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**Datasheet for the decision
of 2 July 2013**

Case Number: T 0914/10 - 3.3.03

Application Number: 01995168.0

Publication Number: 1341843

IPC: C08L23/16

Language of the proceedings: EN

Title of invention:

MINERAL-FILLED ELASTOMER COMPOSITIONS

Patent Proprietor:

Momentive Performance Materials Inc.

Opponent:

Evonik Degussa GmbH

Headword:

Relevant legal provisions:

EPC Art. 56

RPBA Art. 13(1), 13(3)

Keyword:

Inventive step - non-obvious alternative - main request (yes)
Late-filed evidence - admitted (no)

Decisions cited:

Catchword:



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Case Number: T 0914/10 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 2 July 2013

Appellant: Momentive Performance Materials Inc.
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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted on 2 March 2010
revoking European patent No. 1341843 pursuant to
Article 101(3) (b) EPC.**

Composition of the Board:

Chairwoman: B. ter Laan
Members: O. Dury
C. Brandt

Summary of Facts and Submissions

- I. The appeal by the patent proprietor lies against the decision of the opposition division posted on 2 March 2010 to revoke European patent No. EP 1 341 843, based on application No. 01 995 168.0.

- II. Notice of opposition to the patent was filed on 9 October 2007, requesting revocation of the patent in its entirety on the grounds of Art. 100 (a) EPC (lack of novelty and of inventive step).

- III. The decision of the opposition division was based on a main and five auxiliary requests, auxiliary request I being the only request relevant for the present decision. According to the appealed decision, claim 1 of auxiliary request I was novel considering in particular that D1 (WO 99/09036) did not disclose the specific amounts of some of the compounds mentioned in claim 1. However, claim 1 of auxiliary request I did not fulfil the requirements of Art. 56 EPC because "[t]he Patentee was not in a position to present comparative examples proving the benefits due to the composition under consideration vis-à-vis to composition which has not the ratio (B) to (E) as claimed in claim 1 in suit". Therefore, there was no evidence that the problem of preventing premature scorching of the composition at elevated temperatures had been solved.

- IV. On 29 April 2010, the patent proprietor (appellant) lodged an appeal against the above decision. The prescribed fee was paid on the same day. In its statement of grounds of appeal filed on 8 July 2010, corrected by letter dated 9 July 2010, the appellant

requested that the decision of the opposition division be set aside and that the patent in suit be maintained in amended form according to either the main request or any of auxiliary requests 1-11 filed therewith. In addition, annexes I to IV were filed:

Annex I: Impurity Identifications of 3-(Trimethoxysilyl)-1-Propyl-Thioacetate (5 pages)

Annex II: GC/MS Analysis of The Product Mixture of mercaptopropyltrimethoxysilane/Acetic Anhydride Reaction (6 pages)

Annexes III-IV: Experimental data concerning repetitions of example 12 of D1 using various amounts of a blocked mercaptosilane and of a free mercaptosilane

Further arguments, means of proof and new documents were submitted by letter dated 21 January 2013.

- V. By letter dated 28 October 2010 the opponent (respondent) requested that the appeal be dismissed and raised objections *inter alia* pursuant to Art. 56 EPC and Art. 100(b) EPC together with Art. 83 EPC. The following documents, *inter alia*, were also filed:

Anlagen 3-4: Figures plotting "Zugfestigkeit" and "Mooney Scorch" against "Anteil Mercaptosilan zu Ges.silanmenge" related to Annexes III-IV (2 pages)

- VI. In a communication accompanying the summons to oral proceedings issued on 26 February 2013 the Board set out its preliminary view of the case, *inter alia* regarding the inadmissibility of the objection of insufficiency of disclosure. As to inventive step, it questioned in particular whether any data on file

illustrated the subject-matter defined in the main request in either claim 1 or in each of both alternatives of claim 10 in which activator C) is added in step D) either only in step A) or in both steps A) and C).

- VII. By letter dated 2 May 2013, new auxiliary requests 1-4 were filed in replacement of all former auxiliary requests. Also, a further annex was submitted:

Annex VII: Experimental data concerning the preparation of sulphur vulcanisable rubber compositions using various amounts of a blocked mercaptosilane and of a free mercaptosilane

- VIII. At the beginning of the oral proceedings held on 2 July 2013 in the presence of both parties the respondent withdrew its objection pursuant to Art. 100(b) and 83 EPC. During the oral proceedings, the appellant replaced the main request then valid with a new main request, claims 1 and 10 of which read:

"1. A composition of matter comprising:
A) at least one sulfur vulcanizable rubber;
B) at least one latent mercaptosilane coupling agent;
C) at least one latent mercaptosilane activator;
D) at least one filler; and
E) at least one additional mercaptan of the structure
 $(\text{HS-})_r\text{G}(-\text{SiX}_3)_t$,

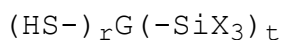
wherein: <...>,

wherein the mixture of the latent mercaptosilane coupling agent and the additional mercaptan contains 80 to 99 weight percent latent mercaptosilane and 1 to 20 weight percent additional mercaptan."

"10. A process for the preparation of a rubber composition consisting essentially of the steps of :

A) thermomechanically mixing in a first pass:

- 1) at least one sulfur vulcanizable rubber;
 - 2) at least one latent mercaptosilane coupling agent;
- and
- 3) at least one filler; and
 - 4) at least one additional mercaptan selected from the group consisting of compounds of the structure



wherein <...>;

wherein the mixture of the latent mercaptosilane coupling agent and the additional mercaptan contains 80 to 99 weight percent latent mercaptosilane and 1 to 20 weight percent additional mercaptan,

B) bringing the temperature of the first pass mix from about 140°C to about 200°C over a period of 1 to 20 minutes; thereby forming a nonproductive first mixture;

C) thermomechanically mixing the nonproductive first mixture in a second pass, optionally in the presence of at least one curing agent, at a temperature of from about 25 to about 100°C for a period of from about 1 to about 30 minutes; thereby forming a productive second mixture;

D) adding at least one latent-mercaptosilane activator in step A), step C), or both step A) and step C);

E) shaping the second mixture; and then

F) curing the second mixture at a temperature between about 130°C to about 200°C for a period of from about 5 to about 60 minutes."

Claims 2-9 and 11-16 were dependent on claims 1 and 10, respectively.

The exact meaning of G, X, r and t for the additional mercaptan E) (indicated above as "<...>") is not relevant for this decision.

IX. The appellant's arguments relevant for the present decision may be summarised as follows:

Main request

Reading of claim 1

- a) Claim 1 was directed to compositions obtainable from components A) to E). In view of the patent specification, those compounds could be present in the composition in a reacted state e.g. components B) and E) being coupled to the filler D) through reaction of their silane groups. The amounts of B) and E) specified in claims 1 and 10 corresponded to the amounts at the moment of their addition. Even if the silanes B) and E) had reacted with filler D), it was possible to determine those amounts in the composition.
- b) Should the Board disagree with that reading, the claims could also be seen in a more restricted

sense as comprising all components A) to E) *per se*, i.e. in a non-reacted state.

Inventive step

- c) Starting from D1 as the closest prior art, the problem to be solved was to provide vulcanisable rubber compositions that did not undergo premature scorch and exhibited improved performance as compared to those of D1 (paragraph [0017] of the patent in suit).
- d) The solution to that problem resided in the compositions according to claim 1 and in the processes according to claim 10. The subject-matter of claims 1 and 10 differed from D1 at least in that it imposed the combination of a blocked mercaptosilane B) and a free mercaptan/ mercaptosilane E) in specific amounts.
- e) Regarding the question whether the problem had been solved by the claimed subject-matter, Compound 20AB of the patent in suit illustrated the subject-matter of claim 1 and of that alternative of claim 10 in which in step D) the activator is added in both step A) and step C). Although not explicitly disclosed, it was derivable from the patent specification that the activator was added to a composition comprising components A), B), D) and E) in which the filler D) and the silanes had not yet reacted. Reference was made in particular to paragraphs [0133], [0134], [0135], [0145] and [0152]. Considering the short times and the slow speed of mixing, the temperature would not have risen to such an extent as to enable the coupling of the filler to the

silanes. That conclusion was supported by the data provided in Table 3 of Annex VII. Should the activator have been added during the nonproductive step and after the coupling of the filler and the silanes had occurred, as argued by the respondent, the process used in experiment 20AB of the patent in suit would not correspond to any of the processes defined in claim 10, which did not make sense.

The data reported in the table of paragraph [0154] of the patent in suit showed that Compound 20AB exhibited improved properties as compared to e.g. Compound 4AB, which was illustrative of the teaching of D1.

- f) That conclusion was confirmed by the compositions prepared in examples 9-13 of Annex VII, which was submitted in reply to the communication from the Board, wherein it was questioned for the first time whether any of the data on file illustrated the subject-matter being claimed. Annex VII was filed as soon as possible and constituted a *bona fide* reply to the objection made. Therefore, it should be admitted to the proceedings. Annex VII also showed an improvement in the overall performance, compared to D1, of compositions prepared according to the alternative of claim 10 in which activator C) had been added in both step A) and step C).

- g) The compositions prepared in examples 3-8 of Annexes III-IV and in examples 2-6 of Annex VII illustrated the subject-matter of the alternative of claim 10 in which activator C) had been added only in step C). Annex IV and Annex VII

(Tables 4-8) showed that compositions prepared according to said process exhibited a better set of properties, in particular in terms of modules, reinforcement index and tensile strength as compared to compositions prepared according to D1. Although it could not be concluded that each property was improved for all embodiments according to the subject-matter now being claimed, the data showed that for each of those compositions various properties were improved. The subject-matter claimed was thus shown to have an improved overall performance compared to D1.

The compositions prepared in examples 9-13 of Annex VII illustrated the subject-matter of that alternative of claim 10 in which activator C) had been added in both steps A) and C). The data of Annex VII also showed an improvement in the overall performance of the compositions prepared as compared to D1.

There was no example on file illustrative of the alternative of claim 10 in which activator C) had been added only in step A) (and not in step C)). However, the respondent had not provided evidence that that embodiment did not solve the technical problem posed.

Under these circumstances the problem of providing improved vulcanisable rubber compositions as compared to D1 was effectively solved by the subject-matter according to claims 1-16 of the main request.

- h) D1 aimed at providing vulcanisable rubber compositions that did not undergo premature scorch

and provided the solution of using blocked mercaptosilanes instead of free mercaptosilanes, i.e. replacing all the free mercaptosilanes with blocked mercaptosilane. Therefore, D1 taught away from the solution provided in the patent in suit. D1 further failed to disclose compositions simultaneously comprising components B), C) and E) according to claim 1 of the main request. In that respect, the appellant had shown in Annexes I-II that, in the compositions of D1, the free mercaptosilane E) was only present in an amount of 0.02 wt.%, which was far less than the amount specified in each of claims 1 and 10 of the main request. In addition, in D1 the filler and the blocked mercaptosilane would have already reacted with each other before the deblocking occurred and the free mercaptosilane was formed. Such free mercaptosilane was also very reactive towards rubber; so it was present only as a transient species. Therefore, the compositions disclosed in D1 could not contain a blocked mercaptosilane B) and a free mercaptan/mercaptosilane E) in the amounts specified in claims 1 and 10 of the main request; so D1 did not teach the use of such amounts.

- i) Under these circumstances, the subject-matter claimed was not obvious over D1.

X. The respondent's arguments relevant for the present decision may be summarised as follows:

Main request

Reading of claim 1

- a) Claim 1 was a product claim directed to a composition comprising components A) to E). Therefore those components had to be present *per se*, i.e. in an unreacted state, in the composition. That line of reasoning was followed by the appellant to distinguish the compositions disclosed in D1 from those claimed and it was also to be adopted for the claims of the patent in suit.

Inventive step

- b) The subject-matter of claim 1 differed from the compositions of the closest prior art D1 only in the specific amounts of components B) and E).
- c) The problem to be solved was to provide vulcanisable rubber compositions that did not undergo premature scorch and maintained the performance of mercaptosilane-containing rubber compositions.
- d) The patent in suit provided no information regarding the moment of addition of the activator apparently used in the nonproductive step of Compound 20AB of the patent in suit (paragraphs [0133] and [0134]). It may have been added only after the filler D) and the silanes B) and E) had reacted e.g. once a temperature of 170°C in process step 7 according to paragraph [0134] was achieved. In that respect, the temperature was expected to rise upon mixing and it could not be ruled out that temperatures suitable for the coupling reaction of the filler and the silanes were achieved already in the first mixing steps. In fact, the second line of paragraph [0152]

indicated the addition of the activator at 170°C. The reference made by the appellant to Table 3 of Annex VII was not relevant since the compositions used in Annex VII and for the preparation of Compound 20AB of the patent in suit, in particular the rubbers, were different. Therefore, it could not be concluded that Compound 20AB of the patent in suit illustrated the subject-matter of claim 1.

The compositions prepared in Annex III-IV or in examples 2-6 of Annex VII also did not illustrate the subject-matter of claim 1 since the activator was only added in the productive step, when the filler and the silanes had already reacted with one another. They could only illustrate the subject-matter of the alternative of claim 10 in which activator C) is added in step C) and not in step A).

The compositions prepared in examples 9-13 of Annex VII illustrated the subject-matter of the alternative of claim 10 in which activator C) is added both in step A) and in step C).

Therefore, there was no example on file illustrating the subject-matter of either claim 1 or of the alternative of claim 10 in which activator C) is added only in step A) and not in step C).

- e) There was also no evidence on file showing that at least one property of the vulcanised rubber was improved over the whole range of the claims. In Anlagen 3-4, the data relating to tensile strength and scorch time reported in Annexes III-IV were analysed taking into account the measurement error

indicated in Anlagen 1-2. Those data demonstrated that there was no improvement within the range of components B) and E) defined in claims 1 and 10.

Under these circumstances, the subject-matter of claims 1 and 10 was not related to any improvement. That conclusion was confirmed by the fact that none of the properties reported in Annex IV was effectively improved over the whole range of the amounts of blocked mercaptosilane B) and free mercaptan/mercaptosilane E) specified in claim 1. Therefore, the technical problem effectively solved resided in the provision of further vulcanisable rubber compositions and processes of preparation thereof as an alternative to those of D1.

- f) Examples 2-4 of D1 (Table 2) showed that the use of a reduced amount of a free mercaptosilane corresponding to component E) according to present claim 1, led to vulcanisable rubber compositions having a scorching time comparable to that of similar compositions with higher amounts of blocked mercaptosilane corresponding to component B) of claim 1. Although some properties deteriorated, the compositions comprising the free mercaptosilane exhibited an improved reinforcing index compared to compositions containing a blocked mercaptosilane. Combinations of blocked and free mercaptosilanes were described in D1 (page 23, lines 13-15; page 47: examples A-C). Annexes 1-2 further showed that the blocked mercaptosilanes prepared in D1 always contained some free mercaptosilanes. A combination of components B) and E) according to claim 1 of the main request was, thus, already disclosed in D1.

- g) Those results provided a hint to the skilled person to solve the technical problem identified above by using small amounts of free mercaptosilane together with blocked mercaptosilanes. Complete replacement of the blocked mercaptosilane with free mercaptosilanes was however not suggested, considering the deterioration in scorching time to be expected. The same conclusion applied when the problem to be solved was seen as improving the reinforcing index.

- h) The subject-matter of at least claims 1 and 10 was, therefore, not inventive.

Admissibility of Annex VII

- i) Annex VII was submitted very late, only after the communication from the Board. The question whether or not any of the data on file illustrated the subject-matter being claimed had however already been identified in the respondent's reply to the statement of grounds of appeal. Hence Annex VII had not been filed as soon as possible. It raised new issues and was based on new arguments in respect of inventive step that the respondent could not deal with without adjourning the oral proceedings. Therefore, it should not be admitted to the proceedings.

XI. The appellant (patent proprietor) requested that the decision under appeal be set aside and that the patent be maintained in amended form on the basis of the (new) main request (claims 1-16) as submitted during the oral proceedings on 2 July 2013 or, alternatively, on the

basis of the auxiliary requests as filed with letter of 2 May 2013.

The respondent (opponent) requested that the appeal be dismissed.

XII. The Board announced its decision at the end of the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

Main request

2. Subject-matter claimed

2.1 Claim 1 is a product claim directed to a composition comprising components A) to E).

2.2 Normally, the wording of the claim has to be taken in its normal meaning. According to the wording used, the compositions of claim 1 must comprise each of the components defined therein as such, i.e. in an unreacted state.

2.3 The appellant argued that compounds A) to E) specified in claim 1 could be present in the claimed composition as such or in a reacted state, e.g. components B) and E) being coupled to the filler D) through reaction of their silane groups.

As explained in paragraphs [0077]-[0078] of the patent in suit the compositions claimed are typically prepared in a sequential, step-wise manner by a process comprising

- a nonproductive step, wherein the rubber and various ingredients, usually exclusive of sulphur and curing agents, are blended in at least one thermomechanical mixing stage, typically at temperatures of 140°C-200°C; followed by
- a productive step, wherein curing agents, sulphur and other ingredients are mixed with the rubber composition at lower temperatures of e.g. 50°C-110°C;
- when desired, a moulding/curing step performed at a temperature of at least 130°C.

As further explained in paragraphs [0082] and [0083] of the patent in suit, several reactions may occur at various steps in the mixing and curing processes. Depending on the experimental conditions, in particular temperature, those components may react as follows:

- a coupling reaction may occur between the filler D) and the $-SiX_3$ group of the latent mercaptosilane coupling agent B) at temperatures around 120°C. It was also agreed by the parties that the same kind of reaction may take place for the additional mercaptan E) having $-SiX_3$ end groups;
- a reaction may take place at temperatures around 140°C between the sulphur-containing portion of the - deblocked - latent mercaptosilane coupling agent B) and the sulphur vulcanisable rubber A). It was also agreed by the parties that the same kind of reaction may take place for the sulphur portion of the additional mercaptan E).

However, those paragraphs are a general description of possible reactions at various stages of the process, but there is no indication in the patent in suit that claim 1 should be read in any other sense than according to its normal meaning, in particular as encompassing compositions wherein the mercaptosilanes B) and E) are coupled to the filler. Therefore, in the present case, there is no reason to depart from the literal wording of the claims, so that the amounts specified therein concern the products as such, i.e. in an unreacted state.

- 2.4 Process claim 10 contains three alternative embodiments in step D), the moment of addition of activator C) being defined as taking place either i) in step A), ii) in step C) or iii) in both steps A) and C).

Claim 10 has no direct reference to claim 1. A composition according to claim 1 may, however, be used in the process according to claim 10, alternative i) or alternative iii), when the activator C) is added in step A) under conditions at which it is not active and components A), B), D) and E) have not reacted.

- 2.5 Hence, the main request includes
- a) claim 1 and claim 10, alternatives i) and iii), which are directed to compositions each comprising components A) to E) as such;
 - b) claim 10, alternative ii), which makes use of a composition comprising only components A), B), D) and E) as such which are processed in a first nonproductive pass at temperatures at which the filler D) reacts with the blocked mercaptosilane B) and the free mercaptan E), the activator C) being added to the reaction mixture so obtained in a further productive step.

3. Inventive step

3.1 Closest prior art

3.1.1 The patent in suit relates to the preparation and processing of mineral-filled elastomer compositions (paragraph [0001] of the patent in suit).

3.1.2 Such compositions and processes are known from D1 (claim 10; examples 12-13), which discloses sulphur vulcanisable rubber compositions prepared by a process in which:

- the rubber, a blocked mercaptosilane and an inorganic filler are mixed;
- together with a curing agent, a deblocking agent is added to the above mixture to deblock the mercaptosilane;
- the mixture is cured.

In the nonproductive step of the process used in D1, the rubber (corresponding to compound A) according to present claim 1), the blocked mercaptosilane (corresponding to compound B) according to present claim 1) and the inorganic filler (corresponding to compound D) according to present claim 1) are mixed together, whereby the filler may react with the $-SiX_3$ group of the blocked mercaptosilane (D1: page 25, lines 13-15).

In the thermomechanical mixing stage of D1, the deblocking agent (corresponding to compound C) according to present claim 1) is added together with the vulcanisation agent, deblocking the coupling agent to result in an additional mercaptan (corresponding to compound E) according to present claim 1), which reacts

through its sulphuric part with the rubber (D1: page 25, lines 15-18).

Therefore, at the moment of addition of the deblocking agent, at least part of the latent mercaptosilane coupling agent reacts with the filler and is no longer present as such in the composition. Besides, when the deblocking agent is added it reacts with the blocked mercaptosilane coupling agent to produce a free mercaptosilane, which itself reacts further with the rubber.

Annexes I-II show that the amount of free mercaptosilane in the composition of example 2 of D1 is only 0.02 wt.%, which is below the lowest amount defined in present claims 1 and 10.

It is also stated on page 23, lines 13-15, of D1 that partial amounts of activator may be used. That statement is supported by example 13 of D1, in which three different amounts of the activator N,N'-diphenylguanidine (DPG) were used in runs A, B and C. However, there is no information on file showing that the amounts of blocked mercaptosilane and free mercaptan/mercaptosilane in any of runs A to C of D1 are in the range defined in claim 1 of the present main request. Also, there is no evidence that such a composition still comprises an activator as required by present claim 1. Finally, in such compositions the silanes will - at least partly - have reacted with the filler and would no longer be present in the amounts in which they were introduced into the composition.

- 3.1.3 Both parties considered D1 to be the closest prior art document, and the Board sees no reason to depart from that view.

3.2 Problem to be solved

Paragraph [0017] of the patent in suit identifies the problem to be solved as being that of providing rubber compositions that do not undergo premature scorch at elevated temperature processing and maintain the performance of mercaptosilane-containing rubber compositions.

During the appeal proceedings, the appellant formulated the problem to be solved as compared to D1 as being to provide sulphur vulcanisable rubber compositions that do not undergo premature scorch at elevated temperature processing (i.e. having a scorch time $t_3@135^\circ\text{C}$ between 7-11 minutes) and that have an overall improved combination of physical and dynamic properties as compared to compositions comprising only a blocked mercaptosilane or only a - non-blocked - mercaptosilane. In view of e.g. paragraphs [0026], [0151] and [0154] of the patent in suit, that formulation of the problem is also acceptable.

3.3 Solution

The solution to the problem identified above resides in the compositions and processes defined in claims 1 and 10.

As can be seen from section 3.1.2 above, the subject-matter of claim 1 and of both alternatives of claim 10 step D) in which the activator is added at least in step A) differs from D1 in that an activator is present together with a combination of a blocked mercaptosilane B) and a free mercaptan/mercaptosilane E) in specific amounts.

The alternative of claim 10 in which in step D) the activator is added only in step C) differs from D1 in that a combination of a blocked mercaptosilane B) and a free mercaptan/mercaptosilane E) in specific relative amounts is used in the nonproductive step.

3.4 Success of the solution - Problem effectively solved

3.4.1 Compound 20AB of the patent in suit (page 24, second table) is a rubber composition prepared in a nonproductive step (paragraphs [0133]-[0134]) and a productive step (paragraph [0145]), respectively, as follows:

[0133] For Compound 20AB 9.7 phr Silane A + 0.16 phr Silane C + 2.0 phr DPG (1 pass), 72% fill factor.

[0134] Temperature of sides, rotors, door set at 5° C.

1. Add SSBR and BR, ram down mix (RDM) 30 seconds at 41 RPM.
2. Add nine-tenths of the total quantity of silica, all silane, RDM 30 seconds.
3. Add one-tenth of the total quantity of silica, all oil, RDM 30 seconds.
4. Brush (sweep), RDM 20 seconds.
5. Brush; RDM 20 seconds.
6. Add ZnO, stearic acid, wax, 6PPD, carbon black, RDM 30 seconds.
7. Brush. RPM to 71, RDM to 170° C.
8. Hold at 170 ° C for eight minutes by adjusting RPM to 26.
9. Dump. Sheet off 1525 mm. roll mill. Cool to room temperature.

[0145] Second pass for 1 pass compounds, and 3rd pass for 2 pass compounds.

1. Band 600 grams of the compound from the end of the 1st or 2nd pass on a 300 mm. roll mill, roll temperatures 50-60° C.
2. Add 3.66 gm sulfur, 4.45 gm CBS, 5.23 gm DPG.
3. Mix by cutting six times on each side, folding the sides into the center of the mill. Allow mixing time between cuts.
4. Sheet off mill and cool to room temperature.

In Compound 20AB SSBR (solution-prepared styrene-butadiene rubber) and BR (polybutadiene), Silane A, DPG, silica and Silane C correspond to components A) to E), respectively, according to present claim 1 (paragraphs [0063] and [0106] of the patent in suit).

The patent in suit contains no indication at which stage of the nonproductive step of the preparation

process of Compound 20AB the activator (DPG) was added, in particular if it was mixed into a composition comprising components A), B), D) and E) as such or in a reacted state. However, DPG is also used in the preparation of compounds 17AB, 21AB and 23 AB, which are prepared using a similar nonproductive step (paragraphs [0131], [0135] and [0139] of the patent in suit). In the first two sentences of paragraph [0152] it is explained that in experiments 17AB and 21AB, small amounts of DPG were used "early on along with a 90% silica addition step", experiments 17AB and 21AB being run at a thermal step of 170°C and 180°C, respectively. In paragraph [0153] it is further indicated that experiment 23AB deals with the addition of DPG and the use of a temperature of 170°C for the silanisation step and that in experiment 22AB the silanisation step is run at 160°C. On the basis of that information, it is concluded that silanisation, i.e. the coupling reaction between the filler D) and the mercaptosilanes B) and E), does not take place until step 7 of the nonproductive step specified in paragraph [0134] of the patent in suit.

That conclusion is in line with the appellant's argumentation that no coupling reaction takes place in steps 1-6 of said process because of the low mixing speed and short mixing times. Although it was agreed by the parties that the temperature increases upon mixing, there is no evidence on file that the temperature rise is such that it leads to the coupling of silica (filler D)) and silanes A and/or C (components B) and E) according to claim 1).

Under these circumstances, Compound 20AB of the patent in suit can be considered to illustrate the subject-matter of claim 1. Since the activator (DPG) is used

both in the nonproductive step (paragraph [0134]) and in the productive step (paragraph [0145]), experiment 20AB also illustrates the alternative of claim 10 in which the activator is added in both steps A) and C).

3.4.2 Since Compound 20AB illustrates the subject-matter now being claimed, it can be compared with other experimental data in order to establish whether its performance is improved over that of prior art compounds. To that end, the appellant relied upon Compound 4AB (table of paragraph [0154]). However, the nonproductive step of experiment 4AB differs from that of experiment 20AB not only in the nature of the components used (4AB: only blocked mercaptosilane and no activator; 20AB: activator (DPG) in combination with both blocked and free mercaptosilane) but also in that it was carried out under different working conditions (e.g. rubber fill factor, different amounts of silica introduced in each of the two addition steps, temperature and mixing speed for the silanisation). Hence no fair comparison can be made between Compounds 4AB and 20AB, and the appellant's argument relying on an improvement in properties shown by these examples cannot be followed.

3.4.3 Annexes III-IV deal with the preparation of compositions according to Example 12 of D1 using various amounts of blocked mercaptosilane (Silquest NXT: component B) of claim 1) and/or free mercaptosilane (Silquest A-1891: component E) of claim 1). Comparative example 1 was performed using only blocked mercaptosilane and is illustrative of the teaching of D1. Examples 3-8 were carried out using mixtures of blocked mercaptosilane and free mercaptan in amounts according to present claim 1. The results

reported in Annex IV show that the scorch time of comparative example 1 and examples 3-8 is acceptable ($t_3@135^\circ\text{C}$ between 7 and 11 minutes). However, no improvement over the whole range of the claims could be identified in any of the properties reported. In that respect, example 4 shows no improvement in tensile strength for a ratio B):E) of 98:2, and example 3 shows no improvement in reinforcement index for a ratio B):E) of 98.83:1.17. Similar conclusions may be drawn regarding each of the parameters reported in Annex IV. Therefore, from the experimental data on file it cannot be concluded that the overall performance of rubber compositions according to the patent in suit is improved.

In view of the above, the appellant's argument that the "overall performance" of rubber compositions was improved because for each of the examples 3-8 at least some properties were improved, even if other properties deteriorated, cannot be followed.

3.4.4 The appellant further relied on Annex VII.

The admission to the proceedings of Annex VII, which was filed at a very late stage of the proceedings, namely in reply to the Board's communication accompanying the summons to oral proceedings, is subject to the Board's discretion (Art. 13(1)(3) RPBA).

The question whether or not any of the data on file illustrated the subject-matter being claimed had already been identified in the respondent's reply to the statement of grounds of appeal, although in a different context (objection under Art. 100(b) EPC). Hence the appellant's justification for the late filing of Annex VII cannot be followed. Annex VII further

raises *prima facie* concerns related to substantive issues, e.g. in relation to Art. 56 EPC. It appears in particular questionable whether Annex VII could demonstrate that an improvement over D1 in at least one property is present over the whole scope of the claims (Tables 6-9). In any case, Annex VII could not help refuting the conclusion drawn above from Annexes III-IV that the improvement relied upon by the appellant is not present over the whole scope of the claims.

Under these circumstances, the Board decided not to admit Annex VII into the proceedings (Art. 13(1) and 13(3) RPBA).

- 3.4.5 There is no information on file illustrating the alternative of claim 10 in which the activator is added in step A) (and not in step C)), which was not contested by the appellant. Therefore, in the absence of any comparison to D1, it cannot be concluded that that embodiment is related to an improvement over D1.
- 3.4.6 Since no improvement over the closest prior art D1 was shown, the technical problem effectively solved by the subject-matter of the main request has to be reformulated and can only be seen as being to provide further sulphur vulcanisable rubber compositions that do not undergo premature scorch at elevated temperature processing and that have the performance of mercaptosilane-containing rubber compositions according to D1.
- 3.4.7 Examples 5/15AB-16AB and example 8/20AB are the only examples of the patent in suit dealing with vulcanised rubber prepared using an additional mercaptan corresponding to compound E) according to claim 1 (see paragraphs [0127-0130] and [0145] of the patent in

suit). Similar rubbers are also prepared in examples 3-8 of Annexes III-IV. In all these examples, the latent mercaptosilane activator (DPG) is added either only in the productive step (15AB, 16AB, Annexes III-IV) or both in the nonproductive and productive steps (20AB). These examples show that the problem identified in section 3.4.6 is effectively solved by a composition according to claim 1 and by a process according to operative claim 10 in which the activator is added in step C) or in both steps A) and C).

None of the examples of the patent in suit actually deals with a process according to operative claim 10 in which the activator is added only in the nonproductive step A). However, in the absence of any evidence to the contrary, it is credible that such a process may successfully lead to the preparation of sulphur vulcanisable rubber compositions having an acceptable scorch time and properties similar to those of D1. That conclusion was never contested by the respondent. Therefore, there is no reason to consider that the problem defined in section 3.4.6 would not be solved by the subject-matter so being claimed.

3.4.8 Since the problem effectively solved is formulated as being to provide an alternative to the closest prior art D1, the respondent's arguments relying on Anlagen 3-4 refuting the alleged improvement over D1 need not be considered.

3.5 Obviousness

3.5.1 It remains to be decided whether it was obvious to modify the teaching of D1 such as to arrive at the subject-matter of claims 1 and 10 of the main request, i.e. in particular whether it was obvious to prepare

compositions comprising both a blocked mercaptosilane B) and a free mercaptan/mercaptosilane E), optionally together with an activator C).

3.5.2 D1 teaches to replace free mercaptans/mercaptosilanes with blocked mercaptosilanes in order to provide sulphur vulcanisable rubber compositions having an improved scorch time (page 21, line 14, to page 22, line 2; page 46: examples 1-4). Hence, in order to arrive at the solution proposed by the patent in suit and use free mercaptosilanes, the skilled person would have to go against the teaching of D1.

3.5.3 In line with its teaching, D1 further nowhere suggests the use of a combination of blocked and free mercaptan/mercaptosilane.

Even if partial amounts of activator were used (i.e. a stoichiometric deficiency) if only part of the blocked mercaptosilane were deblocked (D1: page 23, lines 13-15; page 47, examples A-C), there is no evidence on file showing that the ratio of blocked mercaptosilane B) to free mercaptan/mercaptosilane E) in any of runs A-C of D1 was in the range defined in present claim 1. In that respect, the appellant's argument that the unblocked mercaptosilane B) rapidly reacts with the rubber and is thus present only as a "transient" component in the composition was not contested. Also, there is no evidence that such a composition would comprise an activator C) according to present claim 1, which will be depleted upon activation of the blocked mercaptosilane. Finally, in all the examples of D1 the activator is added in the productive step, after thermomechanical mixing at a temperature of 160-165°C of the rubber, filler and blocked mercaptosilane (D1: page 43, lines 3-17). In those compositions the silanes

have at least partly reacted with the filler and are no longer present in the amounts in which they were introduced into the composition. The teaching on page 23, lines 17-18, of D1 that "the deblocking agent could be added in the curative package or, alternatively, at any other stage in the compounding process as a single component", which was relied upon by the respondent during the oral proceedings before the Board, does not affect that conclusion since it does not explain when and under which conditions (in particular regarding temperature and components) the deblocking agent is to be added.

The respondent further argued that Annexes 1-2 showed that some free mercaptan E) would always be present at least as a residue in compositions of blocked mercaptan B). However, the appellant demonstrated that in the examples of D1 the free mercaptan would be present only in amounts lower than the minimum value specified in claim 1. Besides, as considered above, at the time of addition of the activator C) according to D1 the mercaptosilanes and the filler will already have been coupled and will not be present *per se*.

For these reasons, D1 contains no hint to use a combination of blocked and unblocked mercaptans in amounts as specified in operative claims 1 and 10, let alone in combination with an activator.

- 3.6 Therefore, the main request fulfils the requirements of Art. 56 EPC.

4. Since the main request of the appellant/patent proprietor is allowable, there is no need to deal with the auxiliary requests.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the department of first instance with the order to maintain the patent on the basis of the (new) main request (claims 1-16) as submitted during the oral proceedings on 2 July 2013 and after any necessary consequential amendment of the description.

The Registrar:

The Chairwoman:



E. Goergmaier

B. ter Laan

Decision electronically authenticated