of (21), which comprises Empicol 0627.

It is therefore clear that the presence of crystals in size 5 µm, in (E4), is due to the heating and following cooling steps arbitrarily introduced in the process for reproducing the composition of (21).

Therefore the opponents deviated from the instructions given by the Empicol producer and from the conditions followed in example 3 of (21). For this reason the results reported in declaration 1 are not comparable

with those reported by the proprietors and cannot support a lack of novelty of claim 1 vis-a-vis the composition of (21).

3.3.3 Opponents III, also rely on **other prior documents** contending that the particle size of 10 μm or less is an inherent feature of any pearling agent as indicated in document (54), page 660 and that this size is inevitably obtained by following the traditional preparing process for pearlised composition; process which is described e.g. in (38), table I, formula 1.

Based on this assumption, the opponents III contend that all documents (1) to (6), describing hair-compositions containing a long chain acyl derivative in solid form, either as pearling agent or as opacifier or as conditioning agent, inherently fulfil the condition covered by claim 1.

The Board cannot follow this line of argument. First of all, it is not evident that the long chain acyl derivatives of prior art compositions are actually in solid form.

Document (3) discloses compositions which unambiguously are solutions and, as such, cannot comprise any solid suspended component.

Documents (5) and (6) disclose long chain acyl derivatives, which, because of their function (surfactant or suds boosters) are expected to be in solubilized form.

Even accepting that the acyl derivatives are indeed in solid form, said solid form does not necessarily identify the crystalline state, since the solid could be in an amorphous state.

Also admitting the equivalence of the terms solid/crystal, the fact remains that the cited documents fail to indicate the size of the crystals.

In fact, even if one would accept that (54) identifies particles in the range of 0.2 to 2.0 μm as the best size range for a pearling agent, both document (56) and evidence (E11) [appendixes 9 and 10] indicate that the particles of a pearling agent may be much larger than 10 μm . Therefore the range 10 μm or less, to which claim 1 is now limited, cannot be regarded as inherently disclosed by any prior document citing long chain acyl derivatives.

Hence none of the cited prior documents is prejudicial to the novelty of claim 1.

In view of the above, and since the Board does not see any other more relevant prior document, it is decided that the subject matter of claim 1 according to the first auxiliary request is novel.

3.4 Inventive step

3.4.1 The closest prior art

The parties considered document (21) or, alternatively, document (6) as the closest prior art document.

Though document (21) on the face of it only addresses the final problem of reducing the drying time of hair following the shampooing step, it relates to an invention within the same ambit as the invention of the opposed patent disclosing conditioning-shampoo

compositions, which contain insoluble dispersed silicones as conditioning agents, which should be stable against phase separation while, at the same time, remaining fluid enough to allow silicone deposition.

For this reason the view of the Board is that document (21) is the closest prior art.

3.4.2 The technical problem

As evidenced by the proprietor [photos (E11)] and accepted by the Board, the difference between the composition of example 3 of (21) and the composition of claim 1 lies in the size of the particles of the suspending agent.

As also proved by the proprietors by way of comparative tests, the shampoo composition of (21), comprising the commercial product Empicol 0627 is unstable against phase separation, upon centrifugation and storage [appendix 9, (E11)].

In the light of the above, the technical problem underlying the present invention resides in providing conditioning-shampoo compositions containing insoluble dispersed silicone, which are stable against phase separation of the silicone component while, at the same time, allowing a good silicone deposition.

3.4.3 As a solution for said technical problem, the addition of a long chain acyl derivative in the form of crystals having a particle size of 10µm or less is proposed. The particles are intended to maintain in suspension the droplets of the dispersed silicone and thus to avoid the need for excessive amounts of thickener which would affect the deposition of the silicone on the hair.

The question remains whether the technical problem is actually solved by what is proposed as a solution.

For this purpose, the proprietors have submitted the results of a stability test comparing the composition of document (21) and a commercially available composition (Pert Plus) said to be the shampoo composition of the invention [Appendix 8, (E11)].

The opponents contest the relevance of these results, since it is not evident that Pert Plus is actually identical to the claimed shampoo, a thickener being comprised in this commercially available compound.

For this reason the Board does not consider said comparative test but relies on other undisputed evidence submitted by Opponent II, ie (E2) and by Opponent III, ie declaration 1, (E4).

In the first test (E2), two shampoo compositions were prepared, the former (A) falling within the scope of claim 1, the particles of the of long chain acyl derivative having size of 6.5 μm , the latter (B) falling outside, the particle size of the suspending agent being larger than 10 μm : namely being 14 μm .

Neither of the two compositions comprises a commonly used thickener such as alkyl-cellulose or xanthan-gum, which could contribute to the stabilisation.

The opponents report that a stability test, by centrifugation at 4000 rpm, did not show any phase separation for either formulation and conclude that the stability is independent of the particle size of the suspending agent.

However, the Board would emphasize that the main conclusion to be drawn from this test is that the stability observed for both formulations is the result of the presence of the long chain acyl derivative in crystalline form and particle size of either 6.5 or 14 μm since no other usual thickener was contemplated.

In the second test, (E4), four compositions similar to the shampoo of example 3 of document (21) were reproduced. As declared by the author and as already seen above, all the compositions comprised ethylene glycol stearate in crystalline form having particle size of 5 μm ; therefore falling in the scope of claim 1.

The author reported that all said compositions are stable upon storage and that no phase separation could be detected.

Since, on the other hand, the shampoo composition of (21), which contains a long chain acyl derivative organized in large crystalline agglomerates (larger than 10 μm), proved to be unstable upon storage (E11), the stability observed in (E4) can only be due to the size of the crystals of the long chain acyl derivative.

On the basis of these considerations the Board is satisfied that the underlying technical problem is really solved by what is proposed as a solution.

With regard to the conclusion drawn by the opponents from (E2), that the size limit of 10 μm is arbitrary and does not contribute to the solution of the technical problem, the Board would stress the following:

the size limitation of the particles of the long chain acyl derivative was introduced in claim 1 in order to meet an objection raised during the examination procedure.

The value "10 μm or less" was cited in the originally filed application as the preferred embodiment of the invention. No other possible limitation of the scope of the independent claim, with respect of Article 123(2) EPC, could be envisaged in the description.

Due to this situation, and to the nature of the invention, it seems quite unrealistic to predict that the technical effect of the invention would vanish immediately outside the claimed range. On the contrary, it is reasonable to expect that said effect progressively decreases and then disappears. For this reason, the difference between 10 and 14 μm may be very slight.

Now to appreciate this difference, a stability test should exhibit a very good sensitivity, which may not necessarily be the case in (E2), where a centrifugation of 80 minutes was employed.

As a matter of fact, the proprietors, in order to prove the lack of stability of the shampoo composition of the closest prior art (see (E11)), submitted the composition to a 24-hours centrifugation at a variety of rotation speeds.

The Board therefore concludes that the conditions followed by the opponents in (E2) are not suitable in evaluating the true difference in stability between a composition of the invention and a similar composition containing particle of long chain acyl derivative having size of 14 μm .

3.4.4 The question arises whether the skilled reader of (21) would find any suggestion indicating the proposed solution either in the closest prior art in itself or in any other document that can be combined with the closest prior art.

Though document (21) does not address specifically the problem of the stability of the silicone suspension, this result is partially achieved by emulsifying the siloxane prior to incorporation into the composition and adjusting the viscosity with a thickening agent, as seen in example 3.

More importantly, the document does not disclose nor envisages any problem of phase separation of the silicone component. Therefore the skilled reader of (21) would find no technical motivation in the document itself to consider any modification to improve the stability of the suspension.

Should nevertheless the skilled practitioner contemplate improving the stability, he would probably consider, first of all, those agents which are traditionally known as stabilizers of emulsions or suspensions: namely the surfactants or the thickeners.

Given these circumstances, it seems unlikely to the Board that the skilled person would consider the crystal size of an optional component, such as a pearling agent, as a possible candidate for modification

3.4.5 The opponents have expressed the opinion that the problem of phase separation of silicone suspensions is inherent in any compositions comprising said substance and that the skilled person needs no explicit suggestion to be aware of this.

They also maintain that the long chain acyl derivatives employed in cosmetic compositions as pearling agents are also known to act as stabilizers or emulsifiers. Documents (38), (57) and (59) are cited to prove that the relationship between pearling and stabilizing or emulsifying activity is well known in the art.

- 3.4.6 The Board agrees that the instability of silicone suspensions may be a generally recognized problem and that the long chain acyl derivatives may also perform a stabilizing activity.

Nevertheless, it should be noted that the suspending activity according to claim 1 is restricted to long chain acyl derivatives of specific particle size and that the crystal size suitable to achieve the suspending effect is not necessarily the optimum, or even the correct size to achieve a pearling effect.

As to documents (57) and (59), it is pointed out that they fail to identify any specific range of particle size even for the pearling effect.

Still less pertinence can be attributed to document (38). The opponents have indicated from the item "Conclusion" the sentence " We have seen that shampoos are opacified and pearled either for an aesthetic effect or for stabilizing purpose".

The Board however notes that this conclusion is quite contradictory to the real content of the article. In fact the author points out that the crystalline suspensions of pearling agents are themselves unstable and tend to sediment with the consequent loss of the desired sheen. This is why pearly shampoos are

manufactured to have a very high viscosity. Reference is made to page 70, first paragraph of the lefthand column and to page 74, second paragraph of the righthand column of (38).

Therefore it appears unlikely that the skilled reader would derive from document (38) any suggestion to use a crystalline suspension of a pearling agent, suspension which is itself known to be instable, to further stabilize a silicone dispersion.

Additionally, it should be noted that the documents (56) and (E11) indicates values for the particle size of a pearling agent which are normally higher than the limit established by claim 1. Namely (56) indicates the range of 5 to 30 μm , while (E11) stresses that Empicol 0627, thus a very common pearling agent, comprises ethylene glycol stearate agglomerated in particles much larger than 10 μm .

In conclusion, the above discussed documents neither prove a recognized relationship between suspending and pearling activity nor provide the skilled reader with the necessary support to identify a range of optimum particle size which is valid both for the pearling effect and for the suspending effect. Instead, they appear to point to an optimum size for the pearling activity which is much larger than the claimed higher limit of 10 μm .

3.4.7 Finally, Opponents II, developed a further line of argument based on document (6) as the closest prior art.

Document (6) describes shampoo compositions comprising dispersed insoluble silicone. The addition of thickeners is proposed to stabilize the suspension (column 3, line 66 to column 4, line 16).

The drawback involved in the use of high amounts of thickeners is the loss of pourability and, consequently, of silicone deposition on the hair. This disadvantage is identified in document (23), column 3, line 60 to column 4, line 15.

The use of long chain acyl derivatives, specifically ethylene glycol fatty esters, to stabilize unsaturated quaternary ammonium compounds or particulate antidandruff agents is disclosed in document (13) and (14) respectively.

The opponents maintain that the skilled practitioner in order to remedy the disadvantage inherent in the compositions of (6) would find a suggestion in (13) or (14) to replace the thickening agent of (6) by a long chain acyl derivative in order to stabilize the silicone component.

The Board does not share this opinion, firstly because the two documents (13) and (14) are silent on the essential feature of the invention at issue, i.e. the crystal size of the acyl derivative, secondly because R.E. Bolich proved, by way of the comparative test reported in the declaration (E6), that not all materials, even though they are similar, are capable of being suspended by long chain acyl derivatives.

Therefore the skilled reader could find in (13) or (14) no conclusive teaching that an agent able to stabilize a suspension of a solid particulate antidandruff agent or a quaternary ammonium compound could equally stabilize the suspension of a totally different substance such as the liquid silicone of the present invention.

The conclusion of the Board is thus that neither the closest prior art (21), taken alone, nor document (6), nor any combination of these documents and/or other prior art, suggests the solution proposed by the invention as claimed in the first auxiliary request. This request is therefore allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside
2. The case is remitted to the first instance with the order to maintain the patent on the basis of the Patentee's first auxiliary request, submitted on 25 March 1996, and a description to be adapted thereto.

The Registrar:

The Chairman:

P. Martorana

P. A. M. Lançon