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**Datasheet for the decision
of 4 June 2020**

Case Number: T 1372/16 - 3.3.02

Application Number: 05850021.6

Publication Number: 1844120

IPC: C09D183/04, C04B41/49, C08J3/03

Language of the proceedings: EN

Title of invention:

HIGH-VISCOSITY AQUEOUS EMULSIONS OF FUNCTIONAL ALKOXYSILANES,
CONDENSED OLIGOMERS THEREOF, ORGANOPOLYSILOXANES, THEIR
PREPARATION AND USE FOR SURFACE TREATMENT OF INORGANIC
MATERIALS

Patent Proprietor:

Evonik Operations GmbH

Opponent:

Wacker Chemie AG

Relevant legal provisions:

EPC Art. 100(b)

Keyword:

Grounds for opposition - insufficiency of disclosure (no)

Decisions cited:

G 0003/14



Beschwerdekammern

Boards of Appeal

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Case Number: T 1372/16 - 3.3.02

D E C I S I O N
of Technical Board of Appeal 3.3.02
of 4 June 2020

Appellant: Evonik Operations GmbH
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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted on 5 April 2016
revoking European patent No. 1844120 pursuant to
Article 101(2) EPC**

Composition of the Board:

Chairwoman M. Blasi
Members: A. Lenzen
S. Bertrand

Summary of Facts and Submissions

- I. This decision concerns the appeal filed by the patent proprietor (appellant) against the decision of the opposition division (decision under appeal) to revoke European patent No. 1 844 120 (patent in suit).
- II. In its notice of opposition the opponent (respondent) requested the revocation of the patent in suit in its entirety based on the ground for opposition pursuant to Article 100(b) EPC.

The decision of the opposition division is based on the patent as granted. The opposition division decided that the ground for opposition under Article 100(b) EPC prejudiced the maintenance of the patent. The division took the view that, while the term "*high-viscosity*" in claims 1 and 9 did not indicate any lack of information necessary to carry out the teaching of the opposed patent, the determination of the claimed span range in claims 1 and 9 was insufficiently disclosed.

- III. The following documents, cited during the opposition proceedings, are relevant for the present decision:

- D4 International standard ISO 13320-1, first edition 1999-11-01
- D5 International standard ISO 13320, first edition 2009-12-01
- D11 Coulter[®] LS Series, internet page <http://www.cyto.purdue.edu/cdroms/cyto2/6/coulter/ss000096.htm> retrieved on 11 December 2012

D12 Experimental data from the respondent (10 pages)

IV. With its statement of grounds of appeal, the appellant filed documents D13 to D17. Of these, the following are relevant to the present decision:

D13 Excerpt from the instruction manual for particle size analysers of the Coulter[®] LS series (dated May 1994)

D14 Tabular listing of refractive indices of different silanes

V. With its letter dated 17 March 2017, the appellant filed document D18.

VI. On 28 January 2020 the board issued a communication pursuant to Article 15(1) RPBA 2020 in preparation for the oral proceedings, which had been scheduled upon the parties' requests.

VII. With its letter dated 17 March 2020, the respondent filed:

D19 Experimental data entitled "Messungen zum Brechungsindex"

VIII. On 4 June 2020 oral proceedings before the board took place, at the end of which the board gave its decision. The admission of documents D15 to D18 into the proceedings, which had been objected to by the respondent, had not become relevant for the final decision.

IX. The parties' requests relevant for this decision were as follows.

The appellant requested that the impugned decision be set aside and the patent in suit be maintained as granted, i.e. that the opposition be rejected.

The respondent requested that the appeal be dismissed.

X. The appellant's arguments, in as much as they are relevant for the present decision, can be summarised as follows.

As correctly set out in the impugned decision, the feature "*high-viscosity*" in the claims of the patent in suit was a relative feature. It possibly lacked clarity, but that was not a ground for opposition. The patent in suit provided enough information for the skilled person to assess whether or not a composition was of high-viscosity.

In contrast to the view of the opposition division, however, the patent in suit provided sufficient information for the skilled person to measure the droplet diameters D10, D50 and D90, and thus the span. The Mie theory was the correct model to calculate the droplet diameters. It required knowledge of the real part of the refractive index of the continuous phase, and the real and imaginary parts of the refractive index of the dispersed phase. The skilled person was not free to choose these parameters; they had to be selected with regard to the sample to be measured. D13, a manual of the particle size analysers of the Coulter[®] LS series, i.e. those to which the patent in suit referred, explained how this had to be done. The real part of the refractive index was substance-dependent.

If mixtures of substances were used, it could be determined with standard methods. The respondent's argument that the refractive index at the phase interface between continuous and dispersed phase was decisive was an unsubstantiated allegation. In any event, the data in D19 were not relevant, because they only described homogeneous compositions without any such phase interface. Only the imaginary part of the refractive index had to be estimated. Yet, D13 provided enough guidance in this respect. Selecting a value of 0.01 for the imaginary part of the refractive index of the dispersed phase was a reasonable choice for the silicone emulsions of the patent in suit. The silicone emulsion tested in D12 was not according to the claims of the patent in suit. In any case, the variations of the imaginary part of the refractive index of the dispersed phase resulted in only minor changes of the span value. When trying to put the invention into practice, the skilled person would stick to the information in the patent in suit; they would not use a measuring instrument other than one of the Coulter[®] LS series. Also, it was not necessary for the patent in suit to refer to a technical norm such as D4 or D5, or even to meet its reporting requirements. The respondent's arguments that different models of the Coulter[®] LS series gave different measuring results, and that modifications to or improvements of a certain model or series of instruments affected the measuring results, were unsubstantiated allegations. Further, even if one particular model of the Coulter[®] LS series was no longer commercially available, that would not be decisive for sufficiency of disclosure. For these reasons, the invention of claims 1 and 9 was sufficiently disclosed.

XI. The respondent's arguments, in as much as they are relevant for the present decision, can be summarised as follows.

That the emulsion was of "*high-viscosity*" and that it had a "*span*" within the range of from 3 to 8 was essential for solving the problem set out in the patent in suit, namely a greater penetration depth of the emulsion into porous building materials. However, the skilled person must be able, without undue burden, to determine whether or not an emulsion fell within the ambit of the claim, and whether or not this emulsion actually solved the problem. This was not the case here. The patent in suit did not specify the type of the rotational viscosimeter, the size and geometry of the spindle, or the rotation speed to be used for measuring viscosity, let alone the technical norm according to which the viscosity measurement had to be carried out. Although the patent in suit stated that the droplet size distribution could be determined using a Coulter[®] LS particle size analyser, the measuring method, the measuring conditions and the measuring parameters were missing, as well as a technical norm, such as D4 or D5, according to which the measurement had to be carried out. The reporting requirements of technical norms D4 and D5 were not met in the patent in suit either. The Mie theory was the correct model to calculate the droplet diameters. It required knowledge of the real part of the refractive index of the continuous phase and the real and imaginary parts of the refractive index of the dispersed phase. These parameters should have been specified in the patent in suit, not least for reasons of reproducibility. Further, these three parameters had to be estimated. As was evident from D12, however, already very small deviations had a large impact on the measuring result.

The patent in suit did not require the use of a measuring instrument of the Coulter[®] LS series. Other measuring instruments could also be used, such as a Malvern Mastersizer 2000, and they produced very different results. Different results were also obtained with different models of the Coulter[®] LS series. One model of this series, i.e. the Coulter[®] LS 230, was even no longer commercially available. Further, a certain model or series of instruments could be modified or improved over time. Such modifications or improvements affected the measuring result. The appellant had the measurements carried out externally and could not provide any further details on how they were carried out and which parameters were used. For these reasons, the invention of claims 1 and 9 was not sufficiently disclosed.

Reasons for the Decision

1. The appeal is admissible.
2. Claims 1 and 9 of the patent as granted read as follows.

Claim 1:

"A high-viscosity aqueous oil-in-water emulsion which comprises from 50 to 90% by weight, based on the total weight of the emulsion, of functional alkoxy silanes and/or condensed oligomers thereof and/or organoalkoxy siloxanes, at least one emulsifier and water and has a pH of from 5 to 9 and a mean droplet size of the disperse phase of less than 5 μ m, wherein the width of the droplet size distribution, expressed as the span

$$\frac{D90 - D10}{D50},$$

is from 3 to 8."

Claim 9:

"A process for preparing high-viscosity aqueous oil-in-water emulsions comprising functional alkoxy silanes and/or condensed oligomers thereof and/or organoalkoxy siloxanes, at least one emulsifier and water by (i) premixing the constituents and (ii) emulsifying the mixture in a high-pressure homogenizer having at least one pressure stage and a pressure of from 2 to 15 MPa, so that the mean droplet size is less than 5 μ m, wherein the width of the droplet size distribution, expressed as the span

$$\frac{D90 - D10}{D50},$$

is from 3 to 8."

3. Sufficiency of disclosure (Article 100(b) EPC)

The present appeal is concerned with whether, in view of the features "*high-viscosity*" and "*span*" in claims 1 and 9 of the patent in suit, the ground for opposition relating to sufficiency of disclosure prejudices the maintenance of the patent as granted.

- 3.1 The respondent argued that each of these two features was essential to solve the problem of the patent in suit, namely to achieve a greater penetration depth of the emulsion into a porous building material. Since each of these two essential features was insufficiently

disclosed in the patent in suit, it amounted to an undue burden for the skilled person to determine whether or not an emulsion fell within the ambit of the claims, and whether or not this emulsion actually solved the problem of the patent in suit.

The board notes in this context that the effect of achieving a greater penetration depth is not a feature of claim 1 or 9 as granted and, therefore, the question of whether or not the skilled person was able to identify emulsions which actually solved the problem defined in the patent in suit by providing this effect is not a matter of sufficiency of disclosure (see also G 1/03, OJ EPO 2004, 413, point 2.5.2 of the Reasons).

The present case is, however, concerned with the question of whether the person skilled in the art, having the patent in suit at their disposal and common general knowledge taken into account, was provided with sufficient information enabling them to obtain, without undue burden, emulsions as defined in claims 1 or 9, or to verify whether or not the emulsions obtained were according to either of these claims.

In the present case, the board agrees with the appellant in that the respondent's objections of insufficiency of disclosure based on the features "*high-viscosity*" and "*span*" in claims 1 and 9 are without merits and, thus, that the skilled person was able to carry out the claimed invention.

3.2 The feature "*high-viscosity*"

The feature "*high-viscosity*" in independent claims 1 and 9 is a relative one because a numerical viscosity range is not specified in relation to it. The relative

nature of this feature might cause some ambiguity as regards the delimitation of the claimed subject-matter from the prior art. However, the claims as granted cannot be challenged in this regard (see decision G 3/14, OJ EPO 2015, A102, order).

In Examples 1 to 5, the patent in suit provides exemplary emulsions which it considers to be of "*high-viscosity*". Preceding paragraph [0051] not only mentions the type of viscosimeter to be used (rotational viscosimeter), but also the temperature of the sample during the measurement (20 °C). It is also pointed out in the patent in suit that, in view of the very high viscosity of the samples to be tested, the speed of rotation should be very low and that particular spindles have to be selected (e.g. spindle 4, a spindle of a particular size and geometry, paragraphs [0056], [0070] and [0083]). Paragraph [0052] of the patent in suit states that "*[t]he viscosity determined in this way is above 1000 mPa.s, preferably above 5000 mPa.s*". It is true that the patent in suit does not specify the specific type of rotational viscosimeter and the specific rotation speed to be used. However, the burden of proof was with the respondent (see also Case Law of the Boards of Appeal of the European Patent Office, 9th edition 2019, III.G.5.1.2(c)), and the respondent has not shown that different rotational viscosimeters or different rotational speeds lead to different results. Further, even if that had been the case, the respondent would still have had to show, in order to prove a lack of sufficiency, that the uncertainty associated with the type of viscosimeter or rotational speed was to such an extent that the skilled person would fundamentally be prevented from obtaining a high-viscosity emulsion as required by claims 1 and 9.

In summary, the patent in suit provides the skilled person with sufficient information to obtain high-viscosity emulsions and to verify whether or not an emulsion is of high-viscosity. There is therefore no need for the patent in suit to refer, for example, to a technical norm according to which viscosity has to be measured.

3.3 The feature "*span*"

The span is a measure of the width of the droplet size distribution. It is defined in claims 1 and 9 of the patent in suit with reference to the droplet diameters D10, D50 and D90.

According to paragraphs [0017] and [0018] of the patent in suit, the droplet diameters, and thus the span, can be determined using a Coulter[®] LS particle size analyser. D13 is an excerpt from a manual of the particle size analysers of the Coulter[®] LS series, i.e. exactly those to which the patent in suit refers.

- 3.3.1 On page 14-23 of D13 the differences between the Mie theory and the Fraunhofer approximation (two different optical models commonly used to calculate droplet diameters from an actual measuring result) are explained. The Mie theory should be used when the sample contains a significant fraction of very small material. It takes into account the complex refractive index, consisting of a real and an imaginary part.

In this context, in point 1.2 of its communication pursuant to Article 15(1) RPBA 2020, the board set out what it considered to be common ground between the parties, namely

- that in the present case, the Mie theory was the optical model to be used to calculate the droplet diameters and thus the span;
- that knowledge of the real part of the refractive index of the continuous phase and the real and imaginary parts of the refractive index of the dispersed phase was necessary for that purpose.

This was not contested by any party in the subsequent proceedings.

3.3.2 The real parts of the refractive indices of the continuous and the dispersed phase

On page 14-24, first paragraph under the heading "*Determine Refractive Indices*", D13 explains in general terms how to select the values for the real part of the refractive index:

"The complex refractive index consists of a real part and an imaginary part. The real part is what is generally thought of as the refractive index; [...] The real part of the refractive indices of most samples and suspension fluids are listed in reference books such as the Merck Index or the CRC Handbook of Chemistry and Physics."

Thus, by consulting standard reference books or tables, the skilled person can easily find out which values are to be used as the real parts of the refractive indices for both the continuous and the dispersed phase. As a matter of fact, D13 already contains a list of refractive indices for common liquids in Table 14.1.

The respondent argued in this context that the compositions of the patent in suit contained considerable quantities of emulsifier, these quantities had a considerable influence on the refractive index, and that therefore the values for refractive indices tabulated in standard reference books, for example, could not be used, since they generally related to pure substances only. This was also shown in D19. The tests described there showed that the addition of different emulsifiers to water or isooctyltriethoxysilane led to a considerable change in the refractive index compared with pure water or pure isooctyltriethoxysilane.

This argument fails to convince. The skilled person is well aware of the fact that the refractive index of a mixture of substances is generally different from that of individual substances. This was acknowledged by the respondent when filing D19 (see letter dated 17 March 2020, page 1, penultimate paragraph). For this reason, if the skilled person were to use a mixture of substances rather than a pure substance, they would determine the refractive index of this mixture experimentally using standard methods and would use the value thus obtained as the real part of the refractive index of this mixture, as argued by the appellant. This is exactly what D19 shows.

In the written proceedings (see reply to the statement of grounds of appeal, page 4, paragraph 3), the respondent also put forward that the refractive index at the phase interface between the continuous and the dispersed phase was decisive, not the refractive index of the pure substances. However, the board could not find any support for this argument in the respondent's submissions. In particular, the data in D19 cannot serve as a support for this argument, because D19 only

reports on refractive indices of homogeneous mixtures, not on refractive indices at the phase interface between a continuous and a dispersed phase. The board agrees with the appellant in this respect, namely that this argument is an unsubstantiated allegation and therefore not convincing.

The respondent also argued that Example 1 of the patent in suit used octyltriethoxysilane. As was evident from D14, it had a refractive index of **1.417**. The appellant, however, had stated in its letter dated 7 January 2014, on page 3, paragraph 6 (emphasis added):

*"Im Dokument D12, Absatz 4.2, stellt die Einsprechende selbst fest, dass gemäß eines häufig benutzten Labormodells ein Partikelbrechungsindex von **1,39** und ein Imaginärteil von 0,01 verwendet wird. Somit bestätigt die Einsprechende selbst, dass der Fachmann eine klare Vorstellung hat, mit welchem Partikelbrechungsindex und Imaginärteil die durchgeführte Messung auszuwerten ist."*

According to the respondent, this statement was to be understood as meaning that both refractive indices, 1.417 and 1.39, were equally suitable. Consequently, the skilled person could choose very different values for the real part of the complex refractive index of the dispersed phase. This argument is not convincing. As submitted by the appellant, and the board agrees with this, the above statement refers only to the experimental results of D12. It does not reflect the approach that the skilled person would follow according to D13.

Hence, the board agrees with the appellant that the real parts of the refractive indices for both the

continuous and the dispersed phase do not have to be estimated. They can either be found in standard reference books or tables, or be measured using standard methods. The respondent's argument based on D12 (Table 5, column 2 vs columns 5 and 6), showing that the use of different real parts for the refractive index of the dispersed phase resulted in very different span values, is therefore not relevant.

3.3.3 The imaginary part of the refractive index of the dispersed phase

On page 14-24, under the heading "*Determine Refractive Indices*", D13 also provides guidelines to estimate the imaginary part of the refractive index:

"The complex refractive index consists of a real part and an imaginary part. [...]; the imaginary part represents the absorption coefficient of the material. [...]"

The imaginary or absorptive part of the refractive index for the sample is hard to find in any reference. Use the guidelines below to enter the imaginary part of the refractive index. This does not affect the results substantially unless the value of this parameter changes by a factor of greater than approximately 3.

Guidelines to use as an estimation of the imaginary part of the refractive index:

- *White or transparent powders - 0 to 0.1*
 - *Clear materials, glass, clear polymers - <0.001*

- *Latex, translucent materials, quartz, polymer resins, crystallization processes*
 - *<0.01*
- *Lightly colored translucent material* - *0.01 to 0.1*

- *Gray or lightly pigmented materials, metal oxides, highly-colored materials* - *0.1 to 1*

- *Black or highly pigmented materials, metallic particles, carbon black (imaginary values for metals are similar in magnitude as the refractive index)* - *1 to 10"*.

These are guidelines for the skilled person on how to select the imaginary part. That the choice of the actual value is left to the skilled person means that they can choose, according to these guidelines, either an exact value for the imaginary part, or an approximate value which is not equal to the exact value but which differs only to an extent that it would not significantly affect the measuring result. Were that not the case, it would make no sense to leave the choice of this value to the skilled person when operating a particle size analyser as described in D13. Based on the guidelines provided by D13 (see above), the skilled person would be able to choose an appropriate value for the imaginary part of the refractive index of the dispersed phase in the present case. With respect to D13, the board agrees with the appellant that, in the present case, 0.01 appears to be a reasonable choice for the imaginary part of the refractive index of the dispersed phase concerning silicone emulsions. This is also borne out by D12 (first paragraph under point 4.2), according to which an imaginary part of 0.01 is chosen for silicone

emulsions in a laboratory model frequently used by the respondent.

In this context, the respondent argued that the appellant had derived from D13 a value of 0.01 for the imaginary part of the refractive index of the dispersed phase, but that D13 disclosed a wide range to be possible and thus a considerably smaller value such as 0.001 could also be chosen. Further, according to D13, changing the imaginary part of the refractive index by a factor of greater than approximately 3 affected the measuring results substantially. Since 0.01 and 0.001 differed by a factor of 10, this had to apply even more to this pair of numbers. This was proven by the respondent's results in D12 (Table 5, an excerpt of which is reproduced below):

| | RI 1.39, lmg 0.01 | RI 1.39 lmg 0.1 | RI 1.39 lmg 0 |
|-------------------|---------------------|---------------------|---------------------|
| d10: | 0.199 μm | 0.449 μm | 0.199 μm |
| d50: | 0.762 μm | 1.179 μm | 0.754 μm |
| d90: | 5.102 μm | 7.458 μm | 5.697 μm |
| Verteilungsbreite | 6.44 | 5.94 | 7.29 |

These results were obtained from an emulsion of trimethylsiloxy-terminated polydimethylsiloxane in water using the Mie theory as the optical model ("RI" and "lmg" denoting the real and the imaginary parts of the refractive index of the dispersed phase, respectively). In columns 2 to 4, the RI was kept constant at 1.39. However, changing the lmg from 0.01 over 0.1 to 0 showed that the span value ("Verteilungsbreite") changed considerably.

This is not convincing. As argued by the appellant, and not contested by the respondent, the siloxane used in D12 is not a functional alkoxy silane and/or condensed oligomer of it and/or organoalkoxy siloxane as required

in claims 1 and 9. Without further evidence, it cannot be assumed that the results obtained with the siloxane of D12 necessarily have to apply also to the siloxanes according to claims 1 and 9.

In addition, as also submitted by the appellant, each of the span values 6.44, 5.94 and 7.29 in columns 2 to 4 lies within the range "*from 3 to 8*" in claims 1 and 9. The largest observed change in the span value, i.e. 1.35 (= 7.29 - 5.94), is still comparatively small compared to the range "*from 3 to 8*" in claims 1 and 9. This essentially means that measuring an emulsion with an actual span value ranging from 3 to 8 could result in a span value not falling within that range only if the actual span value is close to the boundaries of that range. Therefore, the tests in D12 can, at most, prove that there is a certain ambiguity at the boundaries of the claimed range. This ambiguity, however, may be associated with a lack of clarity; it is not indicative of a lack of disclosure, as it has not been shown that, as a result of this ambiguity, the skilled person would fundamentally be prevented from obtaining emulsions according to claims 1 and 9.

3.3.4 Measuring instrument for the "*span*"

As already mentioned above, for the measurement of the droplet diameter, the patent in suit (paragraph [0018]) refers to a particular series of instruments:

"The droplet diameter and the width of the distribution can be determined using a Coulter[®] LS particle size analyzer [...]"

The respondent interpreted the wording "*can be determined*" to mean that the droplet diameter could

also be determined with other particle size analysers, such as a Malvern Mastersizer 2000. As was evident from D12 (Tables 5 and 6), results obtained with a Malvern Mastersizer 2000 differed significantly from those obtained with a Coulter[®] LS 230.

This argument is not convincing. When trying to put the invention into practice, the skilled person would use the information provided in the patent in suit. Therefore, they would employ a measuring instrument of the Coulter[®] LS series but would not deliberately choose a measuring instrument that does not belong to this series.

Similar to the above, the respondent argued that the Coulter[®] LS series of instruments comprised different models, such as LS 100Q, LS 200 and LS 230 (see D11, page 1), and further that each model gave different measuring results. It also argued that a certain model or series of instruments could be modified or improved over time and that such modifications/improvements affected the measuring result. As pointed out by the appellant, these arguments have not been supported in any way. The board thus concludes that they are unsubstantiated allegations and therefore not persuasive.

The respondent further argued that the Coulter[®] LS 230 instrument was no longer commercially available. However, the patent in suit refers to a "Coulter[®] LS particle size analyzer" and thus to a whole series of instruments; as mentioned before, evidence that the instruments of Coulter[®] LS series provide different results is lacking. Accordingly, the skilled person can use other models from this series for their measurements. It is therefore not decisive for the

question of sufficiency of disclosure whether a particular model of that series is no longer commercially available.

3.3.5 The respondent also argued that the appellant had the measurements in the patent in suit carried out externally and that it could not provide any further details on how they were carried out and which parameters were used. This indicated a lack of disclosure.

However, the fact that a patent proprietor, i.e. the appellant in the present case, had a measurement carried out externally cannot be an indication of insufficiency of disclosure, since the relevant question to be answered in the context of sufficiency of disclosure is whether or not the skilled person, on the basis of the information provided in the patent in suit, taken together with their common general knowledge, is able to put the invention as defined in the claims into practice. As clear from the above, this question is to be answered in the affirmative. This being the case,

- there is no reason to doubt that measurements made with a Coulter[®] LS series particle size analyser are reproducible;
- it is not necessary for the patent in suit to refer to a technical norm such as D4 or D5, or even to meet its reporting requirements.

3.4 In view of the above, the patent in suit, taken together with the common general knowledge, provides the skilled person with sufficient information to obtain the emulsions claimed and to verify whether or not an emulsion is according to the claims of the

patent in suit. Therefore, the ground for opposition pursuant to Article 100(b) EPC does not prejudice the maintenance of the patent as granted.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The opposition is rejected.

The Registrar:

The Chairwoman:



M. Schalow

M. Blasi

Decision electronically authenticated