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Datasheet for the decision of 3 May 2019

Case Number: T 1210/13 - 3.3.02

Application Number: 03762055.6

Publication Number: 1517901

IPC: C07D301/10

Language of the proceedings: EN

Title of invention:

A METHOD FOR THE START-UP OF AN EPOXIDATION PROCESS AND A PROCESS FOR THE EPOXIDATION OF AN OLEFIN

Patent Proprietor:

Shell Internationale Research Maatschappij B.V.

Opponents:

BASF SE

Scientific Design Company Inc.

Headword:

SHELL / ETHYLENE EPOXIDATION

Relevant legal provisions:

EPC Art. 83, 111(1) RPBA Art. 12(4), 13(1)

Keyword:

Late-filed documents - admitted (yes)
Sufficiency of disclosure - (yes)
Remittal to the department of first instance (yes)

Decisions cited:

T 0608/07, T 2290/12, T 2233/12

Catchword:



Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 1210/13 - 3.3.02

DECISION
of Technical Board of Appeal 3.3.02
of 3 May 2019

Appellant: Shell Internationale Research Maatschappij B.V.

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 26 March 2013 revoking European patent No. 1517901 pursuant to

Article 101(3)(b) EPC.

Composition of the Board:

ChairmanM. O. MüllerMembers:M. Maremonti

L. Bühler

- 1 - T 1210/13

Summary of Facts and Submissions

- The appeal by the patent proprietor (hereinafter "appellant") lies from the decision of the opposition division to revoke European patent No. 1 517 901.
- II. During the opposition proceedings, the appellant filed seventeen sets of claims to be considered as its main request and as auxiliary requests 1 to 16. Independent claims 1 and 8 according to the main request read as follows:
 - "1. A method for the start-up of a process for the epoxidation of ethylene, which method comprises
 - contacting a packed catalyst bed comprising a silver-based epoxidation catalyst, which comprises, in addition to silver, a Group IA metal, and one or more selectivity enhancing dopants selected from rhenium, molybdenum and tungsten with a feed comprising oxygen at a temperature of the catalyst bed from 265 to 300 °C for a period of from 1 to at most 150 hours, and
 - subsequently decreasing the temperature of the catalyst bed to a value of at most 260 °C."
 - "8. A process for the epoxidation of ethylene, which process comprises
 - contacting a catalyst bed comprising a silver-based highly selective epoxidation catalyst, which comprises, in addition to silver, a Group IA metal, and one or more selectivity enhancing dopants selected from rhenium, molybdenum and tungsten, with a feed comprising oxygen at a temperature of

- 2 - T 1210/13

the catalyst bed from 265 to 300 $^{\circ}\text{C}$ for a period from 1 to at most 150 hours, and

- subsequently decreasing the temperature of the catalyst bed to a value of at most 260 °C and contacting the catalyst with the feed comprising ethylene and oxygen."
- III. The following document was cited, inter alia, during the opposition proceedings:

D6: US 4 761 394 A

The opposition division decided that main request and all auxiliary requests did not fulfil the requirements of Article 83 EPC.

IV. In its statement setting out the grounds of appeal, the appellant re-filed the seventeen sets of claims already filed before the opposition division to be regarded as its main request and as auxiliary requests 1 to 16. It argued that the claimed subject-matter was sufficiently disclosed in the contested patent. It corroborated its argumentation by filing, inter alia, the following new items of evidence:

D24: WO 00/17946 A

D25: E.T. McBee et al., "Catalytic Vapor-Phase Oxidation of Ethylene", Industrial and Engineering Chemistry, Vol. 37, No. 5, May 1945, pages 432-438

D26: "Statistics for Business and Economics", E.
Mansfield, W.W. Norton & Company, 1983, page 34

D27: WO 2008/140714 A

- 3 - T 1210/13

- V. In their replies to the statement of grounds, opponents 1 and 2 (hereinafter "respondent 1" and "respondent 2") maintained that the claimed subjectmatter was not sufficiently disclosed.
- VI. In a further letter, respondent 2 corroborated its argument by relying on the following new items of evidence:

D28: US 2009/0234144 A

- D29: E.P.S. Shouten et al., "Oxidation of Ethene in a Wall-cooled Packed-bed Reactor", Chemical Engineering Science, Vol. 49, No. 24A, 1994, pages 4725-4747
- VII. In a subsequent letter dated 13 February 2018 accompanied by Annex 1 and Annex 2, the appellant rebutted the arguments of the respondents and further submitted that documents D28 and D29 filed by respondent 2 should not be admitted into the proceedings. It additionally relied on the following newly filed items of evidence:

D30: US 4 729 826 A

D31: US 4 921 681 A

as well as on decision T 2233/12.

- VIII. On 3 January 2019, the board issued a communication in preparation for the oral proceedings. The board expressed, inter alia, the preliminary opinion that the claimed subject-matter appeared to be sufficiently disclosed.
- IX. In a further letter, respondent 1 commented on issues raised in the board's communication.

- 4 - T 1210/13

- X. Oral proceedings before the board were held on 3 May 2019.
- XI. Final requests

The appellant requested that the appealed decision be set aside and the case be remitted to the opposition division for the examination of novelty and inventive step on the basis of the claims of the main request and of auxiliary requests 1 to 16 as filed with the statement setting out the grounds of appeal.

It also requested that documents D28 and D29 not be admitted into the proceedings.

Respondent 1 requested that the appeal be dismissed. Alternatively, it requested that the contested patent be revoked under Article 100(a) and (c) EPC. It also requested that documents D25 to D27 not be admitted into the proceedings.

Respondent 2 requested that the appeal be dismissed. It also requested that documents D24 to D27 not be admitted into the proceedings. Further, it requested that, should documents D28 and D29 not be admitted, D30 and D31 should also not be admitted into the proceedings.

XII. The arguments of the appellant, where relevant to the present decision, may be summarised as follows:

Admittance of late-filed documents:

- D28 and D29 were filed by respondent 2 almost four years after its reply to the statement of grounds of appeal. No indication was given as to why these documents could not have been filed earlier.

- 5 - T 1210/13

- Such late-filed documents should only be admitted in exceptional circumstances if they were *prima* facie highly relevant, which was not the case here.
- In fact, D28 was published after the priority date of the contested patent. Moreover, it disclosed data concerning the temperature of the coolant and not of the catalyst bed. Indirect methods of temperature measurement were not relevant to the present proceedings.
- D29 described an experimental study carried out on a wide internal diameter reactor tube under very specific laboratory testing conditions. This was not representative of industrial reactor set-ups and process conditions at the priority date of the contested patent. Additionally, a different catalyst was used.
- Therefore, D28 and D29 were not *prima facie* relevant and should not be admitted into the proceedings.

Sufficiency of disclosure:

- The skilled person was aware of the paramount importance of controlling and monitoring the catalyst temperature in olefin epoxidation reactions in view of safety concerns.
- Moreover, they were familiar with direct measurement techniques, i.e. by means of thermocouples inserted into the bed, in order to monitor such reactions, as shown for example in D24 and D25. Thermocouples were commonly used in the field of the contested patent. The measurement

- 6 - T 1210/13

results could be obtained instantaneously with an accuracy within $1\,^{\circ}\text{C.}$

- The indication in paragraph [0018] of the contested patent that the catalyst bed temperature had to be deemed to be the "weight average temperature of the catalyst particles" did confirm that the catalyst temperature as mentioned in the claims had to be intended as the temperature obtained by direct measurements at various points along the catalyst bed. The more measurements were carried out the more accurate the temperature determination.
- Said weight average was calculated by a simple arithmetic calculation, i.e. the simple average of the measured temperatures of the weight fractions of the catalyst bed. In practice, since the weight of the catalyst as a function of the bed length was constant (constant loading density) along the length of the tubular reactor, the weight average became the arithmetic average. The simplicity of such a calculation was readily illustrated in document D30 (column 6, lines 9 to 16).
- This averaging method further minimised the effect of possible transient hot spots on the precision of the temperature measurement.
- Document D29 represented an experimental study aimed at developing a new process in a wide internal diameter reactor tube. A different catalyst was used compared with the catalyst defined in the claims at issue. In the field of olefin epoxidation, reactor tubes had a relatively small diameter compared with other processes such as refining. It was common general knowledge that the radial temperature gradients in such reactor

- 7 - T 1210/13

tubes were not so large, and the simple method of averaging together the temperature measurement points along the tube axis was commonly used in the field to estimate an average temperature.

- The scenario where there was a temperature difference of only 5 °C between the first and second phase of the method of claim 1 was chosen by the respondents with a mind willing to misunderstand the claimed method. Paragraph [0043] of the contested patent taught that during the post break-through phase, the catalyst temperature should be kept low. The value of 260 °C was merely the upper limit of a range decreasing to values as low as 180 °C. Therefore, typical temperature differences between the first and second phase of the method of claim 1 were much greater than 5 °C.
- As to the feature "highly selective epoxidation catalyst" of claim 8, this term has been used in the art to identify a specific class of catalysts. Claim 8 included a specific compositional definition. It was therefore immediately clear to the skilled person whether or not a catalyst fell under the mentioned feature. Moreover, the contested patent gave in paragraph [0026] specific examples of commercially available catalysts that could be used in accordance with the invention.
- Figure 1 of D6 showed results obtained at 40% oxygen conversion. Paragraph [0019] of the contested patent instead made clear that the selectivity referred to in claim 8 referred to zero oxygen conversion. The latter might not be calculated from the values reported in D6. The

- 8 - T 1210/13

- objection of respondent 2 had to be regarded as a clarity objection at the most.
- Reference should be made to previous decision
 T 2233/12 taken on a very similar case, where the
 board decided that the same contested features as
 in the present case did not raise any insufficiency
 problems.
- Therefore, the claimed subject-matter was sufficiently disclosed in the patent in suit.
- XIII. The respondents essentially counter-argued as follows:

 Admittance of late-filed documents:
 - D24 to D27 did not represent documents which might change the outcome of the proceedings. Furthermore, they did not illustrate the common general knowledge in the art. Moreover, no reasons were given to justify the late filing of these documents. Therefore, these documents should not be admitted into the proceedings.
 - Documents D28 and D29 were highly relevant. D28 showed that there might be significant and non-constant temperature differences between the shell-side coolant temperature and the catalyst bed. D29 demonstrated the existence of substantial temperature deviations along both the axial and the radial position of the thermocouples within an epoxidation reactor. Therefore, both documents should be admitted into the proceedings.
 - Should D28 and D29 not be admitted, D30 and D31 should also not be admitted.

- 9 - T 1210/13

Sufficiency of disclosure:

- The contested patent did not clarify which temperature should be intended as the "temperature of the catalyst bed" mentioned in claims 1 and 8 and how this temperature should be measured.
- In fact, the temperature might be measured directly by means of thermocouples or also indirectly by measuring the temperature of the coolant or the gas temperature at the reactor inlet or outlet. The resulting values could be very different from each other.
- Moreover, it was known that the epoxidation reaction was exothermic. This generated temperature gradients both axially and radially. Especially during start-up, the presence of local hot spots was a well-known phenomenon as explained in the contested patent itself, paragraph [0017]. Therefore, different results would be obtained even using thermocouples, depending on their number and placement.
- The skilled person did not have any preference for using thermocouples since these were known for their limited accuracy. They were known to require a certain period of time to adapt to the surrounding temperature, and thus they might not reliably link measured temperatures with time periods. Additionally, the introduction of thermocouples into the catalyst bed could lead to thermal bridges or flow restrictions which would falsify the obtained results.
- Claims 1 and 8 had to be sufficiently disclosed across their full scope. Therefore, also the very

- 10 - T 1210/13

small relative temperature difference of only 5°C between the first and second phase of the claimed method had to be determined. The measurements thus had to be very accurate and to be further correlated with time measurements. No information was provided in the contested patent as to how this might be achieved in the presence of strong temperature fluctuations.

- D28 showed that typical measurement methods which the skilled person had at his disposal, i.e. the measurement of the coolant temperature, suffered from substantial inaccuracies.
- Even assuming that thermocouples were used, D29 demonstrated the presence of temperature gradients within the reactor, both axially and radially. Figure 6(a) showed temperature differences within the reactor of about 40 °C, i.e. higher than the whole first range mentioned in claims 1 and 8.
- In paragraph [0018] the contested patent gave a specific definition of the catalyst bed temperature to be the "weight average temperature of the catalyst particles". Such a definition had no unambiguous acknowledged meaning in the art. In fact, this terminology did not even correspond to the "weighted average catalyst bed temperature" (WABT) as used in e.g. D30 (column 6, lines 9 to 16). This was even more evident in consideration of paragraph [0017]. Here, with reference to prior art, the coolant temperature and the temperature of the catalyst bed were referred to. By referring in paragraph [0018] to the temperature of the catalyst particles, something else must have been intended.

- 11 - T 1210/13

- In paragraph [0018] the appellant deliberately chose to use its own unrecognised definition of the catalyst bed temperature. According to case law, in such a case, the whole description had to be used as the patent's dictionary and the given definition should be followed in order to assess the correct meaning of the temperature mentioned in the claim. However, no guidance was provided in the rest of the patent which would allow the skilled person to understand what temperature was intended and how it should be measured. This lack of information affected the whole ambit of the claim.
- As to the feature "highly selective epoxidation catalyst" of claim 8, the information provided in the description of the contested patent was confusing, even when taking into account the general knowledge in the art. Paragraph [0019] referred to the catalyst selectivity at zero oxygen conversion. Various parameters such as for example the presence, type and amounts of carrier gas, the presence of reaction modifiers or inhibitors in the feed, initial catalyst pretreatments, such as a pre-chlorination, significantly influenced the theoretical selectivity at zero oxygen conversion. None of these parameters was mentioned in said paragraph [0019]. Depending on these parameters, one and the same catalyst would or would not qualify as being a "highly selective catalyst", whatever the meaning of this term might be.
- It was acknowledged that claim 8 mentioned the composition of the catalyst, particularly the present of dopants. However, figure 1 of D6 demonstrated that depending on the dopant (rhenium) concentration, the catalyst selectivity might also

- 12 - T 1210/13

be lower than that obtained without any dopant. The mere mention of the dopant in claim 8 was thus not sufficient to allow a skilled person to understand which catalyst qualified as a "highly selective epoxidation catalyst" within the meaning of claim 8. This ambiguity affected the whole claimed scope.

- The respondents therefore concluded that the claimed subject-matter was not sufficiently disclosed.

Reasons for the Decision

Admittance of late-filed documents into the proceedings

1. The board noted the arguments of the parties concerning the admittance of documents D24 to D31 into the proceedings (XII and XIII, supra). However, the board was convinced that all late-filed documents D24 to D31 merely supported the arguments of the parties filed previously. They neither increased the complexity of the case nor raised new substantial issues. The parties had enough time to familiarise with their contents and to comment on them.

Therefore, by exercising its discretion, the board decided to admit documents D24 to D27 (Article 12(4) RPBA) and D28 to D31 (Article 13(1) RPBA) into the proceedings.

Main request - Sufficiency of disclosure - Article 83 EPC

2. Both respondents submitted that the subject-matter of the main request was not sufficiently disclosed in the contested patent, in view of the feature "temperature"

- 13 - T 1210/13

of the catalyst bed" as included in the following provision defined in claims 1 and 8 (II, supra):

- "contacting a packed catalyst bed comprising [...] with a feed comprising oxygen at a temperature of the catalyst bed from 265 to 300°C for a period from 1 to at most 150 hours, and subsequently decreasing the temperature of the catalyst bed to a value of at most 260°C" (emphasis added by the board).

Respondent 2 further argued that the feature:

- "highly selective epoxidation catalyst"

as mentioned in claim 8 of the main request (II, *supra*) was also not sufficiently disclosed in the patent in suit.

- 3. Temperature of the catalyst bed
- 3.1 Claims 1 and 8 at issue require the "temperature of the catalyst bed" to be maintained "from 265 to 300°C" for a certain period of time (1 to 150 hours), and subsequently decreased "to a value of at most 260°C".

The respondents argued (XIII, *supra*) that there were different methods for measuring the referenced temperature. Since these methods led to different results, the invention defined in claims 1 and 8 was insufficiently disclosed.

3.2 The board notes that the claims specify neither where nor how the mentioned temperature is measured. Therefore, the adopted broad formulation of the claims covers all measurement methods available to the skilled person as long as they are able to meet the claimed requirements. In the board's view, measurements for

determining the temperature of a catalyst bed pertain to the routine practice of a person skilled in the art of catalytic reactions and do not pose any technical difficulty. Given the type of reaction (here: ethylene epoxidation), the skilled person would thus select the most appropriate measuring method, depending on variables such as the longitudinal and radial dimensions of the catalyst bed and the composition of the feed.

3.3 In this respect, it is common general knowledge for the skilled person that direct measurements by means of conventional thermocouples placed at different locations within the bed constitute the most convenient way of obtaining precise real-time measurements of the temperature of a catalyst bed. Such a technique has been known to the skilled person for a long time; see for example document D25 dated May 1945, disclosing (abstract, figures 2 and 3, left-hand column on page 433, page 435, table III and paragraph entitled "Temperature of optimum conversion" on page 436) the use of thermocouples for determining the temperature of a catalyst bed devoted to the catalytic vapour-phase oxidation of ethylene, i.e. the same reaction as defined in claims 1 and 8 at issue.

The skilled person would be fully aware that the more measurement points taken within the bed, the more accurate the temperature determination will be, since temperature gradients in both axial and radial directions, if present, can be taken into account in this way.

In the board's judgement, the choice of the number of measurement points within a catalyst bed belongs to the routine practice of a skilled person, who would select

- 15 - T 1210/13

this number specifically as a function of the catalyst bed's axial and radial dimensions.

3.4 The respondents argued (XIII, *supra*) that the skilled person would refrain from using thermocouples since these were known for their limited accuracy.

Additionally, their introduction into the catalyst bed would lead to thermal bridges or flow restrictions falsifying the obtained results.

However, there is no evidence on file that would support these assertions. Document D29 (paragraph "Measurement and control section" on pages 4730 and 4731, figures 4 and 6(a)) submitted by respondent 2, rather confirms that thermocouples placed at different locations within the catalyst bed are able to provide accurate temperature measurements taking radial and axial temperature gradients into account.

- 3.5 Hence, on the basis of the wording of claims 1 and 8 and common general knowledge, the skilled person would already know that the most accurate way to measure the referenced catalyst bed temperature is to use multipoint thermocouples inserted at various locations within the catalyst bed.
- 3.6 The respondents argued (XIII, supra) that, even assuming that thermocouples were used to measure the temperature at various locations within the bed, the contested patent, particularly paragraph [0018], lacked guidance as to how to deal with these measurements so that the "temperature of the catalyst bed" within the meaning of claims 1 and 8 might be obtained.

The board disagrees. Paragraph [0018] of the contested patent states that the temperature of the catalyst bed "is deemed to be the weight average temperature of the

- 16 - T 1210/13

catalyst particles". The reference to the weight average unambiguously confirms that the catalyst temperature should be measured directly within the catalyst bed by means of thermocouples placed at different locations within the bed. Each measurement would be representative of a certain catalyst mass. The various measurements would then have to be weighted accordingly in order to calculate the weight average temperature. The board is convinced that such a calculation does not pose any difficulty to the skilled person. As mentioned by the appellant, such a calculation is exemplified for example in document D30, disclosing (column 6, lines 4 to 27) that the weight average temperature is obtained as $\Sigma T\Delta W/W$, wherein W is the total catalyst's weight, ΔW is the weight of a catalyst's portion and T is the temperature measured within this catalyst portion.

The board cannot follow the argument of the respondents (XIII, supra) that the above definition given in paragraph [0018] would be unusual or even obscure to a skilled person. In the present case, the board considers the terms "catalyst", "catalyst bed" and "catalyst particles" to be interchangeable and relate all to the total amount of catalyst involved. The board cannot see any other interpretation, which would make sense in the context of the claimed invention (see also T 2233/12, reasons 2.4 and 2.4.1). Therefore, no contradiction can be seen between paragraph [0017] referring to the temperature of the catalyst bed and paragraph [0018] mentioning the temperature of the catalyst particles.

3.7 The respondents further submitted (XIII, *supra*) that the claimed subject-matter covered a temperature difference between the first and second phase of the

- 17 - T 1210/13

claimed method of only 5 °C. The skilled person would not know how to determine such a small temperature difference given the temperature fluctuations, especially in the radial direction, within the bed. D29 was cited as evidence of such fluctuations.

The board, however, has no reason to doubt that, by following the averaging procedure mentioned above, the skilled person is able to determine the "temperature of the catalyst bed" within the meaning of claims 1 and 8 at issue with sufficient accuracy. As to D29, this document discloses ("Reactor section" on page 4730) a wall-cooled tubular bed reactor. The skilled person is aware that when cooling is performed from the wall, a temperature profile would necessarily form with temperature values at the wall lower than in the centre of the tube. The skilled person carrying out the claimed method would, however, recognise the importance of maintaining a substantially uniform temperature within the catalyst bed. They would thus select a sufficiently narrow reactor diameter so that radial temperature gradients become negligible. This is regarded as a routine optimisation procedure for a person skilled in the art of catalysis.

Irrespective of the above, the objection of the respondents may at most be seen to be related to the clarity of the feature "temperature of the catalyst bed" that was already present in claims 1 and 8 as granted and thus cannot be objected to under Article 84 EPC (G 3/14, order). By keeping an adequate distance from the borderline temperatures defined in claims 1 and 8 for the two phases of the claimed method, the skilled person would definitely be able to repeat the claimed invention. Even accepting arguendo that there was some uncertainty at the claim edges with regard to

- 18 - T 1210/13

the feature in question, such uncertainty does not permeate the whole ambit of claims 1 and 8 at issue (T 2233/12, reasons 2.4.2-2.4.3).

- 3.9 The board concludes that the opposition division's finding of insufficiency of disclosure as regards the temperature of the catalyst bed does not hold good.
- 4. Highly selective epoxidation catalyst
- 4.1 The expression "highly selective" in claim 8 of the main request may indeed be regarded as a relative term, causing some ambiguity as to which catalysts fall under this term and which do not. This expression was, however, already present in claim 8 as granted and thus cannot be objected to under Article 84 EPC (G 3/14, order). Moreover, claim 8 further specifies the composition of the mentioned catalyst, said to comprise (II, supra) "in addition to silver, a Group IA metal, and one or more selectivity enhancing dopants selected from rhenium, molybdenum and tungsten". Therefore, any catalyst fulfilling this compositional requirement is to be regarded as a highly selective epoxidation catalyst within the meaning of claim 8. The contested patent, in paragraphs [0026] and [0066], also gives specific examples of catalysts suitable for use in the claimed process. Therefore, the determination of a catalyst according to claim 8 does not place any undue burden on the skilled person trying to reproduce the claimed process (see also T 2233/12, reasons, 1.2 to 1.4).
- During oral proceedings, respondent 2 referred to D6, particularly to figure 1, to corroborate its insufficiency objection (XIII, supra). Document D6 concerns (column 1, line 66 to column 2, line 12) an ethylene oxide catalyst, comprising silver, an alkali

- 19 - T 1210/13

metal, especially cesium, and rhenium. Figure 1 of D6 shows that the catalyst's selectivity is a function of the cesium content of the tested catalyst. On the one hand, such results do not have any bearing on the previous conclusion that any catalyst meeting the compositional requirement of claim 8 falls under the catalyst definition of the claim. On the other hand, the optimisation of the concentration of the individual catalyst's components pertains to the routine practice of a person skilled in the art of catalysis and is not considered to involve any particular technical difficulty.

- Also in this case, the possible ambiguity derived from using the relative term "highly selective" in claim 8 may at most affect the clarity of the subject-matter at the claim edges. In other words, it could cause uncertainty as to whether the skilled person is working within or outside the claim's ambit. Said ambiguity however, does not permeate the entire claimed scope (T 2290/12, reasons 3.1 and T 608/07, reasons 2.5.1 and 2.5.2).
- 4.4 The insufficiency objection based on the epoxidation catalyst's definition is thus also not convincing.

Conclusion

5. None of the arguments put forward by the respondents could raise serious doubts about the sufficiency of disclosure of the claimed subject-matter. The board thus concludes that no undue burden is posed on the skilled person trying to carry out the claimed invention. The requirements of Article 83 EPC are therefore met.

- 20 - T 1210/13

Remittal

6. The patent was revoked for lack of sufficiency of disclosure under Article 83 EPC. It remains to be assessed whether one of the other grounds for opposition invoked by the respondents (added subject-matter, lack of novelty, lack of inventive step) prejudices the maintenance of the contested patent.

Since the main purpose of opposition appeal proceedings is to review the decision taken by the opposition division, the board finds it appropriate to use its discretion under Article 111(1) EPC and to remit the case to the opposition division for further prosecution, in accordance with the appellant's request to this end. It is noted that no objections to the remittal of the case were raised by the respondents.

- 21 - T 1210/13

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the opposition division for further prosecution on the basis of the main request filed with the statement setting out the grounds of appeal.

The Registrar:

The Chairman:



N. Maslin M. O. Müller

Decision electronically authenticated