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**Datasheet for the decision
of 16 March 2016**

Case Number: T 0147/12 - 3.3.03

Application Number: 07754369.2

Publication Number: 2001932

IPC: C08G77/14, C08G77/46

Language of the proceedings: EN

Title of invention:

PROCESS FOR PREPARING SILICONE POLYETHERS

Patent Proprietor:

Dow Corning Corporation
Dow Corning Toray Co., Ltd.

Opponent:

Evonik Degussa GmbH

Relevant legal provisions:

EPC Art. 83, 54, 56

Keyword:

Sufficiency of disclosure - (yes)
Novelty - (yes)
Inventive step - (yes)
Late-filed document - admitted (no)

Decisions cited:

T 0815/07, T 0990/96, T 0083/01, T 0242/88



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Case Number: T 0147/12 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 16 March 2016

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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted on 13 December
2011 rejecting the opposition filed against
European patent No. 2001932 pursuant to Article
101(2) EPC.**

Composition of the Board:

Chairman	F. Rousseau
Members:	D. Marquis
	R. Cramer

Summary of Facts and Submissions

I. The appeal by the opponent (hereafter "appellant") lies from the decision of the opposition division posted on 13 December 2011 rejecting the opposition against European patent N° 2 001 932 (based on application number 07 754 369.2).

II. The patent as granted contained 12 claims of which independent claim 1 read as follows:

"1. A process for preparing a silicone polyether comprising reacting;
(A) a polyether having at least one terminally unsaturated aliphatic hydrocarbon group and an alkali metal content of less than 50 ppm,
(B) an organohydrogensiloxane having an acid number of less than 0.005, via a hydrosilylation reaction."

Claims 2 to 12 were directed to preferred embodiments of claim 1.

III. A notice of opposition against the patent was filed in which the revocation of the patent was requested on the grounds according to Article 100(a) EPC (lack of novelty and lack of inventive step) and Article 100(b) EPC.

The following documents were cited in opposition:

D1: Reproductions (Trials 1-6)

D5: EP 0 839 852 A2

D6: WO 02/44248 A2

D7: Yokoyama Y. et al., J.Am.Soc. Mass Spektrom. 2007, Nov.; 18(11) :1914-20 Epub 2007 Aug.16

D8: Studies on the Flame Spectrochemical Analysis.

VIII: On the Self Interference and Mutual Interference of Alkali Elements; Shigero Ikeda, Science reports of

the Research Institutes, Tohoku University. Ser. A, Physics, chemistry and metallurgy 9, 1-8, 1957 Tohoku University.

The opposition division found that despite the difficulty to determine the alkali content highlighted in D7 and D8 the skilled person was able to determine the alkali metal content and the acid number as claimed using standard methods available in the art. Neither D5 nor D6 disclosed an alkali metal content of the polyether and an acid number of the organohydrogensiloxane falling within the claimed range. The claims of the patent were therefore novel. None of D5 or D6 would give a hint towards the reduction of alkali amounts to solve the problem of providing a further process. The claims were therefore inventive.

- IV. The statement setting out the grounds of the appeal was received on 17 April 2012.
The following documents were cited:
D9: US 2006/0264602
D10a: English translation of JP2006-002053
D11: Experimental evidence filed by the opponent on 14 September 2011.
- V. The reply to the statement of grounds of the appeal was received on 31 October 2012. In the reply, the patent proprietors requested that the documents D9 to D11 not be admitted into the proceedings.
- VI. On 3 December 2015, the parties were summoned to oral proceedings. A communication pursuant to Article 15(1) RPBA was sent by the Board on 24 February 2016.
- VII. Oral proceedings were held on 16 March 2016.

VIII. The appellant's arguments may be summarised as follows:

Sufficiency of disclosure

In order to perform the claimed process, the skilled reader had to know whether he was working within the boundaries of the claim or not. That was not the case in claim 1 of the patent in suit because a reproducible method for the determination of the alkali metal content of the unsaturated polyether was not provided. D11 showed for example that the result of the measurement of the alkali metal content in a polyether depended on the method used. That was also confirmed in D7 and D8. Not knowing whether he was working within the ambit of claim 1, the skilled person could not achieve the effect disclosed in the patent in suit. Applying the conclusions drawn up in the Board of appeal decisions T 83/01 and T 815/07, the patent was not sufficiently disclosed.

Novelty

The decision of the boards of appeal T 990/96 showed that low molecular weight compounds could not be made novel by their purity grade. The presence of alkali metal impurities in the polyether did not constitute a new element that could characterise the process of claim 1.

D5 disclosed a hydrosilylation process involving the reaction of an unsaturated polyether with an organohydrogensiloxane and it also disclosed that the organohydrogensiloxane could be neutralised with sodium bicarbonate. D9 showed that such a neutralisation resulted in an organohydrogensiloxane having an acid

number within the range claimed in the patent in suit. D5 also disclosed the use of these silicone polyethers in personal care applications. That implied an absolute purity of the starting materials and anticipated the starting materials of claim 1 of the patent in suit in all grades of purity. Also, the patent in suit did not exclude the presence of any amount of alkali metal and acid in the process as long as these were not added together with the starting materials (A) and (B). Claim 1 lacked therefore novelty over D5.

D6 disclosed a hydrosilylation process involving an unsaturated polyether and an organohydrogensiloxane. D6 disclosed the purification of the raw materials before reaction which meant that the polyether and the organohydrogensiloxane were according to claim 1 of the patent in suit. D6 was therefore novelty destroying for the claimed subject matter when taking into consideration the decision of the boards of appeal T 242/88.

D10a disclosed a hydrosilylation process involving an unsaturated polyether with an alkali metal content below 2 ppm and an organohydrogensiloxane. The fact that the purity of the organohydrogensiloxane was not mentioned in that document only meant that the purest possible compound had to be used for the reaction. Claim 1 lacked therefore novelty over D10a.

D10a had been filed late but was admissible because it was highly relevant to the questions of novelty and inventive step and because it had been filed at the earliest possible stage of the proceedings and as a response to the decision of the opposition division. D10a was admissible into the proceedings.

Inventive step

D5 represented the closest prior art. D1 and in particular the comparison between the examples 1 and 4 showed that the technical problem posed had not been solved by the claimed subject matter. Also, the formulation of claim 1 allowed for the presence of alkali metal and acid in any amount as long as these were not part of the starting materials. The objective technical problem was therefore the provision of an alternative process. It was clear from D5, D6 and D10a that the person skilled in the art had to use the purest possible starting materials to perform a hydrosilylation resulting in a silicone polyether having good quality. In particular, D5 already taught the neutralisation of the organohydrogensiloxane and D10a taught the use of an unsaturated polyether with an alkali metal content well within the claimed range. The claims of the patent in suit lacked therefore an inventive step.

IX. The respondents' arguments may be summarised as follows:

Sufficiency of disclosure

The contested patent disclosed in paragraph 12 that the alkali metals present in the unsaturated polyether were residues from the catalyst used during polymerisation of alkylene oxides. It was also shown how these catalysts could be avoided in or removed from the unsaturated polyether. The contested patent contained a clear teaching to a skilled person in the art how the unsaturated polyether according to claim 1 could be obtained. The appellant itself had no problems to provide a polyether having the required alkali metal

content as shown by the experimental data contained in D1. The claimed subject matter was therefore sufficiently disclosed.

Novelty

D5 was silent on the alkali metal content of the allyl polyether used for the hydrosilylation reaction. The fact that the silicone polyether resulting from the hydrosilylation could be used in personal care applications did not mean that the starting materials of the hydrosilylation had to be absolutely pure and that these would be according to claim 1 of the patent in suit. D5 was also silent about the acid number of the organohydrogensiloxane. D9 did not demonstrate that the starting materials of D5 had an acid number according to claim of 1 of the patent in suit. Being late filed, post published and lacking relevance it should not be admitted into the proceedings. Similar arguments applied to D6. Claim 1 was therefore novel over D5 and D6.

D10 and D10a were late filed and as only a machine translation of D10 was made available no comments could be provided on that document. D10 and D10a should not be admitted into the proceedings.

Inventive step

D5 represented the closest prior art. The examples of the patent in suit showed that the problem of reducing the amount in hydrolyzable -SiOC- bonds in the product had been solved by the subject matter of claim 1. That could also be seen from the examples provided by the appellant in D1. None of the documents cited in appeal disclosed a process in which both the amount of alkali

metal in the polyether and the acid number of the siloxane were disclosed. Also, none of these documents disclosed the technical effect provided in the patent in suit and therefore, they could not provide any incentive to modify D5 in order to arrive at the claimed subject matter. The claims of the patent in suit were therefore inventive over the prior art cited.

- X. The appellant requested that the decision under appeal be set aside and that the patent be revoked.
- XI. The respondents requested that the appeal be dismissed.

Reasons for the Decision

- 1. Sufficiency of disclosure
 - 1.1 In order to meet the requirements of Article 83 EPC, an invention must be disclosed so as to allow the skilled person to put it into practice, which means in the present case of claim 1, to perform a process for preparing a silicone polyether comprising reacting; (A) a polyether having at least one terminally unsaturated aliphatic hydrocarbon group and an alkali metal content of less than 50 ppm, (B) an organohydrogensiloxane having an acid number of less than 0.005, via a hydrosilylation reaction.
 - 1.2 The alkali metal content characterizing the subject matter of claim 1 is defined in paragraph 12 of the patent in suit as the amount of alkali metals that often remains in the polyether product (A) after its preparation when the latter is prepared in the presence of alkali metal containing catalysts. Paragraph 11 sets out that polyether (A) must have an alkali metal content of less than 50 ppm, alternatively, less than

10 ppm, or alternatively, less than 5 ppm and paragraph 12 indicates that that content may be determined by any analytical method or technique known in the art. A particular method of determination of the alkali metal content is not provided in the description of the patent in suit.

1.3 The objection of the appellant was not that there existed no method of determination of the alkali metal content in polyethers. Rather, the gist of the appellant's argumentation was that D7, D8 and D11 showed that the value obtained for the alkali metal content in the polyether depended on the analytical method used for its determination. Since the patent in suit did not mention which method had been used to determine the alkali content, the skilled person could not perform a reproducible determination of that alkali content and as a result could not know whether he was working within the ambit of claims, resulting in an insufficient disclosure of the claimed subject matter.

1.3.1 D7 is a scientific publication revealing the alkali metal cation affinities for polyoxyethylene dodecylethers by electrospray mass spectrometry. It shows in essence that alkali metal cations interact with helical polyoxyethylene in the gas phase in a way that resembles the host-guest interactions of cyclic polyethers. To that effect, compositions containing a 5 times molar excess and a 50 times molar excess of different alkali metal cations in the polyether were analysed by electrospray mass spectrometry (page 6, last paragraph). It is firstly not apparent how the results reported in D7 could be relevant to the subject matter of the patent in suit since D7 does not deal with analytical methods for the quantitative determination of alkali metal contents in polyethers.

Also, it cannot be derived from D7 if these interactions, observed in the presence of a large excess of alkali metal, would also significantly affect the quantitative determination of alkali metals present in amounts of less than 50 ppm in polyethers. D7 is not relevant to the claimed subject matter.

1.3.2 D8 is a study from 1956 about the determination of alkali metal contents by flame spectrochemical analysis. It shows that the quantitative determination of alkali metal contents performed on mixtures of these metals may be flawed as a result of interferences caused by interactions between the alkali metals present in the mixture. D8 provides a classification of the spectral interferences observed for each alkali metal (Tables 3 to 8) in solution at a concentration of 100 ppm. D8 is therefore not concerned with compositions of polyethers and the content of alkali metal considered in D8 is outside the claimed range (less than 50 ppm). It has not been shown by the appellant how D8 could be relevant to the question of the reproducibility of the quantitative determination of the alkali metal content in polyethers.

1.3.3 D11 is an experimental report provided by the appellant. It describes the preparation of several samples of a polyether (PE1) containing potassium in known amounts of from 0 to 45ppm. The samples were then sent to two independent laboratories for the determination of their potassium contents by atomic absorption spectroscopy using different spectrometers operated under different measuring conditions. Table 1 of D11 shows for each of the tested samples the content of potassium that was determined by each laboratory. The table shows that the contents of potassium that were measured for the same sample differ in some

instances significantly from one laboratory to the other. These contents also differed from the known amount of potassium that was added to the polyether sample. It can therefore be deduced from Table 1 that even when the same method of determination was used (atomic absorption spectroscopy), the measured potassium content that could be determined in polyether compositions depended on the measurement conditions that were applied during analysis of the sample and that that resulted in an uncertainty about the alkali metal content.

It was requested that D11 not be admitted into the proceedings before the Board. That experimental report had already been submitted in a letter dated 14 September 2011 (page 3, fourth paragraph) before the opposition division in support of the argument first made in the notice of opposition filed on 8 Mai 2010 (page 3, first paragraph) that the value of the alkali metal contents in polyethers would depend on their method of determination. It is also apparent from that letter that the experimental report had been prepared in response to the reply to the notice of opposition filed on 28 October 2010 (page 2, second paragraph) in which it the argument submitted by the appellant in the notice of opposition had been refuted. The experimental report now referred to as D11 in appeal proceedings was therefore submitted as a reply to the respondent during first instance proceedings and has been also filed together with the statement of grounds of the appeal. Since D11 was already part of the first instance proceedings and its filing in appeal is compliant with Article 12(2) and (4) RPBA, the Board sees no reason to exclude D11 from the appeal proceedings.

1.4 However, even if D11 shows that the conditions of measurement may lead to variations of the value of the alkali metal content as argued by the appellant, this alone does not constitute a lack of sufficiency of disclosure regarding the claimed subject-matter as a whole since it has not been shown that the uncertainty concerning the alkali metal content affected the claimed process to such an extent that the skilled person wishing to perform the process would face an undue burden. The appellant has shown that the uncertainty concerning the method of determination of the alkali metal content meant that the skilled person could not ascertain whether the value he would obtain is within or outside the claimed range. It has however not been shown that as a result of that uncertainty, the skilled person would fundamentally be prevented from obtaining a polyether (A) according to claim 1 of the patent in suit, i.e. a polyether having at least one terminally unsaturated aliphatic hydrocarbon group and an alkali metal content of less than 50 ppm. Also, even if the alkali metal content in the claimed polyether (A) can only be determined with uncertainty, it has not been shown that the skilled person was not in the position, on the basis of the information disclosed in the patent in suit and with help of his common knowledge, to perform a hydrosilylation leading to a silicone polyether as claimed. The objection of insufficient disclosure raised by the appellant is in fact related to a lack of clarity, which objection cannot be raised against the claims as granted.

1.5 The present case is different from the case decided in T 83/01 in which a parameter (D3,3) referring to the particle size of a fat blend was neither defined in the claims nor in the description and for which no method of measurement was known to the skilled person (point 3

of that decision). In that case, the Board found that since the skilled person was not in the position to perform any measurement of the claimed parameter, the claimed subject matter was insufficiently disclosed. The argument made in the present case was not that there existed no method for the determination of the alkali metal content but rather that that content could not be determined with accuracy. The conclusion reached in T 83/01 can therefore not be applied to the present case since the argument made by the appellant in the present case is a completely different one.

1.6 The present case is also different from the case decided in T 815/07. In T 815/07, claim 1 pertained to an absorbent article comprising an absorbent core and a crotch region defined by its absorbent capacity relative to the absorbent core's total absorbent capacity, the absorbent capacity being determined by test method A. The Board found in that case that the structure of the article claimed would have an effect on the actual amount of liquid absorbed. Because the patent did not provide adequate information about the tested articles as regards their structure, materials or regions, the test method defined in claim 1 resulted in totally arbitrary values for the crotch region's absorbent capacity. The claimed subject matter was therefore not sufficiently disclosed. In the present case however, the alkali metal content only depends on its method of determination, it does not depend on any variable or feature characterizing the process claimed. The conclusion of case T 815/07 cannot be applied to the present case.

1.7 The appellant has not shown that the uncertainty concerning the determination of the alkali metal content was such that a skilled person could not

perform a hydrosilylation reaction by reacting components (A) and (B) as defined in the claims. As a result, the claims of the patent in suit satisfy the requirements of Article 83 EPC.

2. Novelty

2.1 The patent in suit relates to a process for preparing a silicone polyether comprising reacting: (A) a polyether having at least one terminally unsaturated aliphatic hydrocarbon group and an alkali metal content of less than 50 ppm and (B) an organohydrogensiloxane having an acid number of less than 0.005, via a hydrosilylation reaction (claim 1).

2.2 According to the description (paragraphs 11 and 12), the unsaturated polyethers (A) can be typically prepared by the base catalyzed polymerization of alkylene oxides in the presence of an alkali metal hydroxide as a base catalyst. The alkali metal is said to often remain in the resulting polyether product but it can also be removed from the product using known techniques. Alternatively, the polyether product can be obtained by processes known in the art using non-alkali metal based catalysts. It can be derived from the passage of paragraphs 11 and 12 that not all polyethers having at least one terminally unsaturated aliphatic hydrocarbon group will have an alkali metal content of less than 50 ppm. The examples and comparative examples of the patent in suit also show that commercially available products of different grades having residual sodium contents of 1.2 ppm (Sanyo TG501), 5.0 ppm (Dow SF501 MgSil), i.e. within the claimed range less than 50 ppm as well as 200 ppm (Dow Chemical AE501), i.e. outside the claimed range, were readily available to the skilled person. The alkali metal content is

therefore a feature characterizing the unsaturated polyethers (A) of claim 1.

2.3 Also, the organohydrogensiloxane (B) according to claim 1 can be prepared by any method known in the art such as by an acid catalyzed equilibration of a SiH containing siloxane with other siloxanes and neutralization upon completion of the equilibration reaction (paragraph 16). The organohydrogensiloxanes are characterized by their acid number defined as the mass (in mg) of KOH needed to neutralize the acidic species per gram of the organohydrogensiloxane, as determined by titration techniques well known in the art (paragraph 17). The organohydrogensiloxanes can be prepared in such a manner that the acid number is less than 0.005, or they can be alternatively post treated by contacting the organohydrogensiloxane with a base after production so as to ensure the acid number is reduced to less than 0.005 (paragraph 18). The organohydrogensiloxanes used in the processes of the examples and comparative examples of the patent in suit had the average structure $M-D_5D^{H}_{3.5}-M$ produced by acid catalyzed equilibration of M, D and DH siloxane intermediates and had an acid number of less than 0.001, 0.002, 0.003 or 0.010, showing that not all organohydrogensiloxanes necessarily have an acid number as claimed. The acid number is therefore a characterizing feature of the claimed organohydrogensiloxane (B).

2.4 The present case relates to a process for the manufacture of silicone polyethers characterised by the use of starting materials having a required purity as defined by the alkali metal content of the unsaturated polyether and the acid number of the organohydrogensiloxane. The purity level of the

starting components in claim 1 of the patent in suit is therefore an essential technical feature of the process, which must be carried out in the required range of purity.

2.5 Hence, the question to be answered is whether the subject matter of the present claims, i.e. the combination of features, including the alkali metal content of compound (A) and the acid number of compound (B) are directly and unambiguously disclosed in the documents alleged by the appellant to be novelty destroying.

2.6 D5

2.6.1 D5 discloses a process for forming an organosiloxane copolymer composition, comprising (A) forming a reaction mixture which contains (a) one or more organohydrogensiloxanes comprised of units of the formula $R_aH_bSiO_{(4-a-b)/2}$ wherein each R group represents a substituted or unsubstituted monovalent hydrocarbon radical of from 1 to 12 carbon atoms, a and b are integers, each a is 0 to 3, each b is 0 to 1, and (a+b) is 0 to 3. (b) is an unsaturated polyoxyalkylene or alkene component selected from the group consisting of compounds of the formulas $R_1(OCH_2CH_2)_z(OCH_2CH(R_3))_w(OCH(R_3)CH_2)_y-OR_2$, and $CH_2=CH-R_4$ and mixtures thereof, wherein R_1 denotes a monovalent unsaturated hydrocarbon group containing from 3 to 10 carbon atoms; R_2 is selected from the group consisting of hydrogen, alkyl groups containing one to eight carbon atoms, acyl groups containing 2 to 8 carbon atoms, tri-(C1-C8-alkyl)silyl groups, and cycloaliphatic ether groups of 4 to 6 carbon atoms. R_1 and R_3 independently of each other contain 1 to 20 carbon atoms and each is a monovalent alkyl or branched

alkyl group, aryl group, alkaryl group or cycloalkyl group, or R_4 may be hydrogen; z has a value of 0 to 100, the subscripts w and y each are 0 to 120 and $(w+y)$ has a value of 0 to 120, and $(z+w+y) \geq 3$, and (c) an oil component selected from the group consisting of naturally occurring vegetable oils, hydrogenated derivatives thereof, methylated derivatives thereof, and mixtures of any of the foregoing, said oil component being a liquid having a boiling point of at least 175°C .; and (d) a hydrosilylation catalyst; and (B) reacting the components of said reaction mixture to form a reaction product comprising said copolymer and said oil component (claim 1).

- 2.6.2 The organohydrogensiloxanes that can be employed in D5 are disclosed in the passage bridging pages 4 and 5. These are prepared by the acid equilibration of a mixture which may contain cyclic diorganosiloxanes typically octamethyltetrasiloxane, hexamethyldisiloxane or tetramethyldisiloxane and/or another source of organohydrogensiloxane. Usually, the acid equilibration catalyst is concentrated triflic acid or sulfuric acid or a supported resin containing sulfuric acid functionality. The acid number of these organohydrogensiloxanes is not disclosed in D5.
- 2.6.3 The argument was made that the organohydrogensiloxanes according to D5 would meet the requirement of claim 1 of the patent in suit (acid number of less than 0.005) since D5 disclosed that the silanic fluid obtained from the preparation of organohydrogensiloxanes could be rendered neutral through the use of mild bases like sodium bicarbonate or sodium carbonate. D5 does however not mention the acid number of the neutralized organohydrogensiloxanes and it does not describe the neutralization treatment any further. D9 was cited by

the appellant to show that neutralization would result in an organohydrogensiloxane according to claim 1 of the patent in suit. In support of that argument the appellant mentioned example 2 of D9 wherein neutralization of 225g of an organohydrogensiloxane with 0.27g of sodium bicarbonate reduced its acid number from 0.0037 down to 0.0003, below the threshold defined in claim 1 of the patent in suit.

The admissibility of D9 into the proceedings has been contested by the respondents on the grounds that it had been filed late and that it was post published and not relevant. D9 was provided with the statement of grounds of the appeal and as such forms the basis of the appeal proceedings according to Article 12(2) and (4) RPBA. D9 was said by the appellant to have been filed as a reply to the decision of the opposition division in order to demonstrate that the neutralization step carried out in D5 implicitly lead to the acid number defined in present claim 1. Indeed, point 3.1 of that decision concludes that D5 would not disclose a certain maximum acid number of the organohydrogensiloxane. D9 was therefore submitted at the earliest point in time after the decision of the opposition division was notified, that is with the statement of grounds of appeal. The Board sees no reason to exclude D9 from the appeal proceedings.

- 2.6.4 If D5 mentions that organohydrogensiloxanes may be rendered neutral (page 5, line 13), it does not provide any further detail on the neutralization process apart from that it may be conducted with sodium bicarbonate or sodium carbonate. D5 contains no reference to D9 so that it cannot be concluded that the neutralization referred to in D5 would have been performed as in D9. Also, on the basis of the information contained in D5 and D9, it cannot be concluded that the neutralization

of organohydrogensiloxanes will necessarily result in an acid number below 0.005 regardless of the process parameters used in the neutralization (type of base, its amount, process steps), within the range claimed in the patent in suit. As a result, it cannot be concluded that the neutralized organohydrogensiloxanes according to D5 had an acid number within the range of claim 1 of the patent in suit.

2.6.5 The polyethers that can be employed in D5 are disclosed on page 5 but neither their alkali metal content nor their method of preparation is disclosed. On the basis of the information contained in D5, it is not possible to ascertain whether the alkali metal content of these polyethers was within the range of claim 1 of the patent in suit.

2.6.6 A hydrosilylation process is disclosed on page 6 of D5. It is conducted in the presence of a noble metal hydrosilylation catalyst according to a process known in the art. No further requirement applying to that process is disclosed in D5. On the basis of the information contained in D5, it cannot be concluded that the process used would have implied or required a limitation of the acid number of the organohydrogensiloxane or of the alkali metal content of the polyether in the ranges of claim 1 of the patent in suit. Also, the general passage on page 5 (lines 41 to 44) only discloses that the process of D5 would reveal to be advantageous when the resulting product was used in a personal care product. The reference to a personal care product was not shown to imply any specific limitation regarding the acid number of the organohydrogensiloxane or of the alkali metal content of the polyether. It can therefore not be concluded that the process disclosed in D5 falls within the ambit

of claim 1 of the patent in suit.

2.7 D6

2.7.1 D6 discloses a method of making silicone copolymers comprising the steps of: providing one or more static mixing plug flow reactors; continuously feeding reactants to said one or more static mixing plug flow reactors, the reactants comprising hydrogen siloxane, at least one olefinic reactant capable of reacting with the hydrogen siloxane, and catalyst; and continuously withdrawing from a terminal end of said one or more static mixing plug flow reactors a product stream substantially free of unreacted hydrogen siloxane (claim 1).

2.7.2 The olefinic reactants that can be used in the hydrosilylation process of D6 may be polyethers as disclosed on page 10, lines 8 to 15. These are chosen so as to react with a hydrosiloxane (page 14, lines 14 to 16) and may include polyalkyleneoxide mono allyl ether, all ethylene oxide, mixtures of ethylene oxide and propylene oxide, or all propylene oxide (page 15, lines 10 to 18). Nowhere in D6 is the alkali metal content of these polyether olefinic reactants disclosed.

2.7.3 The organohydrogensiloxanes or hydrogen siloxanes used in the process of D6 are disclosed on pages 13 and 14. These compounds are said to be chosen such that the hydrogen siloxane contains sufficient R-containing siloxane units to provide an average of from 1 to 3.0 R radicals per silicon atom and sufficient H-containing siloxane units to provide from 0.01 to 1 silicon bonded hydrogen atoms per silicon atom and a total of R radicals and silicon-bonded hydrogen atoms of from 1.5

to 3.0 per silicon atom. The acid number of the organohydrogensiloxanes used in D6 is nowhere disclosed.

2.7.4 The passage on page 15, lines 29 and 30 of D6 concerns the general properties of the materials used in the hydrosilylation process. It discloses that the raw materials are preferably purified as known by one of skill in the art. D6 however does not provide any further information on the purification technique applied to the raw materials so that it cannot be deduced that that purification was in any way related to the acidity of the organohydrogensiloxanes and the alkali metal content of the polyether olefinic reactants. Thus, the raw materials may well be purified to perform the hydrosilylation process of D6 but still not meet the requirements set out in claim 1 of the patent in suit in terms of their alkali metal content and their acid number. It can therefore not be concluded from the information on file that the process of D6 falls within the ambit of claim 1 of the patent in suit.

2.8 D10/D10a

2.8.1 D10/D10a was introduced by the appellant into the proceedings with the statement of the grounds of appeal on the grounds that its filing was necessary because the opposition division had not found D5 or D6 to be novelty destroying. D10/D10a was therefore not filed in reaction to any specific argument pertaining to the novelty of the claims of the patent in suit in view of D5 or D6. D10/D10a could therefore have been introduced in the first instance proceedings. The Board therefore has the discretionary power to hold these documents inadmissible (Article 12(4) RPBA). The Board has prior

to executing that power taken their relevance for the issues of novelty and inventive step into consideration, on the basis of the machine translation D10a.

2.8.2 D10a discloses a hydrosilylation process comprising the reaction of an unsaturated polyether with an organohydrogensiloxane derived from triethylsilane (paragraph 45). The acid number of the organohydrogensiloxane disclosed in D10a is not disclosed. As a result, it cannot be concluded that the acid number of the organohydrogensiloxanes that can be used according to D10a are according to claim 1 of the patent in suit.

2.8.3 Claim 1 of the patent in suit is therefore novel over D10a. As a result, D10a is not relevant to the question of novelty of the claims.

2.9 The decision of the Boards of Appeal T 990/96 cited by the appellant deals with the problem of the novelty of low molecular organic compounds in the field of preparative organic chemistry. It was held in this decision that it is common practice in this field to purify a particular compound obtained in a particular manufacturing process according to the prevailing needs and requirements, and that, since conventional purification methods are within the common general knowledge in the field, a document disclosing a low molecular compound and its manufacture normally makes this compound available in all desired grades of purity, i.e. the purity level is not an essential feature for the definition of the organic compound. In contrast to T 990/96, the present case relates to a process for the manufacture of polymers having specific properties (i.e. hydrolyzable -SiOC- bonds)

characterised by the use of organic compounds having a required purity as starting components, i.e. the purity level of the starting components is therefore an essential technical feature of the process, which must be carried out in the required range of purity. Even if it could be considered that unsaturated polyethers and organohydrogensiloxanes were available at all grades of purity at the filing dates of D5, D6 and D10a, this would not imply that these components defined in either D5, D6 or D10a would necessarily and inevitably have exhibited the required acid number and alkali metal content as set out in claim 1 of the patent in suit. This in particular depends on the context of the disclosure of these documents. So even if the acid number and alkali metal content as defined in operative claim 1 are not explicitly disclosed in those documents, the question to be answered is whether in the context of these documents, it is implicit in view of the prevailing needs whether the purest compounds had to be used. In the case of T 990/96, which concerned a mixture of stereo isomers which could be separated by fractional crystallisation so that the product resolved into two optically pure enantiomers, the aim was one of achieving an ultimate degree of purity. Consequently, the general statements in T 990/96 concerning the purity of final products cannot be applied directly to the prior art documents cited in the present case because a purification step to obtain the purest possible compounds (A) and (B) is not disclosed in these documents.

- 2.10 In Decision T 242/88, the Board was of the opinion that the definition of a minimal grade of purity of the starting material was not a feature which distinguished the claimed process from the prior art, because it had not been disputed in this particular situation that the

purity of the starting material defined for the claimed subject-matter was usual at the filing date of the relevant patent. However, in the present case it has not been demonstrated that the degree of purity used for compounds (A) and (B) as defined in operative claim 1 was usual in the art at the date of filing of the present application.

- 2.11 It follows from the above considerations that the subject matter of claim 1 of the patent in suit is novel in view of D5, D6 and D10a (Article 54 EPC). The same conclusions apply for the subject matter of dependent claims 2 to 12.
3. Inventive step
 - 3.1 The patent in suit relates to a process for the preparation of silicone polyethers of improved quality having less hydrolyzable -SiOC- bonds (paragraphs 5 and 6). These silicone polyethers are ultimately used in the preparation of polyurethane foams (paragraph 3 and example 25).
 - 3.2 D5 concerns a process for the preparation of organosiloxane copolymer compositions that are suitable as surfactants in polyurethane foams (paragraph 1). D5 was considered to represent the closest prior art by both parties to the proceedings. The Board does not see any reason to deviate from that view.
 - 3.3 The organosiloxane copolymer compositions of D5 are prepared by hydrosilylation of a reaction mixture composed of an organohydrogensiloxane, an unsaturated polyoxyalkylene, an oil component and a hydrosilylation catalyst (claim 1). The process of claim 1 of the patent in suit differs from that of D5 in that the

unsaturated polyoxyalkylene or unsaturated polyether is characterised by an alkali metal content of less than 50 ppm and the organohydrogensiloxane is characterised by an acid number of less than 0.005. The alkali metal content of the unsaturated polyoxyalkylene and the acid number of the organohydrogensiloxane are not mentioned in D5.

3.4 The technical problem said to have been solved by these differences in the patent in suit is the provision of silicone polyether having less hydrolyzable -SiOC- bonds (paragraph 5).

3.4.1 The examples 2, 3, 22, 23 and 24 of the patent in suit describe the hydrosilylation of unsaturated polyethers and siloxanes wherein the content in alkali metal in unsaturated polyethers is between 1.2 and 5.0 ppm and the acid number of the siloxanes is below 0.001. The silicone polyethers obtained in these examples showed no detectable trace of -SiOC- impurity.

As a comparison, the examples 1, 8, 9 describe the preparation of silicone polyethers obtained under the same process conditions (process 1) but in which the alkali metal of the unsaturated polyether (examples 1 and 9) or the acid number of the siloxane (example 8) was outside the ranges of claim 1 of the patent in suit. These examples 1, 8 and 9 show that the silicone polyethers obtained by a process in which either the alkali metal content of the unsaturated polyether or the acid number of the organohydrogensiloxane was outside the claimed range all contained hydrolyzable -SiOC- bonds.

The examples 2, 3, 22, 23 and 24 of the patent in suit therefore show that the problem posed in the patent in suit of providing silicone polyethers having less hydrolyzable -SiOC- bonds has been solved by the

claimed subject matter.

3.4.2 That conclusion is not rebutted by the results of examples 4 and 5 of the patent in suit. These two examples describe the hydrosilylation of an unsaturated polyether containing 1.2 ppm of residual sodium and a siloxane with an acid number below 0.001. The starting materials used in the process of these examples are therefore according to claim 1 of the patent in suit. The process performed in these examples however differs from the process used in the other examples of the patent in suit in that additional alkali metal in the form of an acetate salt was added during hydrosilylation. Regardless of the amount of alkali metal salt added to the reaction medium during the process, examples 4 and 5 are according to claim 1 of the patent in suit since the amount in alkali metal of the unsaturated polyether starting material (A) (1.2 ppm) was within the claimed range. The amount of -SiOC- bonds of the resulting silicone polyethers is not disclosed in the case of examples 4 and 5. As a result, no conclusion can be drawn from the addition of alkali metal to the reaction medium during hydrosilylation reaction as far as the presence of -SiOC- bonds in the silicone polyether is concerned. The fact that the examples were identified as comparative in the patent in suit is at most a clarity issue that has no influence on the validity of the other examples contained therein.

3.4.3 D1 is an experimental report submitted by the appellant. It contains six examples of hydrosilylation according to the process 1 of example 2 of the patent in suit using Platine catalysts (WK and H₂PtCl₆ according to table 1). The unsaturated polyether provided by the appellant in examples 1 and 6 of D1

contains 2.1 ppm of sodium and 2.5 ppm of potassium resulting in a total alkali metal content of 4.6 ppm, within the range claimed in the patent in suit (less than 50 ppm). Prior to hydrosilylation, the unsaturated polyether was treated with china clay in the examples 2, 3, 4 and 5 in order to further reduce its alkali metal content to 0.4 ppm sodium and less than 0.1 ppm of potassium. The organohydrogensiloxane belonged to the sort mentioned in paragraph 30 of the patent in suit with an acid number of 0.006, outside the range of claim 1 of the patent in suit (less than 0.005). In examples 2, 3, 4 and 5 however, that organohydrogensiloxane was treated with 2 wt% sodium hydrogen carbonate to reduce its acid number to less than 0.002. D1 therefore shows that it was possible to obtain unsaturated polyethers and organohydrogensiloxanes with an alkali metal content and an acid number within and outside the ranges of claim 1 of the patent in suit.

3.4.4 Among the six examples of hydrosilylation described in D1, only those silicone polyethers obtained under the same reaction conditions can be compared to one another. That is the case for examples 1 and 4 in which the starting materials were reacted in the same molar amount (1:1) and with the same Platine catalyst (WK). That point was not disputed by the parties. The comparison of the silicone polyethers of examples 1 and 4 relies on the acid number of the organohydrogensiloxane, which is 0.006 in example 1, outside the claimed range (less than 0.005) and less than 0.002 in example 4 (within the claimed range). The amount in -SiOC- bonds in the silicone polyethers resulting from hydrosilylation are reported in Table 1 of D1. That table shows that the amounts in -SiOC- bonds that were determined vary for the same silicone

polyether depending on the way these amounts were calculated, i.e. whether they were calculated relative to i) the -SiC- bonds present in the polyether backbone, ii) the trimethylsilyl end groups of the silicone polyether and iii) all -SiC- bonds of the silicone polyether. Thus, the amount in -SiOC- bonds determined by method i) was 14 mol% in the case of both examples 1 and 4 while method ii) resulted in a value of 15.5 mol% (example 1) and 16.3 mol% (example 4) and method iii) resulted in a value of 3.8 mol% (example 1) and 1.9 mol% (example 4). Therefore, the silicone polyether of example 4 according to the patent in suit could be alternatively seen as equivalent (method i), better (method ii) or worse (method iii) in terms of the amount in hydrolyzable -SiOC- bonds than the silicone polyether of example 1 which was not according to the patent in suit. The Board can only conclude that the evidence provided in D1 is therefore in itself inconclusive in that it does not allow to precisely establish what is the effect of increasing the acid number of the organohydrogensiloxane in the hydrosilylation process.

3.4.5 From the examples provided in the patent in suit and in D1, it can be concluded that the technical problem effectively solved by the subject matter of claim 1 of the patent in suit is the provision of silicone polyether having less hydrolyzable -SiOC- bonds. The examples of the patent in suit show that that problem is effectively solved by a process in which both the alkali metal content of the unsaturated polyether and the acid number of the organohydrogensiloxane must be in the ranges as claimed.

3.5 It remains to be decided whether or not the claimed solution to the problem defined above was obvious in

view of the cited prior art.

- 3.5.1 D5 belongs to the same technical field as the patent in suit but it does not address the problem of hydrolyzable -SiOC- bonds formation occurring in the course of hydrosilylation. The alkali metal content of the unsaturated polyoxyalkylene and the acid number of the organohydrogensiloxane were also not addressed in D5. The passage on page 5, lines 10 to 14 describes the general preparation of organohydrogensiloxanes by acid equilibration of a mixture which may contain cyclic diorganosiloxanes and/or another source of organohydrogensiloxane. As part of that preparation, D5 indicates that the resulting silanic fluid may be rendered neutral through the use of mild bases which are typically sodium bicarbonate or sodium carbonate. It is not disclosed whether that treatment results in an organohydrogensiloxane having an acid number as claimed or not. In addition, that passage does not reveal the reason why the organohydrogensiloxane should be treated with a mild base and so cannot provide a pointer towards the use of that treatment in order to reduce the amount of hydrolyzable -SiOC- bonds that may be produced during hydrosilylation. As a result, D5 cannot render the claimed subject matter obvious.
- 3.5.2 Looking for a solution to the said technical problem, the person skilled in the art starting from D5 would have considered processes in the same technical field as in D6.
- 3.5.3 The object of D6 is merely to provide a continuous hydrosilylation process wherein the reactants do not undergo phase separation, wherein the crude product may be readily usable without further purification and in which the silicone copolymer produced does not require

further purification and is substantially free of hydrogen siloxane starting material (page 5, line 20 to page 6, line 7). D6 does not address the problem solved in the patent in suit, the reduction of hydrolyzable - SiOC- bonds formation as a result of hydrosilylation. Starting from D5 as the closest prior art, D6 can thus not provide an incentive to modify the process of D5 to solve the technical problem posed above.

3.5.4 Also, D6 does not disclose any of the acid number or the alkali metal content of the starting materials. The passage on page 15, lines 29 and 30, indicating that the raw materials were purified as is known by one of skill in the art does not provide any further insight on the purity of these raw materials so that it cannot be inferred from that passage if that purity pertains to the alkali metal content of the unsaturated polyether and the acid number of organohydrogensiloxane and even if it did, the passage does not quantify that purity so that it would not provide an incentive towards the claimed ranges. As no further information on the purification method applied in D6 was made available to the Board, it cannot be concluded that that passage provided an incentive towards the claimed subject matter. Therefore, neither D5 nor D6 provides a motivation towards the claimed subject matter.

3.5.5 D10a aims at avoiding discoloration of the silicone polyether as a result of their preparation (paragraph 1). To that effect, D10a discloses a process based on the use of an unsaturated polyether having (i) an aldehyde content less than 0.1 wt.%; (ii) a peroxide value of less than 5; and (iii) an alkali metal content of less than 2 ppm (claim 1). The problem solved in D10a does not relate to the presence of hydrolyzable - SiOC- bonds on the silicone polyether produced and so

D10a does not provide a hint towards the subject matter of claim 1 the patent in suit. Also, no specific requirements concerning the purity or the acid number of the organohydrogensiloxanes are set out in D10a so that D10a cannot provide any incentive to use organohydrogensiloxanes with an acid number as claimed in the patent in suit. D10a is therefore not relevant to the question of inventive step, and the Board holds the document inadmissible (Article 12(4) RPBA).

3.6 Claim 1 is therefore inventive in view of D5 and the other cited prior art. The same conclusions apply for the subject matter of dependent claims 2 to 12. The main request satisfies the requirements of Article 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



B. ter Heijden

F. Rousseau

Decision electronically authenticated