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# Datasheet for the decision of 13 May 2014

Case Number: T 0328/11 - 3.3.10

00128268.0 Application Number:

Publication Number: 1125919

IPC: C07C69/54, C07C67/62

Language of the proceedings: ΕN

Title of invention:

Stabilized hydroxyalkyl (meth) acrylate

Patent Proprietor:

NIPPON SHOKUBAI CO., LTD.

Opponent:

Evonik Röhm GmbH

Headword:

Relevant legal provisions:

EPC Art. 123(2), 123(3), 83, 54(2), 56

Keyword:

Amendments - added subject-matter (no) Sufficiency of disclosure - (yes) Novelty (yes, after amendment) Inventive step - (yes)

Decisions cited:

T 2017/07

## Catchword:



# Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 0328/11 - 3.3.10

D E C I S I O N
of Technical Board of Appeal 3.3.10
of 13 May 2014

Appellant: Evonik Röhm GmbH (Opponent) Kirschenallee

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Representative: Godemeyer, Thomas

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Respondent: NIPPON SHOKUBAI CO., LTD.

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Osaka-shi, Osaka 541-0043 (JP)

Representative: Breuer, Markus

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 27 December 2010 rejecting the opposition filed against European patent No. 1125919 pursuant to Article

101(2) EPC.

Composition of the Board:

Chairwoman J. Mercey

Members: R. Pérez Carlón

D. Rogers

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# Summary of Facts and Submissions

- I. The appellant (opponent) lodged an appeal against the decision of the opposition division to reject the opposition against European patent No. 1 125 919.
- II. The opposition had been filed on the grounds that the invention was not disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Article 100(b) EPC), and that the subject-matter of the claims of the patent as granted was not novel and did not involve an inventive step (Article 100(a) EPC).
- III. The documents filed before the opposition division included the following:

D1: US 3,441,599

D2: US 3,875,211

D12: Roorda, Verbeek and Junginger, Journal of

Chromatography, 403 (1987) 355-357

D23: US 2,484,487

IV. The following experimental evidence was filed by the appellant before the opposition division:

D34a: Time to polymerisation of 2-hydroxyethyl methacrylate in the presence of AlBN.

D34b: Time to polymerisation of 2-hydroxyethyl methacrylate at 100°C without initiator.

D34c: Summary of results in D34a and D34b

V. Of the documents filed during the appeal proceedings, the following, all filed by the appellant, are relevant for the present decision: - 2 - T 0328/11

D36a: JP 52-23019 (English translation)

D37: Toxnet: Initial submission: Repeated inhalation toxicity of hydroxypropyl acrylate (final report) with cover letter dated 042192. Year of publication: 1992

D38: EP A 0 620 206

VI. The appellant filed the following experimental evidence and expert opinion during the appeal proceedings:

D35: Expert opinion of Prof. Vogel

D40: Analysis of compositions containing methacrylic acid

and the respondent (patent proprietor) filed:

D42: Experimental report

- VII. The opposition division considered that the invention was sufficiently disclosed for it to be carried out by a person skilled in the art, and that the subject-matter of the claims of the then pending main request, namely the patent as granted, was novel over documents D1, D2 and D12. Either of documents D1 or D2 represented the closest prior art. These documents disclosed compositions which did not comprise hydroxyalkyl saturated carboxylates, and none of the documents on file disclosed that said carboxylates could have a stabilising effect, with the consequence that the subject-matter of claims 1 and 3 of the patent in suit was inventive.
- VIII. During the oral proceedings before the board of appeal, which took place on 13 May 2014, the respondent requested that the decision under appeal be set aside and that the patent be maintained upon the basis of

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claims 1 to 3 of auxiliary request 3 filed under cover of a letter dated 14 November 2011, and withdrew all higher ranking requests. Independent claims 1 and 3 of this request read as follows:

Claim 1: "A stabilized hydroxyalkyl (meth)acrylate, which contains a hydroxyalkyl saturated-carboxylate in an amount of 0.01 to 1 wt% of hydroxyalkyl (meth)acrylate along with a phenol compound in an amount of 0.001 to 0.5 wt% of hydroxyalkyl (meth)acrylate, wherein the blending rate of the hydroxyalkyl saturated-carboxylate to the phenol compound is in a range of 0.1 to 100 times by weight, and wherein the hydroxyalkyl saturated-carboxylate is at least one member selected from the group consisting of hydroxyethyl acetate, hydroxyethyl propionate, hydroxyethyl isobutyrate, hydroxypropyl acetate, hydroxypropyl propionate, and hydroxypropyl isobutyrate."

Claim 3: "A method of stabilizing hydroxyalkyl (meth)acrylate, comprising a step of adding to hydroxyalkyl (meth)acrylate a hydroxyalkyl saturated-carboxylate in an amount of 0.01 to 1 wt% of hydroxyalkyl (meth)acrylate and a phenol compound in an amount of 0.001 to 0.5 wt% of hydroxyalkyl (meth)acrylate, wherein the blending rate of the hydroxyalkyl saturated-carboxylate to the phenol compound is in a range of 0.1 to 100 times by weight, and wherein the hydroxyalkyl saturated-carboxylate is at least one member selected from the group consisting of hydroxyethyl acetate, hydroxyethyl propionate, hydroxyethyl isobutyrate, hydroxypropyl acetate, hydroxypropyl propionate, and hydroxypropyl isobutyrate."

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IX. The arguments of the appellant relevant for the present decision are the following:

The range of 0.01 to 1 wt% was not disclosed in the application as originally filed in combination with the specific carboxylates required by claims 1 and 3, which contained for this reason added subject-matter.

The appellant argued that the invention was not sufficiently disclosed for it to be carried out by the skilled person because compositions comprising amounts of phenol and hydroxyalkyl saturated carboxylate at the lower end of the ranges defined in claim 1 of the granted patent, more particularly containing 10 ppm phenol and 1 ppm hydroxyalkyl saturated carboxylate, could not be prepared. Firstly, it was necessary for (meth) acrylic acid and its esters to contain an amount of phenol effective to prevent polymerisation, so that compositions comprising amounts of phenol at the lower end of the range defined in claim 1 could not be obtained as they would polymerise. Furthermore, as explained in the expert opinion of Prof. Vogel (D35), methacrylic acid always contained acetic acid which could not be removed therefrom as their boiling points were very close and, under the reaction conditions required for producing hydroxyalkyl methacrylate, acetic acid reacted to form hydroxyalkyl acetate with the consequence that it was not possible to obtain compositions containing an amount of hydroxyalkyl saturated carboxylate at the lower end of the range required by claim 1.

The subject-matter of claim 1 was not novel over the disclosure of documents D1, D2, D12, D23, D36a and D37 for the following reasons:

D1, D2 and D23 disclosed the preparation of compositions comprising hydroxyethyl (meth)acrylate stabilised by phenol compounds. Although none of these documents mentioned the presence of hydroxyalkyl saturated carboxylates, they were unavoidable impurities which must have been part of said compositions.

Document D12 disclosed a chromatograph of a hydroxyethyl methacrylate sample containing 0.006% of hydroquinone, and further containing hydroxyethyl acetate. Although D12 did not disclose the amount of the latter, comparison with other peaks of the chromatogram showed that it must necessarily fall within the range required by claim 1.

Comparative example 1 of D36a disclosed the preparation of hydroxyethyl methacrylate from methacrylic acid containing hydroquinone in the presence of chromium acetate. D36a further disclosed that under the reaction conditions chromium acetate yielded hydroxyethyl acetate, whose presence was detected. Assuming that the conversion of chromium acetate and that of methacrylic acid had been quantitative, the product necessarily contained an amount of hydroxyethyl acetate within the range required by claim 1.

D37 disclosed on page 4 a composition containing 1.00% of hydroxyethyl acetate, 0.1% of hydroxyethyl propionate and 349 ppm of methyl ether of hydroquinone. Although the total relative amount of hydroxyalkyl saturated carboxylates fell outside that required by claim 1, the amount of hydroxyethyl propionate fulfilled the requirement set by claim 1.

Document D38, which was the closest prior art,

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disclosed stabilised hydroxyalkyl (meth)acrylate compositions (example 6, run 47) comprising pmethoxyphenol and 4H-TEMPO. The data in the patent in suit and in D42 could not prove any effect beyond the retardation of the polymerisation due to dilution, whereas that in D34b and D34c proved that an increase in the amount of hydroxyalkyl saturated carboxylate diminished the stability of the mixture; the hydroxyalkyl saturated carboxylate did not, thus, contribute to the stabilising effect. The problem underlying the claimed invention was, therefore, the mere provision of alternative, stabilised compositions. The solution, namely compositions containing a known impurity and stabilised by a phenol, was obvious for the person skilled in the art with the consequence that the subject-matter of claims 1 and 3 was not inventive.

X. The arguments of the respondent relevant for the present decision are the following:

Claim 1 found a basis in the combination of claims 1 and 2 and the passages on page 3, last line, page 4, line 7, and page 4, line 15 of the application as originally filed.

The argument of the appellant that the compositions of claim 1 containing hydroxyalkyl saturated carboxylate and phenol in amounts at the lower end of the required ranges could not be obtained had not been substantiated by experimental evidence, such that the objection that the invention was not sufficiently disclosed must, thus, be rejected.

Documents D1, D2 and D23 did not mention the source of acrylic or methacrylic acid used as starting material. Even assuming that these acids could, under some

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circumstances, contain acetic acid, novelty could only be denied provided that acetic acid was inevitably present, and the appellant had not proven that this was the case.

Figure 1 of document D12 disclosed a chromatograph of a hydroxyethyl methacrylate sample. The values mentioned in the third paragraph of page 356 referred, however, to a different sample, from which methacrylic acid had been removed. This was consistent with the fact that the relative amount of the compounds corresponding to peaks 3 and 5 was reversed with respect to that in figure 1. The amount of hydroxyethyl acetate in the sample of figure 1 could thus not be determined in the manner argued by the appellant.

The appellant estimated the relative amount of hydroxyethyl acetate and hydroquinone in comparative example 1 of document D36 upon the basis of various assumptions which had not been substantiated and, therefore, the presence of hydroxyethyl acetate in an amount as required by claim 1 had not been proven beyond any reasonable doubt.

Lastly, even if D37 was considered as part of the state of the art, which was challenged, it was apparent that the amount of hydroxyalkyl saturated carboxylate in D37 was higher than that required by claim 1.

The subject-matter of claim 1 was, therefore, novel over the prior art cited against it.

Turning to inventive step, document D38 was the closest prior art. The claimed compositions which contained at least one of hydroxyethyl acetate, hydroxyethyl propionate, hydroxyethyl isobutyrate, hydroxypropyl

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acetate, hydroxypropyl propionate and hydroxypropyl isobutyrate in an amount of 0.01 to 1 wt% of hydroxyalkyl (meth)acrylate and a blending rate of 0.1 to 100 times with respect to the phenol compound, and the claimed methods of stabilising with said compositions were inventive, since the state of the art was silent about any stabilisation effect of hydroxyalkyl saturated carboxylates, and the effect obtained went beyond that of a mere dilution effect. For these reasons, the subject-matter of claims 1 and 3 was inventive.

- XI. The final requests of the parties were the following:
  - The appellant requested that the decision under appeal be set aside and that the European patent No. 1 125 919 be revoked.
  - The respondent requested that the decision under appeal be set aside and that the patent be maintained upon the basis of the claims of auxiliary request 3, filed under cover of a letter dated 14 November 2011.
- XII. At the end of the oral proceedings, the decision was announced.

## Reasons for the Decision

1. The appeal is admissible.

#### Amendments:

2. Independent claims 1 and 3 differ from claim 1 as granted in that the amount of hydroxyalkyl saturated- 9 - T 0328/11

carboxylate, which was of "0.0001 to 2 wt%" in the granted version, is restricted to "0.01 to 1 wt%".

The amendment does not extend the protection conferred by the patent as granted and fulfils, hence, the requirements of Article 123(3) EPC.

3. The features of claim 1 result from the combination of that of claims 1 and 2 and the passages on page 3, last line, page 4, line 7, and page 4, line 15 of the application as originally filed.

Dependent claim 2 finds a basis in claim 4 as originally filed.

Claim 3 finds a basis in the same passages cited with respect to claim 1 together with page 3, line 5 of the application as originally filed.

The requirements of Article 123(2) EPC are, hence, fulfilled.

3.1 The appellant argued that the passage on page 4, line 7 of the application as originally filed did not disclose, in combination, the amount of hydroxyalkyl saturated carboxylate and the specific carboxylates required by claims 1 and 3. However, the board holds that the amount on page 4, line 7 applies to all embodiments of the invention such that this amount may be combined with the specific carboxylates of claim 2 as originally filed and, hence, does not represent added subject-matter. This argument is, thus, dismissed.

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# Sufficiency of disclosure:

4. The appellant argued that compositions comprising either hydroxyalkyl saturated carboxylate or phenol in amounts at the lower end of the ranges defined in claims 1 and 3 could not be obtained and for this reason the invention was not sufficiently disclosed for it to be carried out by a person skilled in the art.

This was because hydroxyalkyl (meth)acrylate was very easily polymerised and stabilisers in an amount sufficient to prevent polymerisation were generally added in its manufacturing process, storage and transportation (see paragraph [0002] of the patent in suit).

The lower amounts claimed were not effective stabilising amounts, such that compositions comprising such low amounts could not be obtained as the hydroxyalkyl (meth) acrylate would polymerise.

In addition, the expert opinion, D35, indicated that hydroxyalkyl acetate was inevitably formed in the synthesis of hydroxyalkyl (meth)acrylate, since said synthesis involved the reaction of (meth)acrylic acid with alkylene oxide, (meth)acrylic acid always containing traces of acetic acid, when this was prepared by an oxidative process, and in fact technical methacrylic acid marketed before 1999 contained acetic acid in an amount greater than 0.01 wt%. This acetic acid then also reacted with the alkylene oxide to give the hydroxyalkyl acetate.

4.1 However, the second, third and fifth samples of methacrylic acid in document D40, submitted by the appellant itself, contain an amount of acetic acid

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below 0.01 wt%. Reaction of such a composition with alkylene glycol would thus result in a composition comprising hydroxyalkyl methacrylate containing less than 0.01 wt% hydroxyalkyl acetate, and D42 shows that hydroxyethyl methacrylate containing 0 ppm hydroxyethyl acetate can be obtained by distillation. Thus, there is no reason to doubt that hydroxyalkyl (meth)acrylate with a content of hydroxyalkyl acetate at the lowest limit of the range required by claim 1, namely 0.01 wt%, can be obtained.

4.2 The appellant further argued that hydroxyalkyl (meth)acrylate compositions always required an amount of phenol sufficient to stabilise them against polymerisation, and for this reason compositions comprising phenol in an amount at the lower end of the range defined by claim 1, which clearly lay below an effective stabilising amount, could not be obtained as the hydroxyalkyl (meth)acrylate would polymerise.

However, document D38 discloses compositions comprising various hydroxyalkyl (meth)acrylates and only 6.5 ppm of p-methoxyphenol (entry 51), which lies below the lower limit of 10 ppm (0.001 wt%) required by claim 1. The board thus sees no reason why compositions comprising phenol in an amount at the lower end of the range defined by claim 1 could not be obtained.

In any case, since the claim does not exclude the presence of compounds other than those specifically defined, the compositions may contain another stabilising agent in any quantity.

The invention is, thus, sufficiently disclosed for it to be carried out by a person skilled in the art.

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## Novelty:

- 5. Claim 1 relates to a composition comprising:
  - a hydroxyalkyl (meth)acrylate,
  - 0.01 1 wt% of a hydroxyalkyl saturatedcarboxylate selected from:

hydroxyethyl acetate,
hydroxyethyl propionate,
hydroxyethyl isobutyrate,
hydroxypropyl acetate,
hydroxypropyl propionate,
hydroxypropyl isobutyrate, and

- 0.001 to 0.5 wt% of a phenol,

wherein the blending rate of the hydroxyalkyl saturated carboxylate and phenol is of 0.1 to 100 times by weight, and

wherein the percentages refer to the amount of hydroxyalkyl (meth)acrylate.

- 6. The appellant challenged the novelty of the subject-matter of claim 1 over the disclosures of the prior art documents D1, D2, D12, D23, D36a and D37.
- 7. Documents D1, D2 and D23:
- 7.1 Example X of document D1 discloses the preparation of hydroxyethyl acrylate from acrylic acid stabilized by hydroquinone. Example 1 of document D2 discloses the preparation of hydroxyethyl methacrylate from methacrylic acid stabilized by hydroquinone monomethyl ether. Lastly, the examples of document D23 describe

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the preparation of hydroxyethyl acrylate and hydroxyalkyl methacrylate by esterifying the corresponding acids, which contain hydroquinone as stabiliser, with ethylene oxide.

The appellant acknowledged that these documents failed to explicitly disclose the presence of any of the hydroxyalkyl saturated carboxylates required by claims 1 and 3. However, it argued that these compounds were unavoidable impurities which were necessarily obtained together with hydroxyalkyl (meth)acrylate.

The appellant again relied on the expert opinion D35 for supporting this argument. D35 states that methacrylic acid obtained by oxidative processes, and acrylic acid obtained by partial oxidation of propene, always contained traces of acetic acid which could not be distillatively removed due to their close boiling points. Acetic acid reacted with ethylene oxide under the conditions for preparing hydroxyethyl methacrylate to give hydroxyalkyl acetate, with the consequence that the compositions obtained by the processes of D1, D2 and D23 necessarily contained hydroxyethyl acetate in the amounts required by claim 1.

However, document D35 (see point 4) merely discloses that (meth)acrylic acid, when obtained by specific oxidation methods, contained acetic acid. Documents D1, D2 and D23 are silent about the method used for preparing (meth)acrylic acid, and there are other, non-oxidative, methods for obtaining said acids other than those referred to in D35, such as the Reppe, ethenone or cyanohydrin processes, as argued by the respondent.

Furthermore, document D40 (see point 4.1 above) analyses various samples of commercially available

methacrylic acid. Three of them contain acetic acid in an amount below 0.01 wt% which would lead to hydroxyalkyl methacrylate containing hydroxyalkyl acetate also in an amount below 0.01 wt%. Thus, methacrylic acid does not necessarily contain an amount of acetic acid which would lead to hydroxyalkyl methacrylate containing hydroxyalkyl acetate in the amount required by claim 1.

For these reasons, it cannot be concluded that the compositions of documents D1, D2 and D23 referred to above inevitably contain hydroxyethyl acetate in the amount required by claim 1, with the consequence that the subject-matter of claim 1 is novel over these documents.

#### 8. Document D12:

Figure 1 of document D12 discloses a chromatogram of hydroxyethyl methacrylate (HEMA, peak 4) which contains hydroxyethyl acetate (HEA, peak 2). D12 further discloses the amount of hydroquinone in the HEMA sample as stated by the manufacturer to be 0.006%. Document D12 discloses, therefore, a composition comprising the three components required by claim 1. It remains to be examined whether said components are present in the required percentages.

8.1 The appellant argued that according to the third paragraph on page 356, the amount of dimethylacryl ethane (DME), which corresponded to peak 5 of the chromatogram of figure 1, was 0.2%. Although D12 did not quantify peak 2, in the light of the similar height of peaks 2 and 5 the amount of HEA must be close to 0.2% and, hence, fall within the range of hydroxyalkyl saturated carboxylate required by claim 1.

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However, the third paragraph on page 356 seems to refer to a different sample than that leading to the chromatogram in figure 1, namely to one which is "a typical batch of HEMA, which prior to polymerisation was carefully rinsed from methacrylic acid by filtration over a basic aluminiumoxide column...". It is apparent from this sentence that, before the analysis, a filtration step had been carried out. Thus, although peak 3 in figure 1 corresponding to methacrylic acid (MAA) is clearly higher than peak 5 (DME), in the paragraph relating to the sample filtered over alumina, DME is described as being present in an amount 40 times greater (0.2%) than MAA (5. 10-3%).

For these reasons the board concludes that the chromatogram shown in figure 1 is derived from another sample than that for which quantitative results are given in the third paragraph on page 356, such that the relative amounts given in said paragraph cannot be used to estimate the amount of hydroxyethyl acetate in figure 1.

This argument of the appellant is, hence, unconvincing. The board concludes that document D12 fails to disclose all the features of claim 1 of the main request.

### 9. Document D36a:

Comparative example 1 of document D36a discloses the preparation of hydroxyethyl methacrylate from methacrylic acid containing hydroquinone over chromium acetate as catalyst. Document D36a indicates that the presence of hydroxyethyl acetate in the product was confirmed by gas chromatography, but is silent as to the amount obtained.

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The appellant estimated the amount of hydroxyethyl acetate in the reaction mixture assuming that the conversion of chromium acetate to hydroxyethyl acetate was quantitative, that that of methacrylic acid to hydroxyethyl methacrylate was also quantitative, and that no hydroquinone reacted during the process.

However, as argued by the appellant itself, using D35 and D40 as support (see point 7.1 above), methacrylic acid can contain additional acetic acid which, if present, would contribute to the amount of hydroxyalkyl acetate in the final product. The conversion of chromium acetate and of methacrylic acid is not necessarily quantitative: comparative example 1 also mentions another by-product derived from methacrylic acid (ethylene glycol dimethacrylate) and unreacted methacrylic acid. For these reasons, there are doubts that the relative amount of hydroxyethyl acetate inevitably falls within the range required by claim 1.

In addition, the amount of hydroquinone is specified only at the beginning of the reaction. Since it may be consumed by, for example oxygen radicals or heavy metal ions during the reaction, its amount at the end of the reaction is unknown. Hence, not only is the amount of hydroxyethyl acetate not known, but its ratio to the amount of phenol compound is also unknown, such that it cannot be derived without all reasonable doubt from said example that the blending rate of the hydroxyethyl acetate to the hydroquinone is in the range of 0.1 to 100 times by weight as required by claim 1.

Thus the board concludes that there are considerable doubts that comparative example 1 inevitably results in a composition falling within claim 1, the argument of

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the appellant being based on assumptions which lack experimental evidence. The board thus concludes that document D36a does not directly and unambiguously disclose a composition comprising the components in the amounts required by claim 1.

#### 10. Document D37:

- 10.1 The composition on page 4 of document D37 contains 97.09% of hydroxypropyl acrylate, 1.1% of hydroxyalkyl saturated carboxylate, of which 1.00% is hydroxypropyl acetate and the remaining 0.1% is hydroxypropyl propionate, and 349 ppm of methyl ether of hydroquinone. The relative amount of hydroxyalkyl saturated carboxylate with respect to hydroxypropyl acrylate is 1.13 wt% and, hence, higher than that required by claim 1, with the consequence that the subject-matter of claim 1 is novel over D37.
- 10.2 The appellant argued that the relative amount of hydroxypropyl propionate (ca. 0.1 wt% with respect to hydroxypropyl acrylate) fell within the range required by claim 1. Claim 1 required that the amount of only one of the carboxylates fell within the range defined therein, independently of the amount of any other hydroxyalkyl saturated carboxylate which could also be present and, for this reason, claim 1 was not novel.

A composition which is specified in a claim to comprise a component in an amount defined by a numerical range of values is characterised by the feature which requires the presence of that component within that range, as well as by the implicit proviso which excludes the presence of that component in an amount outside of that range (T 2017/07, point 2.2 of the reasons, not published in OJ EPO).

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Claim 1 requires "a hydroxyalkyl saturated-carboxylate in an amount of 0.01 to 1 wt%" and which "is at least one member selected from the group consisting of hydroxyethyl acetate, hydroxyethyl propionate, hydroxyethyl isobutyrate, hydroxypropyl acetate, hydroxypropyl propionate, and hydroxypropyl isobutyrate".

In spite of the open formulation "contains" in claim 1, the wording "at least one" indicates that the required amount refers to the combined amount of carboxylates such that the claim excludes a total amount of said carboxylates greater than 1 wt%.

The appellant's argument based on document D37 is, therefore, unconvincing.

- 10.3 The respondent challenged whether document D37 was available to the public before the filing date of the patent in suit. In the light of the outcome of the analysis of document D37 with regard to novelty, it is not considered necessary to decide on this point.
- 11. The board concludes, thus, that the composition of claim 1 and, by the same token, the method of claim 3, are novel over the cited prior art (Article 54 EPC).

#### Inventive step:

#### 12. Closest prior art:

Both parties considered document D38 to represent the closest prior art, and the board sees no reason to differ.

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D38 discloses (see run no. 47 in Table 6, corresponding to Example 6) compositions stabilised against polymerisation which contain one of 2-hydroxyethyl acrylate (entry HEA), 2-hydroxypropyl acrylate (entry HPA), 2-hydroxyethyl methacrylate (entry HEMA) or 2-hydroxypropyl methacrylate (entry HPMA) combined in each case with 13 ppm of methoquinone (p-methoxy phenol) and 1 ppm of 4H-TEMPO.

D38 discloses, thus, ternary mixtures comprising a hydroxyalkyl (meth)acrylate, a phenol and a further stabilising component, namely 4H-TEMPO.

D38 fails to disclose compositions comprising a hydroxyalkyl saturated carboxylate.

These facts were not disputed.

## 13. Technical problem underlying the invention:

The technical problem underlying the claimed invention is regarded as providing alternative stabilised hydroxyalkyl (meth) acrylate compositions comprising a phenol compound and a further component which contributes to the stabilising effect.

#### 14. Solution:

Claim 1 proposes stabilised hydroxyalkyl (meth)acrylate compositions which contain at least one of hydroxyethyl acetate, hydroxyethyl propionate, hydroxyethyl isobutyrate, hydroxypropyl acetate, hydroxypropyl propionate, and hydroxypropyl isobutyrate, in an amount of 0.01 to 1 wt% of hydroxyalkyl (meth)acrylate and at a blending rate of 0.1 to 100 times with respect to the

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phenol compound.

#### 15. Success:

15.1 The appellant argued that the problem as formulated above was not effectively solved by the compositions of claim 1. It relied on the data it had provided in D34b and D34c which showed that the time to polymerisation of hydroxyethyl methacrylate containing only 470 ppm of 2-hydroxyethyl acetate was greater than when it contained 2090 ppm of the same compound, whereas if 2-hydroxyethyl acetate would have any stabilising effect on hydroxyethyl methacrylate, the sample containing the greatest amount should have polymerised later. These results should show that 2-hydroxyalkyl acetate had no stabilising effect on the tested samples.

However, these data were obtained with different samples, possibly from different batches. The appellant has failed to show that these samples contained exactly the same components with the exception of the required phenol and saturated carboxylates, and for this reason the effect on the stability of the mixture could be due to differences in these unidentified components.

15.2 The data in the patent in suit show that hydroxyethyl acetate has a positive effect on the stability of hydroxyethyl acrylate. This effect is apparent when comparing Examples 1 and 2, which contain 0.03 wt% of hydroquinone methyl ether and, respectively, 0.1 wt% and 0.01 wt% of hydroxyethyl acetate according to which the time to initiate polymerisation at 100°C is >130 h and 100 h, respectively, with the Comparative Example in paragraph [34] in which 0.03 wt% hydroquinone methyl ether but no hydroxyethyl acetate is present, which polymerised at 100°C after only 65 hours:

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Example	hydroxyethyl	p-methoxy-	time to polymeri-
	acetate (wt%)	phenol(wt%)	sation (h)
1	0.1	0.03	> 130
2	0.01	0.03	100
comparative	_	0.03	65

15.3 The respondent further filed an experimental report (D42) showing that the presence of hydroxyethyl acetate and hydroquinone monomethyl ether in a composition led to the hydroxyethyl methacrylate being stabilised for longer than when it contained hydroxyquinone monomethyl ether alone. Indeed there was a direct correlation between the amount of hydroxyethyl acetate added and the time for initiating polymerisation of hydroxyethyl methacrylate.

The appellant challenged the data in D42 since the sample used was described as "having a purity of 99.9%" such that 0.1% of the composition of the samples used was not identified. However, all the experiments were carried out with mixtures containing the same commercially available hydroxyalkyl methacrylate, so that any effect of the unknown 0.1% must have been achieved in every run, with the consequence that the comparison put forward in D42 shows that a stabilising effect over and above that of the phenol compound alone was achieved.

15.4 For these reasons, the board considers that the respondent has provided sufficient evidence showing that the problem as formulated above has been credibly solved.

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- 16. Finally, it remains to be examined whether the claimed solution was obvious for the person skilled in the art.
- 16.1 None of the documents on file hint or disclose that hydroxyalkyl saturated carboxylates could contribute to the stabilisation of hydroxyalkyl (meth)acrylate compositions. In the absence of such information, the board concludes that the subject-matter of claim 1 and, by the same token, the method of claim 3 is inventive in the sense of Article 56 EPC.
- 16.2 The appellant alleged that any effect of the hydroxyalkyl saturated carboxylates on the stability of hydroxyalkyl (meth)acrylate was merely due to a dilution effect.

However, this is merely speculation which has not been substantiated by experimental evidence which show, for example, that the addition of small amounts of other "inert" compounds lead to an increase in the time until start of polymerisation

The board is not, therefore, persuaded by this argument.

#### Order

### For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- The case is remitted to the department of first instance with the order to maintain the patent with the following claims and a description to be adapted thereto:

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Claims 1 - 3 according to auxiliary request 3 filed under cover of a letter dated 14 November 2011.

The Registrar:

The Chairwoman:



M. Kiehl J. Mercey

Decision electronically authenticated