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*B01J 31/02* (2006.01) *C07C 6/04* (2006.01)  
*B01J 31/22* (2006.01)

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(81) **Designated States** (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN,

[Continued on next page]

(54) Title: BIOPOLYMER- SUPPORTED IONIC LIQUID PHASE METATHESIS CATALYST

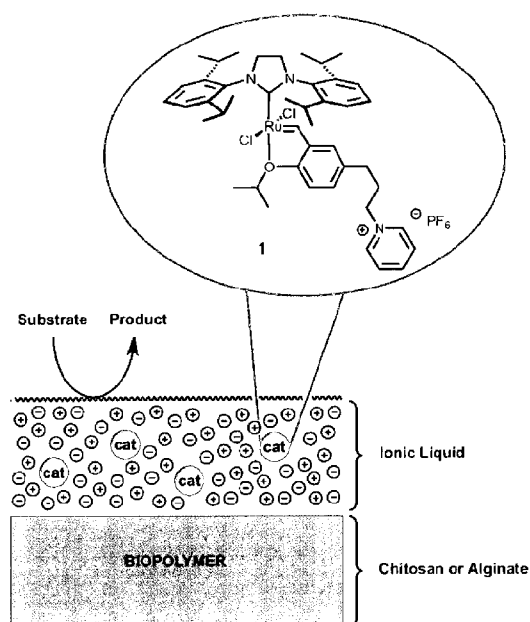


Figure 10

(57) **Abstract:** The present invention concerns a supported ionic liquid phase catalyst comprising a support and a catalyst dispersed in an ionic liquid on said support. The support comprises a biopolymer, preferably chitosan or alginate, and the catalyst is a metathesis catalyst, preferably a pyridinium-tagged Grubbs-Hoveyda II catalyst. Further, the present invention concerns a method of performing an olefin metathesis, in particular RCM. The method of the invention comprises the use of a supported ionic liquid phase catalyst. Furthermore, the present invention concerns the use of a supported ionic liquid phase catalyst in olefin metathesis reactions. Finally, the present invention also concerns an ionic liquid exhibiting a suitable adsorption to a biopolymer. The ionic liquid comprises [Butyl-Methyl-imidazolium]X salts, wherein X is selected in the group consisting of PF<sub>6</sub> or BF<sub>4</sub> or PF<sub>3</sub>(C<sub>2</sub>F<sub>5</sub>)<sub>3</sub>.



HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

**(84) Designated States** (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU,

LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

**Published:**

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

**(88) Date of publication of the international search report:**

29 August 2013

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/EP2012/003081

## Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☒ Claims Nos.: 2-7(completely); 1, 8-14(partially)  
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:  
see FURTHER INFORMATION sheet PCT/ISA/210
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:  
1-14

### Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

# INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2012/003081

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|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------|
| <b>A. CLASSIFICATION OF SUBJECT MATTER</b><br>INV. B01J31/06      B01J31/02      B01J31/22      C08B37/00      C07C6/04<br>ADD.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                                                                       |                                                                                                                               |
| According to International Patent Classification (IPC) or to both national classification and IPC                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                       |                                                                                                                               |
| <b>B. FIELDS SEARCHED</b><br>Minimum documentation searched (classification system followed by classification symbols)<br>B01J C08B C07C                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                       |                                                                                                                               |
| Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                       |                                                                                                                               |
| Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)<br>EPO-Internal, WPI Data                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                                                                                                                                       |                                                                                                                               |
| <b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                       |                                                                                                                               |
| Category*                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | Citation of document, with indication, where appropriate, of the relevant passages                                                                    | Relevant to claim No.                                                                                                         |
| Y                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | DE 10 2009 017498 A1 (SUED CHEMIE AG [DE])<br>28 October 2010 (2010-10-28)<br>cited in the application                                                | 1,8,9,<br>11-14                                                                                                               |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | the whole document                                                                                                                                    | 10                                                                                                                            |
| -----                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                       |                                                                                                                               |
| Y                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | WO 2008/065187 A1 (ECOLE NALE SUP ARTES<br>METIERS [FR]; CENTRE NAT RECH SCIENT [FR];<br>MAUDUI) 5 June 2008 (2008-06-05)<br>cited in the application | 1,8,9,<br>11-14                                                                                                               |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | page 1, lines 6-7; claims 1,5,19,20,21,22;<br>compounds 11-14<br>page 2, line 22 - page 3, line 9<br>page 11, lines 16-20<br>pages 24-27              | 10                                                                                                                            |
| -----                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                       |                                                                                                                               |
| -/--                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                                                                                                                                       |                                                                                                                               |
| <div style="display: flex; justify-content: space-between;"> <span><input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C.</span> <span><input checked="" type="checkbox"/> See patent family annex.</span> </div>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |                                                                                                                                                       |                                                                                                                               |
| <div style="display: flex;"> <div style="flex: 1;"> <p>* Special categories of cited documents :</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="flex: 1;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&amp;" document member of the same patent family</p> </div> </div> |                                                                                                                                                       |                                                                                                                               |
| Date of the actual completion of the international search<br><br><div style="text-align: center; font-size: 1.2em;">15 March 2013</div>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                                                                                                                                                       | Date of mailing of the international search report<br><br><div style="text-align: center; font-size: 1.2em;">10/07/2013</div> |
| Name and mailing address of the ISA/<br>European Patent Office, P.B. 5818 Patentlaan 2<br>NL - 2280 HV Rijswijk<br>Tel. (+31-70) 340-2040,<br>Fax: (+31-70) 340-3016                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                                                                                                                                                       | Authorized officer<br><br><div style="text-align: center; font-size: 1.2em;">Goebel, Matthias</div>                           |

## INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2012/003081

| C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT |                                                                                                                                                                                                                                                                                                                                                                             |                       |
|------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| Category*                                            | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                                                                                                                                                                                          | Relevant to claim No. |
| Y                                                    | PRASHANT H. DESHMUKH ET AL: "Alkene metathesis: the search for better catalysts", DALTON TRANSACTIONS, no. 24, 1 January 2007 (2007-01-01), page 2479, XP055055782, ISSN: 1477-9226, DOI: 10.1039/b703164p cited in the application                                                                                                                                         | 1,8,9,<br>11-14       |
| A                                                    | page 2488 - page 2489; figure 13; compounds 84-87<br>page 2481                                                                                                                                                                                                                                                                                                              | 10                    |
| Y                                                    | JÉRÔME BAUDOUX ET AL: "Development of new SILP catalysts using chitosan as support", GREEN CHEMISTRY, vol. 9, no. 12, 1 January 2007 (2007-01-01), page 1346, XP055055824, ISSN: 1463-9262, DOI: 10.1039/b709226a cited in the application                                                                                                                                  | 1,8,9,<br>11-14       |
| A                                                    | the whole document                                                                                                                                                                                                                                                                                                                                                          | 10                    |
| Y                                                    | RENAUD MOUCEL ET AL: "Importance of the Conditioning of the Chitosan Support in a Catalyst-Containing Ionic Liquid Phase Immobilised on Chitosan: The Palladium-Catalysed Allylation Reaction Case", ADVANCED SYNTHESIS & CATALYSIS, vol. 352, no. 2-3, 15 February 2010 (2010-02-15), pages 433-439, XP055055832, ISSN: 1615-4150, DOI: 10.1002/adsc.200900515             | 1,8,9,<br>11-14       |
| A                                                    | the whole document                                                                                                                                                                                                                                                                                                                                                          | 10                    |
| Y                                                    | A. KERAANI ET AL: "Immobilisation of an ionically tagged Hoveyda catalyst on a supported ionic liquid membrane: An innovative approach for metathesis reactions in a catalytic membrane reactor", CATALYSIS TODAY, vol. 156, no. 3-4, 31 October 2010 (2010-10-31), pages 268-275, XP055055835, ISSN: 0920-5861, DOI: 10.1016/j.cattod.2010.04.024 cited in the application | 1,8,9,<br>11-14       |
| A                                                    | pages 268-270; figure 2<br>ref [28]                                                                                                                                                                                                                                                                                                                                         | 10                    |
| -----<br>-/--                                        |                                                                                                                                                                                                                                                                                                                                                                             |                       |

## INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2012/003081

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                                                                                                                                                                        | Relevant to claim No. |
|-----------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| A         | <p>TRNKA T M ET AL: "The development of L2X2Ru=CHR olefin metathesis catalysts: An organometallic success story", ACCOUNTS OF CHEMICAL RESEARCH, ACS, WASHINGTON, DC, US, vol. 34, no. 1, 4 November 2000 (2000-11-04), pages 18-29, XP002407672, ISSN: 0001-4842, DOI: 10.1021/AR000114F cited in the application pages 19-20; figure 4</p> <p>-----</p> | 1,13,14               |
| A         | <p>HRYNIEWICKA A ET AL: "New efficient ruthenium metathesis catalyst containing chromenyl ligand", JOURNAL OF ORGANOMETALLIC CHEMISTRY, ELSEVIER-SEQUOIA S.A. LAUSANNE, CH, vol. 695, no. 9, 1 May 2010 (2010-05-01), pages 1265-1270, XP026989819, ISSN: 0022-328X [retrieved on 2010-02-24] the whole document</p> <p>-----</p>                         | 10                    |
| A         | <p>BARBASIEWICZ M ET AL: "Probing of the Ligand Anatomy : Effects of the Chelating Alkoxy Ligand Modifications on the Structure and Catalytic Activity of Ruthenium Carbene Complexes", ADVANCED SYNTHESIS AND CATALYSIS, WILEY, WEINHEIM, DE, vol. 349, 1 January 2007 (2007-01-01), pages 193-203, XP002604756, ISSN: 1615-4169</p> <p>-----</p>        | 10                    |

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2012/003081

| Patent document<br>cited in search report | Publication<br>date | Patent family<br>member(s) | Publication<br>date |
|-------------------------------------------|---------------------|----------------------------|---------------------|
| DE 102009017498 A1                        | 28-10-2010          | NONE                       |                     |
| -----                                     |                     |                            |                     |
| WO 2008065187 A1                          | 05-06-2008          | CA 2671082 A1              | 05-06-2008          |
|                                           |                     | EP 2097431 A1              | 09-09-2009          |
|                                           |                     | EP 2476688 A1              | 18-07-2012          |
|                                           |                     | FR 2909382 A1              | 06-06-2008          |
|                                           |                     | JP 2010511017 A            | 08-04-2010          |
|                                           |                     | US 2010087644 A1           | 08-04-2010          |
|                                           |                     | US 2013144060 A1           | 06-06-2013          |
|                                           |                     | WO 2008065187 A1           | 05-06-2008          |
| -----                                     |                     |                            |                     |

**FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210**

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-14

a catalyst (i) comprising (i-1) any biopolymer support, (i-2) any catalyst suitable for (olefin) metathesis, (i-3) any ionic liquid, present on the support in any way and containing catalyst (ii) dispersed therein to any degree; (process) using (i) in olefin metathesis reactions;

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2. claim: 15

a selection of three specific alternative ionic liquids.

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## FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Claims Nos.: 2-7(completely); 1, 8-14(partially)

1 Present claims \*1, 13, 14\* relate to an extremely large number of possible catalyst compositions and methods (using it). Support and disclosure in the sense of Article 6 and 5 PCT is to be found however for only a very small proportion of the compositions/methods/uses claimed, see pages 4, line 7 - 5, line 3; 7, lines 10-12 and 28; 11, lines 24-27; 23, lines 34-36 and the worked examples. Thus the disclosure is limited to only one specific catalyst, i.e. of Grubbs-Hoveyda II type ("GH-II"), namely compounds 1, 3 and a close analogue thereto, compound 4, specially adapted for one specific ionic liquid ("IL"; via pyridinium tags), together on one specific biopolymer support, pre-treated in a special way (cross-linked and freeze-dried or dried with scCO<sub>2</sub>). Apparently only a very limited choice is possible, see pages 11, lines 24-27 (even stressing that "multiple tests" had to be performed for just the specific Ru complex 1) and 23, lines 34-35, \*no guidance\* being given \*in the application\* how to extend this to cover the whole claimed range of claim 1. Only \*certain\* polysaccharides are disclosed and described as suitable (page 7, lines 10-17 and 28; page 8, lines 8-14: chitosan and alginate), hence no information whether other polymers falling under the generic term would work as well and under which circumstances: e.g. polynucleotides (RNA, DNA)? polypeptides or proteins? polylactic acid? Moreover, it is generally known that e.g. native chitosan \*dissolves\* in common ILs, in particular the envisioned bmim ILs. Hence a special pre-treatment is apparently needed, which, however, must not interfere with catalysis. No indications as to this are found in the claims and only very limited guidance is found in the description and in the worked examples. Page 7, lines 6-8 clearly states that very little research has been made with respect to biopolymers for supports in SILP catalysis, in particular with the envisioned metals. Hence there appears, admittedly, to exist \*no prior art for guidance\* either. It is furthermore known that \*Schrock catalysts\* with Mo or W are \*very sensitive\* to polar groups, especially if containing acidic hydrogens (i.e. chitosan, alginic acid, the preferred supports of the application), see e.g. ref. [1d] = D7 (pages 19-20, Fig 4), [1f] = D3 (page 2481, Sch. 3), document numbering according to the order of appearance in the annexed communication relating to the results of the partial international search. Furthermore the limitations of the prefix "bio" as to its ramifications with respect to the encompassed polymers are not clear either, since apparently no commonly agreed on \*delimiting\* definition appears to exist. Thus e.g. PLA is often considered to be a biopolymer but in fact only the lactic acid \*monomer\* is of biological origin (cf. e.g. <http://en.wikipedia.org/wiki/Biopolymer>). On the other hand, the biopolymers actually used in the application are \*synthetic derivatives\* of embodiments of claimed biopolymers, in particular those \*cross-linked\* with \*glutaraldehyde\* or \*Ca(2+)\* ions. The non-compliance with the substantive provisions is to such an extent, that the search was performed taking into consideration the non-compliance in determining the extent of the search of the claims (PCT Guidelines 9.19 and 9.23). The search of claims 1 and 8-14 was consequently restricted to the specifically disclosed compositions/methods/uses which appear to be

## FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

supported and a generalisation of their structural formulae: claim 1: \*restricted\* to (I) chitosan or alginate supports according to claim 11, (II) the pyridinium-tagged GH-II catalysts of any of claims 8-10, (III) the ionic liquids defined in claim 12 and used in the wexs; claims 13, 14: SILP catalyst \*restricted\* as for claim 1 ; dependent claims 8-12: \*restricted\* accordingly. 2 In view of clarity and support deficiencies mentioned above, applying mutatis mutandis, dependent claims 2-7 are \*not searched\* (Article 17(2) PCT).

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guidelines C-IV, 7.2), should the problems which led to the Article 17(2) declaration be overcome.