

(19) World Intellectual Property Organization
International Bureau(43) International Publication Date
12 October 2006 (12.10.2006)

PCT

(10) International Publication Number
WO 2006/107900 A3

(51) International Patent Classification:

G01N 29/02 (2006.01) G01N 9/00 (2006.01)
G01N 29/036 (2006.01) G01N 11/10 (2006.01)
G01N 29/22 (2006.01) G01N 27/00 (2006.01)

(21) International Application Number:

PCT/US2006/012374

(22) International Filing Date: 31 March 2006 (31.03.2006)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

60/667,818 1 April 2005 (01.04.2005) US

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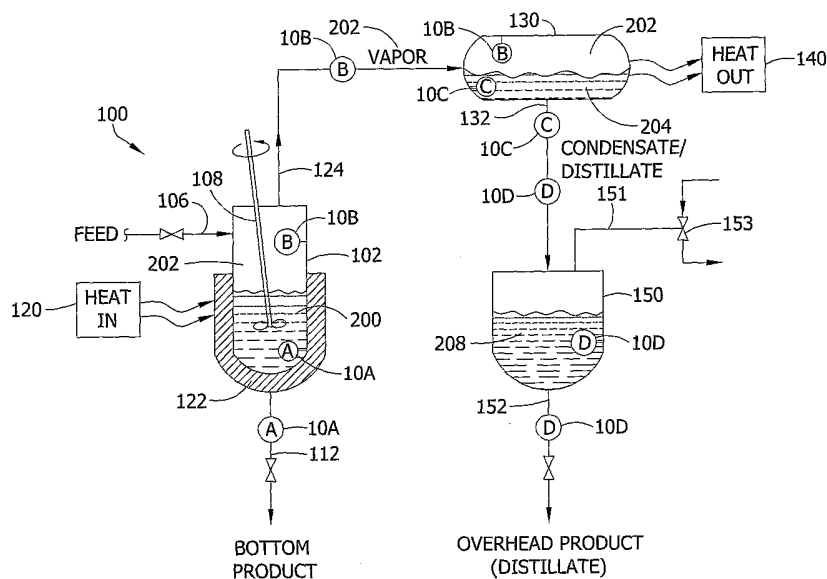
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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

[Continued on next page]

(54) Title: MONITORING BY MEANS OF AN ON-LINE SENSOR AND FLUIDIC OPERATIONS INVOLVING UNIT SEPARATION AND REACTION OPERATIONS



(57) Abstract: Fluid sensor methods and systems adapted for monitoring and/or controlling distillation operations in fluidic systems, such as batch distillation operations or continuous distillation operations, are disclosed. Preferred embodiments are directed to process monitoring and/or process control for unit operations involving endpoint determination of a distillation, for example, as applied to a liquid-component-switching operation (e.g., a solvent switching operation), a liquid-liquid separation operation, a solute concentration operation, a dispersed-phase concentration operation, among others.

WO 2006/107900 A3



Published:

— *with international search report*

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

18 January 2007

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2006/012374

A. CLASSIFICATION OF SUBJECT MATTER

INV. G01N29/02 G01N29/036 G01N29/22 G01N9/00 G01N11/10
G01N27/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

G01N B01D

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 practical, search terms used)

EPO-Internal, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 00/54044 A (MARCONI APPLIED TECHNOLOGIES LIMITED; PIKE, ANDREW; BYFIELD, MARK, PHI) 14 September 2000 (2000-09-14)	1-12, 18-23, 28,30, 32-36, 48,50, 51, 55-59, 62-64, 66-75, 78,80, 81,163, 164,166
Y	abstract ----- -/--	13-17



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"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)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"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

"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

"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.

"&" document member of the same patent family

Date of the actual completion of the international search

25 July 2006

Date of mailing of the international search report

24. 10. 2006

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INTERNATIONAL SEARCH REPORT

International application No

PCT/US2006/012374

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6 088 630 A (CAWLFIELD ET AL) 11 July 2000 (2000-07-11) abstract	1,13-17, 23,64, 67,71, 73,163
X	----- EP 0 304 232 A (BP OIL INTERNATIONAL LIMITED) 22 February 1989 (1989-02-22) abstract	23-25, 28, 32-34, 51,73
X	----- BOZENHARDT H F: "MODERN CONTROL TRICKS SOLVE DISTILLATION PROBLEMS" HYDROCARBON PROCESSING, GULF PUBLISHING CO. HOUSTON, US, vol. 67, no. 6, June 1988 (1988-06), pages 47-50, XP001048628 ISSN: 0018-8190 abstract	23,73
Y	----- WO 2004/036207 A (SYMYX TECHNOLOGIES, INC; MATSIEV, LEONID; VARNI, JOHN, F; KOLOSOV, OLE) 29 April 2004 (2004-04-29) cited in the application abstract	1,23,64, 67,71, 73,163
P,X	----- WO 2005/066092 A (3M INNOVATIVE PROPERTIES COMPANY; BAETZOLD, JOHN, P; BENSON, KARL, E;) 21 July 2005 (2005-07-21) abstract	1,23,64, 67,71, 73,163

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2006/012374

Box 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.: -
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 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 fee, this Authority did not invite payment of any additional fee.

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.:

see annex

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.1

Continuation of Box II.2

Claims Nos.: -

The present independent method claim 23 and apparatus claim 73 relate to a large number of possible methods by claiming an online sensor other than a temperature, pressure or flow sensor.

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 methods claimed, see [Near Infrared (NIR) and Fourier Transform Infrared (FTIR), paragraph 0008; mechanical resonator sensors such as flexural resonator sensors or torsional resonator sensors, as well as refractive index sensors, paragraph 0102; electrical property sensor selected from the group consisting of a dielectric sensor, a conductivity sensor and an optical property sensor, paragraph 0143].

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 claim 23 (PCT Guidelines 9.19 and 9.23).

The search of method claim 23 and mutatis mutandis apparatus claim 73 was restricted to those claimed methods which appear to be supported: A method for monitoring a unit operation that includes separating one or more components of a multi-component composition by distillation, the method comprising providing a multi-component composition comprising one or more liquid components, vaporizing at least a portion of at least one liquid component of the composition to form a vapor, condensing the vapor to form a condensate, recovering at least a portion of the condensate as a distillate, and monitoring the vapor, the condensate or the distillate using at least one on-line sensor, the at least one on-line sensor being other than a temperature sensor, pressure sensor or flow sensor and the at least one on-line sensor being selected from the group consisting of Near Infrared (NIR) and Fourier Transform Infrared (FTIR) sensor, mechanical resonator sensors, refractive index sensors, dielectric sensors, conductivity sensors and optical property sensors.

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

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

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 Guideline C-VI, 8.5), should the problems which led to the Article 17(2) declaration be overcome.

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(part), 2-22; 23(part), 24-25, 28, 30, 32-36, 48, 50-51, 55-59, 62-63; 64(part), 66; 67-70; 71-72; 73(part), 74-75, 78, 80-81; 163(part), 164, 166;

Method for monitoring the separation of components of a multi-component composition by distillation wherein a first, second and third mechanical resonator are contacted with the composition, a vapor, a condensate or a distillate and the response of the resonators is monitored.

2. claims: 23(part), 26-27, 52-54; 73(part), 76,77

System and method for monitoring the separation of components of a multi-component composition by distillation, the method comprising monitoring the vapor, the condensate or the distillate using one on-line sensor to determine fluid-composition-dependent properties, such as viscosity, density, dielectric constant and/or conductivity of the vapor, the condensate or the distillate.

3. claims: 23(part), 29, 31; 73(part), 79

System and method for monitoring the separation process of components of a multi-component composition by distillation, wherein the vapor, the condensate or the distillate is monitored using a refractive index sensor.

4. claims: 23(part), 37-47, 60, 61; 207-208;

Method for monitoring specific separation processes of components of a multi-component composition by distillation with an online sensor, wherein the distillation comprises a specific operation, such as a liquid-component switching operation (claims 37-41, 60) or a concentration operation (claims 42-47, 62) or a distillation column process comprising different stages (claims 207, 208).

5. claims: 1(part), 49

Method for monitoring the separation of components of a multi-component composition by distillation wherein a tuning fork resonator is contacted with a component and the response of the tuning fork resonator is monitored.

6. claims: 82-144; 145-157; 158-162; 180-189; 190-196; 197-206;

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Method for monitoring liquid and vaporous components of a multi-component composition in specific operations in fluidic systems, such as an industrial manufacturing, extraction or refining process comprising altering the composition of a feed mixture (claims 82-144), a distillation process comprising mixed solvent solutions (claims 145-157), a liquid/liquid extraction process comprising a component transfer (claims 158-162), a process comprising removal of a condensed phase component (claims 180-189), a membrane separation process (claims 190-196) or a sorption process (claims 197-206) wherein a sensing surface of a flexural mechanical resonator is contacted with a component and the response of the flexural mechanical resonator monitored.

7. claims: 64(part), 65; 163(part), 165; 218-231

Method for monitoring a crystallization reaction, the method comprising:
providing a multi-component composition in a process vessel, the multi-component composition being a solution or a dispersion comprising one or more liquid components and at least one non-polymeric organic component dissolved or dispersed in the one or more liquid components,
reacting the at least one non-polymeric organic component in the process vessel under reaction conditions, and
monitoring the progress of the reaction in the process vessel by a mechanical resonator response at a first time and at a second time during the reaction.

8. claims: 167-179

Method for monitoring a non-polymeric chemical reaction, wherein a sensing surface of a mechanical resonator is contacted with a fluid comprising a product or a by-product of the reaction and the response of the resonator is monitored.

9. claims: 209-217

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Method for evaluating a parameter of the kinetics of a chemical reaction wherein a sensing surface of a mechanical resonator is contacted with fluid selected from the group consisting of a fluid reaction medium in which the reaction is or has been conducted, a fluid comprising a source of a reactant for the reaction, a fluid comprising a catalyst or source of catalyst for the reaction, a fluid comprising a product of the reaction, a fluid comprising a by-product of the reaction, a fluid that is separated from a fluid reaction medium during or after the reaction and stimulating the sensor while in contact with said fluid; monitoring the response of the resonator; deriving a kinetic parameter of the reaction from data obtained by monitoring said response.

10. claims: 233-239

Method for determining the temperature at which a test solution comprising a known concentration of a solute in a solvent is at its maximum metastable supersaturation concentration, the method comprising:
preparing said test solution at a temperature sufficient to cause all of said solute to be dissolved;
cooling said solution while the solution is in contact with a flexural mechanical resonator; and
monitoring the temperature during cooling and the response of said resonator to detect precipitation, said known concentration of the test solution constituting the maximum metastable supersaturated solution concentration at the precipitation temperature.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2006/012374

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