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Datasheet for the decision of 19 February 2015

Case Number: T 0354/12 - 3.3.03

Application Number: 96309143.4

Publication Number: 0780425

IPC: C08K3/22, C01F5/14

Language of the proceedings: ΕN

Title of invention:

Heat deterioration resistant flame retardant, resin composition and molded articles

Patent Proprietor:

KYOWA CHEMICAL INDUSTRY CO., LTD.

Opponent:

Bromine Compounds Ltd.

Relevant legal provisions:

EPC Art. 83, 54, 56

Keyword:

Sufficiency of disclosure - (yes) Novelty - (yes) Inventive step - Main and first auxiliary request (no) Inventive step - Second auxiliary request (yes)



Beschwerdekammern **Boards of Appeal** Chambres de recours

European Patent Office D-80298 MUNICH **GERMANY** Tel. +49 (0) 89 2399-0 Fax +49 (0) 89 2399-4465

Case Number: T 0354/12 - 3.3.03

DECISION of Technical Board of Appeal 3.3.03 of 19 February 2015

Appellant 1: KYOWA CHEMICAL INDUSTRY CO., LTD.

305 Yashimanishi-machi (Patent Proprietor)

Takamatsu-shi, Kagawa 761-01 (JP)

Representative: Raynor, Stuart Andrew

J A Kemp

14 South Square Gray's Inn

London WC1R 5JJ (GB)

Appellant 2: Bromine Compounds Ltd.

Makleff House (Opponent)

P.O.B. 180

Beer-Sheva 84101 (IL)

Representative: Long, Giorgio

Jacobacci & Partners S.p.A.

Via Senato 8 20121 Milano (IT)

Decision under appeal: Interlocutory decision of the Opposition

Division of the European Patent Office posted on 15 December 2011 concerning maintenance of the European Patent No. 0780425 in amended form.

Composition of the Board:

Chairman B. ter Laan D. Marquis Members:

R. Cramer

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Summary of Facts and Submissions

- I. The appeals by the patent proprietor and the opponent lie from the decision of the opposition division of 15 December 2011 maintaining European patent N° 0 780 425 (based on application number 96309143.4) in amended form.
- II. The patent was granted with a set of 11 claims of which independent claim 1 reads as follows:
 - "1. A synthetic resin composition having heat deterioration resistance and flame retardancy, comprising:
 - (a) a synthetic resin; and
 - (b) magnesium hydroxide particles in a proportion of 15 to 80% by weight based on the total weight of (a) and (b),

wherein the magnesium hydroxide particles have

- (i) an average secondary particle diameter, measured by a laser diffraction scattering method, of not more than 2 $\mu\text{m}\textsc{,}$
- (ii) a specific surface area, measured by a BET method, of 1 to $10 \text{ m}^2/\text{g}$, and containing
- (iii) a total amount of iron compound and manganese compound of not more than 0.02 % by weight in terms of metals."

Claims 2 to 11 were dependent claims of claim 1.

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

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IV. By a decision dated 15 December 2011, the opposition division maintained the patent in amended form on the basis of the auxiliary request filed during the oral proceedings. The decision was based *inter alia* on the following documents:

P1: W095/19935

P2: Kirschbaum G. and Nykyforuk R.: "Aluminum Hydroxide and Magnesium Hydroxide New Developments and their Applications in Polymer Compounding"; Regional Technical Conference "The Marketing of Polymer Modifiers and Additives ", September 23-25, 1991, pp 89-98

P3: US5286285

P5: US4098762

P7: R. Rothon, Particulate Filled Polymer Composites

page 5, para 1.2.2.5

P9: US4698379

P12: Declaration of Prof. Roger Rothon

P19: Declaration by Keiko Katsuki

In the decision it was held that the main request (claims as granted) fulfilled the requirements of Article 83 EPC and was novel over P1 and P3 but that it lacked novelty over P2. The auxiliary request however met the requirements of Articles 84, 123(2), 83 EPC and was also novel and inventive.

V. On 14 February 2012, the patent proprietor lodged an appeal against the decision of the opposition division and paid the prescribed appeal fee on the same day. The statement setting out the grounds of the appeal was filed on 17 April 2012. The patent proprietor requested that the patent be maintained on the basis of the main request (claims as granted) or any of the first or second auxiliary requests filed with the statement of

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grounds of the appeal.

Claim 1 of the first auxiliary request reads:

- "1. A synthetic resin composition having heat deterioration resistance and flame retardancy, comprising:
- (a) a synthetic resin; and
- (b) magnesium hydroxide particles in a proportion of 15 to 80% by weight based on the total weight of (a) and (b),

wherein the magnesium hydroxide particles have

- (i) an average secondary particle diameter, measured by a laser diffraction scattering method, of not more than 2 μm ,
- (ii) a specific surface area, measured by a BET method, of 1 to $10~\text{m}^2/\text{g}$, and containing wherein the total amount of iron compound, manganese compound, cobalt compound, chromium compound, copper compound, vanadium compound and nickel compound in the magnesium hydroxide particles is not more than 0.01 % by weight in terms of metals."

Claims 2 to 8 were dependent claims of claim 1.

Claim 1 of the second auxiliary request reads:

- "1. A synthetic resin composition having heat deterioration resistance and flame retardancy, comprising:
- (a) a synthetic resin; and
- (b) magnesium hydroxide particles in a proportion of 15 to 80% by weight based on the total weight of (a) and (b),

wherein the magnesium hydroxide particles have

(i) an average secondary particle diameter, measured by

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a laser diffraction scattering method, of 0.4 to 1.0 μm and

(ii) a specific surface area, measured by a BET method, of 1 to $10~\text{m}^2/\text{g}$, and containing wherein the total amount of iron compound, manganese compound, cobalt compound, chromium compound, copper compound, vanadium compound and nickel compound in the magnesium hydroxide particles is not more than 0.01 % by weight in terms of metals."

Claims 2 to 7 were dependent claims of claim 1.

- VI. On 9 February 2012, the opponent lodged an appeal against the decision and paid the prescribed appeal fee on the same day. The statement setting out the grounds of the appeal was filed on 16 April 2012. The opponent requested that the patent be revoked.
- VII. With a letter dated 29 October 2012, the patent proprietor submitted a reply to the statement of grounds of appeal of the opponent.
- VIII. By letter dated 9 November 2012, the opponent submitted a reply to the statement of grounds of appeal of the patent proprietor.
- IX. On 28 October 2014, the parties were summoned to oral proceedings which were held on 19 February 2015.
- X. The patent proprietor's arguments may be summarised as follows:

Main request

-Sufficiency of disclosure

The general process of preparation of magnesium hydroxide particles was known in the art. Paragraph 26 of the patent in suit and P5 both taught how to control the agglomeration of the particles in order to satisfy both requirements (i) and (ii) as claimed. P12 provided a way to estimate the particle size from the BET surface area and the calculated values put forward by the opponent at the oral proceedings actually showed that the preparation disclosed in P5 did lead to particles satisfying both requirements. P5 and its examples provided a teaching about the temperature that had to be applied during the heat treatment in order to obtain those particles. The objection against requirement (iii) was not understood.

- Novelty

P1 did not explicitly disclose the iron and manganese content of the magnesium hydroxide particles resulting from the preparation process of examples 1 and 2. It could also not be inferred from P1 that the metal content of the magnesium hydroxide particles was as low as that of the starting solution.

P2 only provided fragmentary information about the resin composition and the magnesium hydroxide particles disclosed therein. There were gaps of information concerning the disclosure of the properties of the magnesium hydroxide particles and the resin composition.

The claimed subject matter was therefore novel over P1 and P2.

- Inventive step

P1 was the closest prior art. The supplementary examples on pages 13 and 14 of P19 showed an improvement of the mechanical properties of the claimed resin compositions immediately after moulding and after heat deterioration. The problem to be solved was to provide resin compositions with improved tensile strength, elongation, impact strength and heat deterioration resistance. The teaching of P7 was very general and did not address the mechanical properties of the resin compositions. Also, it applied to any filler containing polymer and was not specifically relevant for magnesium hydroxide particles. Further, P7 made a distinction between active and non-active forms of some metal impurities without giving any definition of those terms, so that the skilled person would not have considered it. The claimed subject matter was therefore inventive.

First auxiliary request

P7 did not disclose the same list of metal impurities now part of claim 1, nor did P2 or P3. None of the prior art documents disclosed that a low amount of those metal impurities was necessary in order to solve the technical problem posed.

Second auxiliary request

P1 remained the closest prior art. The examples of Table II page 12 of P19 showed an improvement of the tensile strength due to the limitation of the average secondary particle diameter of the magnesium hydroxide. The technical problem solved was to provide resin compositions with improved tensile strength before and after heat deterioration. The teaching of P3 did not

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apply to the particles of P1 because they had a different BET surface area.

XI. The opponent's arguments may be summarised as follows:

Main request

-Sufficiency of disclosure

The preparation of the claimed magnesium hydroxide particles was not disclosed in the patent in suit. The paragraphs 10 and 26 of the patent referred to the Japanese document JP115799/1977 for their preparation. The US patent document P5 represented the content of JP115799/1977. The particles of examples 1, 2 and 3 of P5 had a BET surface area within the claimed range but their average secondary particle diameter was not disclosed. There was therefore no sufficient quidance in P5 for the skilled person to prepare particles with a diameter within the claimed range. Although the average secondary particle diameter of the magnesium hydroxide particles described in P5 could be calculated according to P12, as shown in the document entitled "Determination of the average secondary particle diameters for the examples of P5", that calculation showed discrepancies with the examples of P9 which could therefore not be used to fill that information gap.

Also, the patent in suit provided a range for the pressure used during the heat treatment but contained no information about the temperature of the process. P5 disclosed a temperature range of 150 to 250°C but that was not sufficient to obtain the claimed particle diameter. Indeed, P11 disclosed that the diameters of magnesium hydroxide particles varied significantly from

1.6 μm to 2.4 μm when the temperature of the heat treatment was raised from 150°C to 180°C. That dependency was not known to the skilled person.

Finally, the last sentence of paragraph 26 of the patent in suit was equivocal as it implied that requirement (iii) of claim 1 could be fulfilled even when the starting materials contained high amounts of iron and manganese. Also, the patent in suit only relied on the purity of the starting materials but did not contain any guidance concerning the iron and manganese contents of water or any other materials used in the preparation process of the magnesium hydroxide particles. Claim 1 therefore lacked sufficiency of disclosure.

- Novelty

Examples 1 and 2 of P1 disclosed a composition of an ethylene vinyl acetate resin comprising 60 weight-% magnesium hydroxide particles. The particles produced had a median particle size and a BET surface area satisfying requirements (i) and (ii) of claim 1 of the patent in suit. The magnesium hydroxide particles were obtained from a solution of starting materials containing less than 0.02% of iron and manganese compounds. The precipitation was carried out in a stainless steel (SS) vessel which is known not to leak iron nor manganese and the person skilled in the art would have understood that the remaining apparatus used in the process had to fulfil the same purity requirement because the purity issue was important in P1. The magnesium hydroxide particles therefore implicitly fulfilled requirement (iii) of the claimed subject matter.

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P2 described the use of ultrapure magnesium hydroxide particles as flame retardant for plastics. Table 1 on page 94 disclosed particle sizes and BET surface areas satisfying requirements (i) and (ii) as claimed. Table 2 showed that those particles also satisfied requirement (iii). A polypropylene composition comprising 60 weight-% of those particles was disclosed in a further passage describing flame properties of magnesium hydroxide and was also mentioned in the abstract of P2.

The subject matter of claim 1 of the main request lacked therefore novelty over P1 and P2.

- Inventive step

The closest prior art was P1. The technical problem of providing a composition comprising magnesium hydroxide particles with increased tensile strength and other technical properties was not solved, in view of the data provided. In particular, all the exemplified compositions in the patent in suit and in P19 involved magnesium hydroxide particles that had been surface treated. The technical problem effectively solved was therefore to provide alternative resins. P1 and P7 already taught how to obtain particles with a high level of purity in order to provide thermal stability. The claimed subject matter therefore lacked an inventive step.

First auxiliary request

The arguments against the inventive step of the main request also applied to the claims of the first auxiliary request since the purity requirement disclosed in P7 was not limited to specific metal

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compounds. Starting from P1 and considering P7, the skilled person would readily understand that all the metal impurities had to be reduced in order to solve the technical problem. The claims of the first auxiliary request also lacked an inventive step.

Second auxiliary request

P19 did not show any supplementary technical effect for the further limitation of the particle size of the magnesium particles. Also, P19 did not use the same resin as in P1. The teaching of P1 already encompassed particles of a diameter between 0.5 to 5 μ m so that the claimed subject matter of the second auxiliary request also lacked an inventive step.

- XII. The appellant (patent proprietor) requested that the decision under appeal be set aside and that the patent be maintained as granted (main request), or on the basis of the first or second auxiliary request filed with its statement of grounds of appeal.
- XIII. The respondent (opponent) requested that the decision under appeal be set aside and that the European patent be revoked.

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Reasons for the Decision

1. The appeal is admissible.

Main request

- 2. Sufficiency of disclosure
- Claim 1 of the main request relates to a synthetic resin composition having heat deterioration resistance and flame retardancy, comprising magnesium hydroxide particles that have to satisfy the three requirements: (i) an average secondary particle diameter, measured by a laser diffraction scattering method, of not more than 2 μm ,
 - (ii) a specific surface area, measured by a BET method, of 1 to 10 m^2/g , and
 - (iii) containing a total amount of iron compound and manganese compound of not more than 0.02 % by weight in terms of metals.
- 2.1.1 Paragraph 26 of the patent in suit indicates that the method for producing the magnesium hydroxide particles is not particularly limited in so far as they satisfy those requirements. That passage also discloses that magnesium hydroxide particles satisfying the requirements for (i) average secondary particle diameter and (ii) specific surface area can be produced by employing a method and conditions described in Japanese Document JP115799/1977, the content of which was seen by the parties as being represented by the US document P5.

2.1.2 The magnesium hydroxide particles of P5 are prepared by a process disclosed in column 3, line 43 to column 4, line 28 as well as in the examples. According to that process, a basic magnesium chloride or basic magnesium nitrate of formula $Mg(OH)_{2-x}A_x.mH_2O$ wherein A is Cl or NO_3 , x is a number of more than 0 and less than 0.2 and m is a number of 0 to 6 is provided. The magnesium chloride or magnesium nitrate is reacted with an alkaline substance in an aqueous medium. Then, magnesium hydroxide particles are produced by heating that basic magnesium chloride or basic magnesium nitrate in an aqueous medium at an elevated pressure. That heat-treatment can be performed at a temperature of about 150°C to about 250°C. Particles obtained by this process are characterized by a strain in the $\langle 101 \rangle$ direction of not more than 3.0 10^{-3} , a crystallite size in the same direction of more than 800 Å, and a specific surface area, determined by the BET method, of less than $20 \text{ m}^2/\text{g}$ (column 1, lines 18 to 28). The magnesium hydroxide particles described in examples 1, 2, 3 (all made from magnesium chloride) and example 5 (made from magnesium nitrate) of P5 all have a BET surface area of less than 10 m^2/q , as required in claim 1 of the main request.

The average secondary particle diameters of the magnesium hydroxide particles of P5 is however not disclosed in that document. Instead, P5 discloses that the particles have a markedly small strain in the <101> direction, a large crystallite size in the same direction, and a very low specific surface area determined by the BET method, and that as a result of those structural characteristics, the surface polarity of the crystallites is extremely small or nearly zero, and secondary aggregation of the crystallites does not appreciably occur (column 4, lines 29 to 38), the

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latter being indicative of a small average secondary particle diameter as indicated in paragraph 17 of the patent in suit.

2.1.3 According to page 5 of P12, it is known that the adsorption surface of a material consisting of spherical particles of the uniform diameter relates to the diameter of the particles as follows:

$$S = 6/(\rho.D) \tag{1}$$

where S is the total gas-adsorbing surface (m²/g), D is the diameter of the particles (μm), and p is the density of the material (g/cm³). For real particles of magnesium hydroxide (ρ = 2.36 g/cm³), the above relation provides a formula:

$$D_{BET} = 2.5 / S_{BET}$$
 (2)

where D_{BET} is BET-equivalent spherical particle diameter, approximately equal to the average primary particle diameter, and S_{BET} is the specific surface area measured by BET. Using formula (2) on the specific surface area measured by BET of the magnesium hydroxide particles of the examples 2, 3 and 5 of P5, the opponent was able to calculate their average primary particle diameters. Then, taking into account the average degree of agglomeration calculated from the data available in examples 1 and 4, the opponent could come to the conclusion that the average secondary particle diameters of the particles of the examples 1, 2, 3 and 5 of P5 was comprised between 0.61 and 1.41 μm .

Therefore, even if P5 does not explicitly disclose the values of the secondary average particle diameters of

the magnesium hydroxide particles produced therein, that parameter can apparently easily be derived from the values of the specific surface area measured by the BET method available in P5. It can also be seen from the calculations provided by the opponent himself that both the secondary average particle size and the BET surface area of the magnesium hydroxide particles according to examples 1, 2, 3 and 5 of P5 satisfy both requirements (i) and (ii) as set out in claim 1 of the patent in suit. These examples of P5 therefore provide a guidance to the skilled person to prepare the above mentioned magnesium hydroxide particles.

2.1.4 The opponent referred to P9, in which comparative example represents a repetition of example 1 of P5, and pointed to differences between the secondary average particle size and the BET surface area of the magnesium hydroxide particles calculated from example 1 of P5 and its repetition in comparative example 2 of P9.

However, the results obtained in comparative example 2 of P9 in fact confirm the results obtained in P5. The variation observed is likely to arise from variations in the exact compositions of starting materials used in experiments lying almost 10 years apart. The results obtained in P12 show nonetheless that the average secondary particle diameters of the particles of those examples of P5 with a BET surface area within the claimed range are all between 0.61 µm and 1.41 µm, well within the claimed range of less than 2 µm. This shows that magnesium hydroxide particles satisfying requirements (i) and (ii) can be obtained by following the process disclosed in examples 1, 2, 3 and 5 of P5.

2.1.5 The opponent also criticised that the patent in suit gave no details about the temperature applied during

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the preparation process, which, according to P11, influenced the particle diameter without the skilled person being aware of it.

P5 discloses ranges of pressure (from 5 to 30 kg/cm²) and temperatures of about 150° to about 250°C. Several conditions of temperatures are specifically disclosed in P5: 180°C for 8 hours (example 1), 170°C for 8 hours (example 2), 200°C for 4 hours (example 3) or 170°C for 4 hours (example 5). From the above mentioned examples it can be seen that the temperature of the heat treatment is generally comprised in a range between 170 and 200°C. P5 does not suggest that the temperature of the heat treatment is in any way critical to the diameter of the magnesium hydroxide particles as long as it is sufficient to perform the expected hydrothermal recrystallisation of magnesium hydroxide. That is reflected in the wording of P5 "The heat-treatment at an elevated pressure can be performed at a temperature of, say, about 150° to about 250°C." (column 4, lines 26 to 28) and also corresponds to hydrothermal recrystallisation of magnesium hydroxide known in the art, for example that of P1, page 6, line 30 to page 7, line 7.

P11 contains a rework of example 1 of P1 which discloses the preparation of magnesium hydroxide particles from a magnesium nitrate solution. The slurry obtained from the precipitation of that solution was hydrothermally treated in an autoclave at $180\,^{\circ}\text{C}$ for 4 hours. The filtered magnesium hydroxide was reslurried in water and heated. A solution of stearic acid and NH₄OH was then added and the resulting coated material was filtered, washed and dried. The table on page 5 of P11 reveals that the coated magnesium hydroxide particles obtained had an

average secondary particle diameter of 2.4 µm and a BET surface area of 2.8 m^2/g . P11 mentions that in another experiment in which the hydrothermal treatment temperature was set at 150°C for 4 hours, the coated magnesium hydroxide particles obtained had an average secondary particle diameter of 1.6 µm and a BET surface area of $4.7 \text{ m}^2/\text{q}$. Pl1 however does not describe the exact process conditions of the second experiment so that it cannot be unequivocally concluded from that passage that the different particle properties obtained solely resulted from the change in temperature from 180°C to 150°C of the heat treatment. This is also reflected in the same passage of P11 which remains elusive as to the cause for the difference in particle size diameters obtained "This is probably because the temperature of the hydrothermal treatment of Experiment 1 (180° C x 4 hours) is higher than that of the present invention." On the basis of that passage of P11 alone, the Board is of the opinion that the opponent did not convincingly show that the temperature during heat treatment was critical to the average secondary particle diameter of the magnesium hydroxide particles.

In view of the foregoing, the Board is of the opinion that the opponent did not convincingly show that the temperature during heat treatment was critical to the average secondary particle diameter of the magnesium hydroxide particles.

2.1.6 It can be concluded from the above that P5 discloses a general method for the preparation of magnesium hydroxide particles and that P5 contains some guidance as to the method and also the temperature during the heat treatment leading to magnesium hydroxide particles satisfying both requirements (i) and (ii) as set out in

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claim 1 of the patent in suit.

2.2 As for requirement (iii), the last sentence of paragraph 26 of the patent indicates that "in the above production, by selecting starting materials containing no impurities or trace amounts of impurities, especially an iron compound and a manganese compound (and/or other metal compounds described above), magnesium hydroxide particles satisfying the above requirement (iii) can be obtained." That passage unambiguously refers to the metal impurities described in paragraph 23, from which it is clear that the total amount in terms of metals of iron and manganese, as the impurities contained in the magnesium hydroxide particles, corresponds to the requirement (iii). From that passage, it can be understood that it is further desirable that the total amount of the metals of heavy metal compounds including a cobalt compound, chromium compound, copper compound, vanadium compound and nickel compound satisfies the same requirement. The interpretation of the last sentence of paragraph 26 according to which the starting materials could be selected so as to contain no or only trace amounts of impurities of other metal compounds other than iron and manganese and nevertheless should satisfy requirement (iii) does not make sense in the context of paragraph 23 and would not have been read as such by the skilled person. Even isolated from that context, the opponent did not show or even argue why the person skilled in the art could not satisfy the requirement (iii) by purification of the magnesium hydroxide particles after their preparation. Finally, paragraph 26 provides quidance concerning the amount of iron and manganese impurities that might be contained in the starting materials. That passage cannot be interpreted as meaning that only the selection of starting materials

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will lead to magnesium particles satisfying requirement (iii). It was not disputed that the skilled person could, on the basis of the common general knowledge of that field, select the other components used during the preparation of the magnesium hydroxide particles so as to fulfil requirement (iii). The Board is therefore satisfied that the patent in suit provides sufficient guidance to prepare magnesium hydroxide particles according to claim 1 of the main request.

- 2.3 Therefore, the subject matter of claim 1 of the main request is sufficiently disclosed in the patent in suit (Article 83 EPC).
- 3. Novelty
- 3.1 Examples 1 and 2 of P1 disclose two ethylene vinyl acetate (EVA) resin compositions at a loading of 60 weight-% of magnesium hydroxide having a median particle size measured by a laser diffraction scattering method (page 7, line 28) of 1.9 µm and 1.4 µm respectively and BET specific surface areas of 2.9 m²/g and 5.5 m²/g. The total amount of iron compound and manganese compound in terms of metal impurities is not explicitly disclosed.
- 3.1.1 According to the method of example 1, magnesium hydroxide was precipitated from a nitrate solution containing 35 g/L Mg, 0.9 g/L Ca, 1.5 mg/L Ni, less than 1 mg/L Fe and less than 1 mg/L Mn. The precipitation of that solution was carried out in a baffled "SS" vessel, after which the resulting slurry was hydrothermally treated in an autoclave, the mixture filtered and washed. Regardless of whether "SS" means that the vessel used for the precipitation step in example 1 was made of stainless steel or not, the

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process disclosed in P1 further involves several other preparation steps such as mixing, filtering, washing and transport the effect of which on the total amount of metal impurities is not mentioned and cannot be overlooked as these steps would not necessarily be performed in an environment that does not release iron and manganese compounds. As it was undisputed among the parties that any given production apparatus may leach significant amounts of metal impurities to the magnesium hydroxide solution (P22, page 7, fourth paragraph), even if the amount of metal impurities in the starting solution of example 1 was below 0.02% by weight, it cannot be concluded that the amount of metal impurities present in the magnesium hydroxide product resulting from the whole process was within the range of claim 1 of the patent in suit. Even if the magnesium hydroxide product formed in the process of P1 may be subjected to a purification step (page 7, lines 8 and 9), that is only an indication that the purity of the product might be a concern but it does not constitute a direct and unambiguous disclosure of a total amount of iron compound and manganese compound of not more than 0.02 %.

This reasoning is also valid for the composition of example 2 of P1, which was conducted in essentially the same way as that of example 1.

As a result, P1 does not unambiguously disclose magnesium hydroxide with a total amount of iron and manganese compound within the range of claim 1 of the main request. The subject matter of claim 1 of the main request is therefore novel over P1.

3.2 P2 is a technical paper concerning a conference. P2 contains a section about magnesium hydroxides on pages

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94 and 95 in which some characteristics of ultrapure magnesium hydroxide particles are disclosed. Typical particle sizes (0.95 to 1.15 μm for type H7 and 0.95 to $1.05 \ \mu m$ for type H10) and the BET surface areas (6 to 8 m^2/q) for those two types of magnesium hydroxide particles are disclosed in Table 1. A chemical analysis of those ultrapure particles is made available in Table 2. P2 suggests on page 94 that those ultrapure magnesium hydroxide particles may be used as flame retardants in thermoplastic resins but that section does not explicitly disclose the amount of particles in the composition. Another section of P2 on pages 96 to 98 refers to a polymer composition containing magnesium hydroxide in polypropylene in an amount of 60%, but the sole magnesium hydroxide particles identified in that section are derived from sea water and have a median particle size of 5 μm (Table 4, reference 1), therefore suggesting that those magnesium hydroxide particles are not the same as the smaller ultrapure particles disclosed in the previous section. Other magnesium hydroxides containing polypropylene and polyamide resin compositions are disclosed in Tables 4 and 5 on pages 97 to 98 but the magnesium hydroxides used (VPF 8812, VPF 8814 and VPF 8927) are not defined nor are their properties disclosed so that it cannot be concluded that those compositions are according to claim 1 of the main request. It can only be those unidentified compositions that are mentioned in the abstract since they are the only compositions of magnesium hydroxide products in an amount of 60% in polypropylene and polyamide that are clearly and unambiguously disclosed within P2. It can be concluded that P2 does not clearly and unambiguously disclose a resin composition according to claim 1 of the main request.

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- 3.3 Claim 1 of the main request is therefore novel. As a result, the dependent claims 2 to 11 are also novel. The main request satisfies the requirements of Article 54 EPC.
- 4. Inventive step
- 4.1 The closest prior art
- 4.1.1 The patent in suit relates to a synthetic resin composition composed of magnesium hydroxide particles which rarely deteriorates by heat during the thermal molding and has excellent heat deterioration resistance and flame retardancy (paragraph 1).
- 4.1.2 P1 relates to a process for preparing flame retardant compositions comprising magnesium hydroxide (page 1, lines 1 to 5). By common consent between the parties, the closest prior art was P1 which also represented the closest prior art in the contested decision. The Board sees no reason to take a different view.

The technical problem

- 4.1.3 The patent in suit as well as examples 1 and 2 of P1 disclose several thermoplastic resin compositions containing magnesium hydroxide particles characterized by an average secondary particle diameter of not more than 2 μ m and a specific BET surface area of 1 to 10 m²/g. Both documents also disclose the preparation of moulded samples therefrom and report the mechanical properties thereof.
- 4.1.4 The mechanical properties reported in examples 1 and 2 of the moulded compositions of P1 cannot be directly compared to those reported in the patent in suit as the

compositions of P1 differ from those of the patent in suit by their type of resin (ethylene vinyl acetate in P1 and propylene in the patent in suit). However, the declaration P19 provides tables (pages 13 and 14) which summarize the characteristics and the physical properties of polypropylene compositions containing magnesium hydroxide particles, the average secondary particle diameters and the BET surface area of which are within the ranges of claim 1 of the main request. The magnesium hydroxide particles of those compositions were all obtained by the same preparation process and only differ in their amounts of iron and manganese; those amounts are within the range of claim 1 (below 0.02%) in the case of examples 1, 8, 9, 10 and 11 and outside that range (0.0307% and 0.0877%) in the case of comparative examples 9 and 10. The properties of compositions moulded therefrom are reported in the table of page 14. Even if the protocol for the preparation of the resin compositions on page 6 of P19 might lead one to believe that the magnesium hydroxide particles they contained were subjected to an additional surface treatment, in fact all the particles disclosed on pages 13 and 14 were treated in the same manner so that the differences reported for the properties of the resin compositions can only be attributed to the differences in the amounts of iron and manganese of the particles. The examples on pages 13 and 14 of P19 therefore allow a fair comparison with those compositions of the closest prior art P1.

4.1.5 The tensile strength reported on page 14 (between 1.82 and 1.90 Kgf/mm², vs. 1.79 Kgf/mm²), elongation ratio (between 21 and 35% vs. 12 and 15%) and Izod impact strength (between 12.3 and 15.4 Kgf.cm/cm, vs. 9.5 and 10.5 Kgf.cm/cm) of the compositions of P19 immediately after moulding are all higher for the examples

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representing the subject matter of claim 1 of the main request than for the comparative compositions, showing that the mechanical properties of the resin compositions according to claim 1 of the main request are improved over those of the two comparative examples 9 and 10 of P19.

- 4.1.6 Also, the tensile strength (between 1.80 and 1.87 Kgf/mm², vs. below 0.5 Kgf/mm²), elongation ratio (between 17 and 32% vs. below 1%) and Izod impact strength (between 8.5 and 13.5 Kgf.cm/cm, vs. below 1 Kgf.cm/cm), whitening (12 to 23 days vs. 6 and 9 days) and 10 wt% weight loss (320 to 600 hours vs. 190 and 260 hours) of the moulded compositions reported on page 14 subjected to the heat deterioration acceleration test are all higher for the examples representing the subject matter of claim 1 than the comparative compositions, showing that the mechanical properties after heat deterioration of the resin compositions according to claim 1 of the main request are also improved over those of the two comparative examples 9 and 10 of P19.
- 4.1.7 The problem can therefore be seen as to provide flame retardant synthetic resin compositions having improved mechanical properties before and after heat deterioration and improved resistance to heat deterioration.

4.2 The solution

The solution to that problem is the composition according to claim 1 of the main request and specifically compositions in which the magnesium hydroxide particles have an iron and manganese content of below 0.02% by weight.

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4.3 Obviousness

- 4.3.1 P1, the closest prior art, mentions the desire to improve the mechanical properties of magnesium hydroxide containing moulded resins (page 2, lines 2 to 10). It emphasises the negative impact of the presence of transition metal impurities on the production process of the magnesium hydroxide particles and teaches reduction of the amount of iron and manganese in the starting solution down to 1 ppm (page 5, lines 16 to 21). P1 also suggests a purification step to remove trace impurities from the magnesium hydroxide particles so produced (page 5, lines 21 to 27 and page 7, lines 8 and 9). However, P1 does not actually disclose the amount of impurities in the magnesium hydroxide particles used in the composition.
- 4.3.2 P7 is an excerpt of a book on particulate filled polymer composites. It discloses in chapter 1.2.2.5 on page 5 that the purity of a filler is of importance for the properties of the filled resin. According to P7, impurities are trace elements such as certain transition metals which may be absorbed on the filler. Those metals (eg. iron, manganese, copper) can seriously affect the colour and thermal stability of the products even at levels of a few parts per million. Although P7 appears to distinguish between transition metal impurities in active and non-active form, it is clear from P7 that transition metals as a whole are undesirable, as even the non active form may still have an effect on the polymer properties (second paragraph of chapter 1.2.2.5). From a practical point of view, the purification of the particles is performed as a whole by a washing step with water and does not differentiate between impurities that are in active

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form and those that are in non-active form, and would apply to the method of P1, which uses that washing step (P1, page 7, lines 9 and 11).

The teaching of P7 is therefore to remove transition metal impurities even if present in amounts of as low as a few ppm as these may affect the colour and thermal stability of polymer resins. In paragraph 24 of the patent in suit the expressions thermal stability, mentioned in P7, and heat deterioration resistance are interchangeably used to describe the same technical effect. Therefore, for the purpose improving heat deterioration resistance, the teaching of P7 would be considered by the skilled person.

As magnesium hydroxides are commonly referred to as fillers in the prior art (e.g. P3, column 1, lines 53 to 59) and because magnesium hydroxides are also known to contain undesirable transition metal impurities (e.g. P3, column 2, lines 35 to 59 and P1, page 5, lines 13 to 27), the teaching of P7 is also applicable to magnesium hydroxides.

4.3.3 Therefore, applying the teaching of P7 - i.e. removing transition metal impurities for improving the thermal stability of polymer resins - to compositions of P1, which also suggests to purify the magnesium hydroxide particles, would lead to a composition according to claim 1 of the main request, which is, for that reason, not inventive.

The additional improvement of the mechanical properties of the resin compositions immediately after moulding is obtained as a result of the purification of the magnesium hydroxide particles and is therefore a technical effect obtained as a bonus to the improvement

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of the thermal stability of the resins. The person skilled in the art, solving the problem of improving resistance to heat deterioration of resins, would inevitably also arrive at an improvement of the mechanical properties before and after heat deterioration.

4.3.4 In view of the above, the main request lacks an inventive step (Article 56 EPC).

First auxiliary request

- 5. Modifications
- 5.1 Claim 1 of the first auxiliary request corresponds to claim 5 of the patent in suit. The claims of the first auxiliary request fulfil the requirements of Article 123(3) EPC.
- 5.2 The application as originally filed provides a basis for claim 1 of the first auxiliary request in originally filed claim 6 when it refers to claims 5, 4, 3 and 1 further limited to the range of not more than 0.01% by weight found in the passage of page 5 lines 1 to 11. The claims of the first auxiliary request fulfill the requirement of Article 123(2) EPC.
- 5.3 The allowability of the modifications performed in the claims of the first auxiliary request have not been contested by the opponent nor does the board see a reason to object to these claims.
- 6. Sufficiency of disclosure and novelty
- 6.1 The opponent did not object to the sufficiency of disclosure and novelty of the claims of the first

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auxiliary request. The above reasoning on the sufficiency of disclosure and novelty apply mutatis mutandis to the first auxiliary request.

7. Inventive step

- 7.1 Claim 1 of the auxiliary request differs from that of the main request only in that the total amount of iron compound, manganese compound, cobalt compound, chromium compound, copper compound, vanadium compound and nickel compound in the magnesium hydroxide particles is not more than 0.01 % by weight in terms of metals.
- 7.2 Neither the patent in suit nor the supplementary test report P19 provide any experimental evidence of a further technical effect arising from the additional purification of the transition metals cobalt, chromium, copper, vanadium and nickel from the magnesium hydroxide particles. Also, the Table on page 14 of P19 shows that the mechanical properties and resistance to heat deterioration of the compositions of examples 1, 8, 9 and 10 are only slightly improved when the amounts in transition metal impurities are below 0.01% by weight as compared with the composition of example 11 containing a total amount of transition metal impurities of 0.0183%. As a result the technical problem that can be formulated for the subject matter of claim 1 of the first auxiliary request is the same as that formulated for the main request.
- 7.3 P1 alone describes that the metal impurities are constituted by any of the transition metals, which the person skilled in the art knows to comprise cobalt, chromium, copper, vanadium and nickel (page 5, lines 16 to 18). Purification may be achieved by the process already suggested in P1, which purification does not

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discriminate between the transition metal impurities present on the particles as it is essentially a washing step with water. Also, the data provided in P19 (Table I on page 5) summarizing the contents of transition metals of the magnesium hydroxide particles show that iron is the main impurity and that in all the compositions cobalt, chromium, copper, vanadium and nickel are present in very low amounts. As a result, P1 contains a motivation to reduce the content not only of impurities in general but also the content of transition metals in particular.

7.4 Hence, the above considerations in respect of inventive step of the subject matter of claim 1 of the main request are not affected by the definition of the transition metal content of the magnesium hydroxide particles. Thus, claim 1 of the first auxiliary request does not involve an inventive step (Article 56 EPC).

Second auxiliary request

- 8. Modifications
- 8.1 Claim 1 of the second auxiliary request corresponds to claim 5 of the patent in suit when it refers to claim 2. The claims of the second auxiliary request fulfill the requirement of Article 123(3) EPC.
- 8.2 The application as originally filed provides a basis for claim 1 of the second auxiliary request in originally filed claim 6 when it refers to claims 5, 4, 3, 2 and 1 further limited to the range of not more than 0.01% by weight found in the passage of page 5 lines 1 to 11. The claims of the second auxiliary request fulfill the requirement of Article 123(2) EPC.

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- 8.3 The allowability of the modifications performed in the claims of the second auxiliary request have not been contested by the opponent nor does the board see a reason to object to these claims.
- 9. Sufficiency of disclosure and novelty
- 9.1 The opponent did not object to the sufficiency of disclosure and novelty of the claims of the second auxiliary request. The above reasoning on the sufficiency of disclosure and novelty apply mutatis mutandis to the second auxiliary request.
- 10. Inventive step
- 10.1 Claim 1 of the second auxiliary request differs from that of the first auxiliary request in that the range describing the average secondary particle diameter is further limited to 0.4 to 1.0 µm.
- 10.2 Representative examples of compositions according to claim 1 of the second auxiliary request are found in P19, page 11, Table 2. The compositions of examples 5, 6, 7 and comparative examples 7 and 8 contain magnesium hydroxide particles of a similar BET surface area of below 10 m^2/q and a content in transition metal impurities below 0.01% by weight according to Table I on page 4. Those compositions only differ in the average secondary diameter of the magnesium hydroxide particles which is 0.63 µm in example 5 and above 1.0 μm in the examples 6, 7 and comparative examples 7 and 8. Example 5 is therefore representative of the compositions of claim 1 of the second auxiliary request. The compositions according to the examples 6, 7 and comparative examples 7 and 8 are representative of the closest prior art P1, which discloses the use of

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magnesium hydroxide particles with a secondary particle diameter of 1.9 μm (example 1 of P1) and 1.4 μm (example 2 of P1).

- 10.3 A look at Table II on page 12 of P19 reveals that the composition according to claim 1 of the second auxiliary request shows a marked improvement of the tensile strength before and after heat deterioration compared to the compositions not according to the second auxiliary request. Hence, the technical problem that can be derived from that Table is to provide flame retardant synthetic resin compositions with improved tensile strength. That problem is effectively solved by the composition according to claim 1 of the second auxiliary request.
- 10.4 Although P1 generally contemplates the use of magnesium hydroxide particles of an average secondary particle diameter between 0.5 and 5.0 μ m (page 7, line 26), it does not hint at particles according to claim 1 of the second auxiliary request in order to solve the technical problem posed. Also, no indication can be found in the cited prior art that an improved tensile strength of synthetic resin compositions can be obtained with particles of an average secondary particle diameter of between 0.4 and 1.0 μ m.
- 10.5 Therefore, no document, either taken alone or in combination, suggests the solution for the problem now being claimed. The subject matter of claim 1 of the second auxiliary request is inventive (Article 56 EPC).

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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 in amended form on the basis of the second auxiliary request filed with the patent proprietor's statement of grounds of appeal, and a description to be adapted thereto.

The Registrar:

On behalf of the Chairman (according to Art. 8(3) RPBA):



B. ter Heijden

R. Cramer

Decision electronically authenticated