#### (12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization

International Bureau





(10) International Publication Number WO 2013/008003 A1

(43) International Publication Date 17 January 2013 (17.01.2013)

(51) International Patent Classification: C12H 1/22 (2006.01) G01N 33/14 (2006.01) G01N 21/35 (2006.01)

(21) International Application Number:

PCT/GB2012/051621

(22) International Filing Date:

10 July 2012 (10.07.2012)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

1111837.9

11 July 2011 (11.07.2011) GB

(71) Applicant (for all designated States except US): M SQUARED LASERS LIMITED [—/GB]; Venture Building, 1 Kelvin Campus, West of Scotland Science Park, Maryhill Road, Glasgow Strathclyde G20 0SP (GB).

(72) Inventors; and

(75) Inventors/Applicants (for US only): MALCOLM, Graeme Peter Alexander [GB/GB]; M Squared Lasers, Venture Building, 1 Kelvin Campus, West of Scotland Science Park, Maryhill Road Glasgow Strathclyde G20 0SP (GB). MAKER, Gareth Thomas [GB/GB]; M Squared Lasers Limited, Venture Building, 1 Kelvin Campus, West of Scotland Science Park, Maryhill Road Glasgow Strathclyde G20 0SP (GB). ROBERTSON, Gordon [GB/GB]; M Squared Lasers Limited, Venture Building, 1 Kelvin

Campus, West of Scotland Science Park, Maryhill Road Glasgow Strathclyde G20 0SP (GB).

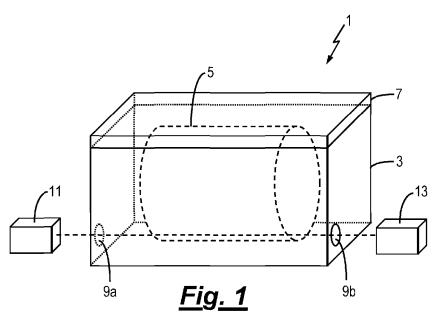
- (74) Agent: LINCOLN IP; 9 Victoria Street, Aberdeen, Aberdeenshire AB10 1XB (GB).
- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, 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, 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))

[Continued on next page]

(54) Title: MATURATION APPARATUS AND METHODS



(57) Abstract: The invention provides an apparatus and a method that reduces fluid loss from a cask during a maturation process by sealably enclosing the cask in a vessel that provides an expansion volume to receive fluid vapour from the cask, a monitoring system and a method that monitors fluid loss from a cask during a maturation process using a light source and a detector to determine the presence of fluid vapour in the vicinity of the cask, a corresponding system for controlling a maturation process in which environmental conditions are controlled, and a cask leak testing system and method making use of the above.



 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))

Maturation apparatus and methods

 The present invention relates to apparatus and methods for use in maturation processes, and in particular apparatus and methods for reducing fluid loss from a cask during a maturation process, and apparatus and methods for controlling and for monitoring a maturation process. A particular embodiment provides a vessel to sealably enclose the cask and provide an expansion volume to receive fluid vapour from the cask.

## Background to the invention

Scotch malt whisky production involves several stages, the most important of which is arguably the maturation process by which new-make whisky is matured for several years in wooden casks.

Whisky is typically ~60% water, ~40% ethanol (and ~0.1% other constituents), when it is casked, but during the maturation process (which typically takes ten to twenty years) a proportion of the fluid volume in the cask is lost to the atmosphere. This is affectionately referred to in the trade as the "angels' share".

2

1 The angels' share is, in Scotland, typically around 2% volume per annum. Elsewhere in the world the loss can be as high as 5% per annum. Some whisky producers may have 2 tens of millions of whisky casks undergoing maturation at any one time so these losses are 3 4 clearly significant. 5 In fact, the angels' share is reported to cost on the order of 10-15% of the production cost. 6 7 It is therefore desirable to reduce or prevent this lost volume of product. Experiments have 8 been conducted in which casks have been shrink-wrapped; however while fluid loss is 9 eliminated (or significantly reduced) there is a corresponding elimination (or significant reduction) in air ingress which is believed to negatively affect the maturation process and 10 11 hence the taste of the final product. 12 13 Wines, cognacs, armagnacs, sherries, ports, whiskeys (e.g. Bourbon) and beers may also be matured in barrels (as may balsamic vinegar), and the angels' share loss problem is 14 also known to affect these maturation processes (to lesser or greater extents). This is 15 therefore a wide reaching problem, and a solution that at least partially solves the problem 16 17 will provide major economic benefits. 18 19 In view of the foregoing, it is an object of at least one embodiment of an aspect of the present invention to provide an apparatus that can reduce or prevent fluid loss during a 20 21 maturation process, and a corresponding method. 22 23 It is also an object of at least one embodiment of an aspect of the present invention to 24 provide a system for monitoring and/or controlling a maturation process, and a 25 corresponding method.

PCT/GB2012/051621

3

## Summary of the invention

WO 2013/008003

1 2

3 According to a first aspect of the invention, there is provided an apparatus to reduce fluid

4 loss from a cask during a maturation process, the apparatus comprising a vessel to

sealably enclose the cask and provide an expansion volume to receive fluid vapour from

the cask.

7

5 6

8 In conventional maturation processes, whisky casks (for example) leak ethanol vapour to

9 the surrounding environment. By sealably enclosing the whisky cask and providing an

10 expansion volume, ethanol vapour is initially able to leave the cask until the partial

11 pressure of ethanol vapour (or other fluid lost from the cask) within the vessel reaches an

equilibrium value at which there will be no further leakage.

13 14

Preferably, the apparatus further comprises a monitoring system arranged to monitor the

presence of fluid vapour within the vessel.

16 17

The monitoring system permits monitoring of the leak rate as a function of time.

18 19

Preferably, the monitoring system comprises a light source and a detector, the detector

arranged to receive light from the light source and the monitoring system configured to

determine a relative transmission of the light through the vessel.

2223

20

21

Preferably, the apparatus comprises at least one aperture in a wall of the vessel, the light

source and detector arranged on opposite sides of the aperture. Preferably, the apparatus

comprises two apertures located in walls of the vessel and defining an optical path through

the vessel intersecting the light source and the detector. Alternatively, the apparatus

27 further comprises a mirror arranged to receive and reflect light from the light source to the

detector via a same aperture. Preferably, the at least one aperture comprises a window.

For example, the window may comprise calcium fluoride (CaF<sub>2</sub>).

29 30

34

28

31 Preferably, the light source comprises a laser source. Most preferably, the light source

32 comprises an infrared laser source. Optionally, the light source comprises an optical

33 parametric oscillator mid-infrared source. Alternatively, the light source comprises a

quantum cascade laser source. The light source may be tuned to an absorption line or

WO 2013/008003

33

34

in the vicinity of the cask.

4

PCT/GB2012/051621

absorption band of ethanol. Alternatively, or additionally, the monitoring system comprises 1 2 an active infrared hyperspectral imaging system. 3 4 Advantageously, the vessel comprises a lid. The lid and/or main body of the vessel may be provided with a rubber gasket or O-ring to provide a seal there between. Preferably, 5 6 the vessel is rectangular. Alternatively, the vessel is cylindrical or cask-shaped. 7 8 Optionally, the vessel is sized to receive a plurality of casks. Alternatively, or additionally, 9 the vessel is in fluid communication with one or more like vessels with or without 10 respective monitoring systems. 11 According to a second aspect of the invention, there is provided a method of reducing fluid 12 13 loss from a cask during a maturation process, the method comprising sealing the cask within a vessel having an expansion volume, and receiving fluid vapour from the cask in 14 15 the expansion volume of the vessel. 16 17 Preferably, the method further comprises monitoring the presence of ethanol within the 18 sealed vessel. Most preferably, the method further comprises monitoring the presence of 19 water vapour within the sealed vessel. 20 21 Preferably, the method comprises obtaining a background measurement of the presence 22 of ethanol within the sealed vessel. Preferably, the method comprises recording the 23 presence of ethanol within the sealed vessel as a function of time. 24 25 Embodiments of the second aspect of the invention may include one or more features 26 corresponding to features of the first aspect of the invention or its embodiments, or vice 27 versa. 28 According to a third aspect of the invention there is provided a monitoring system to 29 30 monitor fluid loss from a cask during a maturation process, the monitoring system comprising a light source and a detector, the detector arranged to receive light from the 31 32 light source and the monitoring system arranged to determine the presence of fluid vapour

5

WO 2013/008003

PCT/GB2012/051621

1 Preferably, the light source comprises a laser source. Preferably, the light source 2 comprises an optical parametric oscillator mid-infrared source. Alternatively, the light 3 source comprises a quantum cascade laser source. The light source may be tuned to an 4 absorption line or absorption band of ethanol. 5 6 Embodiments of the third aspect of the invention may include one or more features 7 corresponding to features of the first or second aspects of the invention or its 8 embodiments, or vice versa. 9 10 According to a fourth aspect there is provided a method of monitoring fluid loss from a cask during a maturation process, the method comprising emitting light from a light source, 11 receiving light from the light source at a detector, and determining a relative transmission 12 13 of the light in the vicinity of the cask. 14 15 Preferably, the method comprises locating one or both of the light source and detector 16 proximal to the cask. 17 18 Embodiments of the fourth aspect of the invention may include one or more features 19 corresponding to features of the first, second or third aspects of the invention or its 20 embodiments, or vice versa. 21 22 According to a fifth aspect of the invention, there is provided a system for controlling a 23 maturation process, the system comprising the apparatus of the first aspect and a control 24 system configured to control environmental conditions within the sealed vessel. 25 26 Preferably, the vessel is sized to provide sufficient atmospheric oxygen for at least a 27 portion of the maturation process. Alternatively, or additionally, the vessel is sized to 28 provide sufficient water vapour for at least a portion of the maturation process. 29 30 Preferably, the system comprises at least one pump operable to control an ambient 31 pressure within the vessel. Optionally, the pump is operable to maintain a target ambient 32 pressure within the vessel. 33

Optionally, the system comprises one or more gas sources in fluid communication with the vessel, operable to control the presence of one or more gases within the vessel.

1	
2	Preferably, the system further comprises one or more heating and/or cooling devices
3	configured to control a temperature of the vessel.
4	
5	Embodiments of the fifth aspect of the invention may include one or more features
6	corresponding to features of the first to fourth aspects of the invention or its embodiments,
7	or vice versa.
8	
9	According to a sixth aspect of the invention, there is provided a method of controlling a
10	cask maturation process, comprising sealing the cask within a vessel, and controlling
11	environmental conditions within the sealed vessel.
12	
13	Preferably, the method comprises controlling the temperature within the vessel.
14	
15	Preferably, the method comprises controlling the atmospheric pressure within the vessel.
16	
17	Optionally, the method comprises adding one or more gases to the vessel.
18	
19	Advantageously, the method comprises adding one or more substances selected to
20	simulate a geographic location during maturation.
21	
22	Advantageously, the method comprises maintaining a positive atmospheric pressure within
23	the vessel. Additionally, or alternatively, the method comprises controlling a partial
24	pressure of water vapour within the vessel to control the relative loss of water versus
25 26	ethanol from the cask.
27	Optionally, the method comprises periodically purging the vessel. This permits further gas
28	exchange between the cask and the expansion volume in the vessel, most preferably after
29	the equilibrium condition is reached.
30	and equilibrium contained to reached.
31	Embodiments of the sixth aspect of the invention may include one or more features
32	corresponding to features of any of the first to fifth aspects of the invention or its
33	embodiments, or vice versa.
34	

7

1 According to a seventh aspect of the invention, there is provided a cask leak testing 2 system, comprising the apparatus of the first aspect, the system of the third aspect, or the 3 system of the fifth aspect. 4 Optionally, the system comprises imaging means configured to obtain one or more images 5 of the cask to identify the location and size of one or more leaks in the cask. Optionally, 6 7 the imaging means comprises an active infrared hyperspectral imaging system. 8 9 Embodiments of the seventh aspect of the invention may include one or more features corresponding to features of any of the first to sixth aspects of the invention or its 10 11 embodiments, or vice versa. 12 13 According to an eighth aspect of the invention, there is provided a method of testing a cask for leaks, comprising sealing the cask within a vessel of the first aspect and employing a 14 monitoring system of the third aspect. 15 16 17 Preferably, the method comprises filling the cask with a test gas, and detecting the 18 presence of the test gas within the expansion volume of the vessel using the monitoring 19 system. 20 21 Embodiments of the eighth aspect of the invention may include one or more features 22 corresponding to features of any of the first to seventh aspects of the invention or its 23 embodiments, or vice versa.

8

### Brief description of the drawings 1 2 There will now be described, by way of example only, various embodiments of the 3 invention with reference to the drawings, of which: 4 5 6 Figure 1 illustrates in schematic form an apparatus to monitor and control whisky 7 maturation process in accordance with an aspect of the present invention; and 8 Figure 2 is a graph of relative transmission of a light beam directed through the apparatus 9 illustrated in Figure 1 versus time, during the initial stages of a maturation process, in 10 accordance with an aspect of the present invention. 11

PCT/GB2012/051621

## Detailed description of preferred embodiments

1

3 The following example is described in the context of the maturation of whisky within a

4 whisky cask, however it will be understood that the invention finds utility in other

5 maturation processes; for example of wine, cognac, armagnac, sherry, port, whiskey (e.g.

9

6 Bourbon), beer and balsamic vinegar. Furthermore, while wooden casks are typically

employed it is understood that casks made from other materials (such as plastics or

metals as increasingly used in wine maturation) shall not fall outside the scope of

protection set out herein.

WO 2013/008003

10 11

12 13

14

15

7

8

9

process, comprising a vessel 3 to house a whisky cask 5. The vessel 3 is rectangular box, in this embodiment made of aluminium, and comprises a lid 7 to provide access to the vessel. The lid 7 is held in place using screws and has a rubber gasket (not shown) such that when the lid 7 is screwed down the vessel 3 is sealed. The vessel provides an

Figure 1 illustrates an apparatus 1 for monitoring and controlling the whisky maturation

expansion volume (i.e. the internal volume not occupied by the cask 5) into which vapour

17 (e.g. ethanol vapour) from the cask may expand. As will be demonstrated below,

experimental results show that this prevents further fluid loss from the cask once an

equilibrium condition is reached.

19 20

21

22

18

Two apertures 9a, 9b are provided at opposing ends of the vessel, defining an optical path through the vessel. The apertures are sealed by way of calcium fluoride (CaF<sub>2</sub>) windows affixed thereto, although any suitable material for the windows may be used.

232425

26 27

28

29

30 31 Light from an infrared laser source 11, in this example a mid-infrared optical parametric oscillator source outputting 70mW at approximately 3306nm (although it will be readily apparent that any other suitable infrared light source may be employed), is directed through the apertures to a detector 13. This particular wavelength coincides with the O-H and C-H stretch absorption bands of ethanol, and accordingly transmission through the vessel gives an indication of the presence of ethanol within the vessel. The detector 13, in this case a laser power meter, is connected to a data logger (not shown), for example a PC with a suitable data acquisition card, to record transmitted power as a function of time.

32 33

34

35

In use, a background level for determining relative transmission or absorption is obtained without the cask 5 present in the vessel 3, although the background measurement could

34

35

WO 2013/008003 PCT/GB2012/051621

10

1 be taken immediately after the cask 5 is placed in the vessel 3 (before or after the lid 7 is 2 in place) before any significant ethanol leakage occurs. Subsequently, the lid 7 is secured in place, creating a seal. As noted above, ethanol will leak out of the cask 5 in the form of 3 4 ethanol vapour, which results in absorption of the laser light within the vessel 3. This absorption is detected by way of a reduction in optical power through the vessel 3, 5 6 detected by the detector 13. 7 8 It is envisaged that an alternative embodiment of the invention comprises the laser source 9 and power meter housed inside the vessel, in which case the apertures are not required. It is also envisaged that the monitoring system of the invention may be employed as a 10 stand-alone monitoring system separate from a vessel, and employed to monitor fluid loss 11 12 from a cask by detecting the presence of, say, ethanol vapour proximal to the cask. 13 14 Figure 2 illustrates an exemplary graph 21 of relative transmission 23 as a function of time, 15 illustrating the leak rate of ethanol vapour from a test cask (5) housed within a vessel. 16 Relative transmission is determined by dividing the measured optical power at time *t* with 17 the background measurement obtained at the start of the process. 18 19 The applicant has noted that the leak rate decays over time, tending towards a plateau or 20 continuum value at which leakage from the cask will cease. Extrapolation of a fit to the 21 experimental data, indicated by reference number 25, in this example indicates that 22 equilibrium would be reached within two to three days. At this stage the leakage from the 23 cask has stopped and the amount of ethanol vapour is fixed. 24 25 It is envisaged that instead of two apertures providing a pass-through, a single aperture may be provided with a retro-reflecting mirror inside the vessel – for example on the 26 27 opposing side of the vessel from the single aperture. The advantage would be two-fold; 28 the absorption pathlength (and hence, sensitivity) would be doubled and the vessel could 29 be monitored from one side. 30 31 The whisky cask 5 is shown sitting horizontally within the vessel, however it will be understood that the cask 5 may sit vertically or at any other orientation. Furthermore, 32 while the vessel 3 is illustrated as being a rectangular box, any other suitable shape of 33

container may be employed. For example, it may be useful to be cylindrical or cask-

shaped in order to conform to existing storage facilities. Furthermore, while the vessel of

this exemplary embodiment has been described as comprising aluminium, any other
 suitable material may be employed.

Of course a vessel may be sized to accommodate multiple whisky casks; a particular advantage being that the maturation process can be monitored and controlled for all said whisky casks at once, thus improving consistency of product between casks. Alternatively, several vessels could be linked by conduits such that conditions are shared throughout, whereupon a single one of said vessels could be monitored as described herein in the knowledge that conditions within or changes made to that vessel will correspond with or result in corresponding changes in conditions in the linked vessels.

While the exemplary embodiment has been described as monitoring the presence of ethanol within the vessel volume using a laser and a power meter, other useful information may be gleaned by employing a spectrometer or spectrophotometer to analyse the atmospheric composition within the vessel. The spectrometer may be of any suitable kind, for example a tuneable diode laser absorption spectrometer or an active infrared hyperspectral imaging system such as the Applicant's intra-cavity optical parametric oscillator based system. Thus, a detailed analysis of the composition of the atmosphere within the vessel might be determined in real-time.

The foregoing description of the invention provides an apparatus and a method that first and foremost prevents fluid loss from the cask once an equilibrium position has been reached.

By addition of a monitoring means, such as a light source and corresponding detector, the apparatus and method also allows the level of ethanol vapour leaking from the cask to be monitored. Optionally, the atmospheric composition can also be determined. The cask is also protected from external influences such as airborne pollutants.

Furthermore, the invention provides for a system and a method for controlling the maturation process. It has been found that simply sealing casks, for example by shrink-wrapping, does eliminate fluid loss however it also affects the maturation process because ingress of air into the cask from the surroundings is also prevented. This is expected to be of significant detriment to the taste of the whisky – whisky should be allowed to "breathe" as it matures.

WO 2013/008003

33 34

35

process.

12

PCT/GB2012/051621

1 2 To remedy this issue, the vessel provides an expansion volume. Furthermore, environmental conditions within the vessel 3, and particularly the expansion volume, can 3 4 be controlled throughout the maturation period. The system comprises a control system that allows the pressure within the vessel 3 to be controlled, as well as the relative 5 humidity and atmospheric composition and of course temperature. This control may 6 7 comprise maintaining the same environmental conditions throughout an entire maturation 8 process or alternatively varying the environmental conditions as required. By controlling 9 the environmental conditions the maturation process of the whisky can also be controlled. 10 It is envisaged that substances may be added to the vessel during the maturation process 11 12 to simulate desirable atmospheric conditions. For example, salt water could be injected to 13 simulate the sea air of a shore-side maturation location. 14 15 In a particular embodiment of the invention, the control system is used to maintain a small positive atmospheric pressure within the sealed vessel. Accordingly, the fluid loss from 16 17 the cask resulting from the angels' share is minimised while still allowing for air (oxygen) 18 ingress into the cask to allow the whisky to mature properly. 19 20 As an extension of this embodiment, the vessel may be sized to provide sufficient 21 atmospheric oxygen for an entire maturation process or portion thereof. As described 22 above, the partial pressure of ethanol within the vessel resulting from the angels' share will 23 plateau over the course of, say, a few days yet proper oxygenation is provided for over the 24 course of a number of years. If necessary, the vessel can be purged and refilled every few 25 years. Even with regular purging, the anticipated loss to the angels' share will be significantly reduced over the entire maturation process. 26 27 28 By way of example, it is found that the ratio of water loss to ethanol loss from a cask is dependent on the prevailing atmospheric conditions; principally relative humidity and 29 temperature, although other conditions may also have an effect. For example, higher 30 31 temperatures are found to increase losses of both ethanol and water. Higher humidity results in increased ethanol loss (relative to water) and lower humidity results in increased 32

water loss (relative to ethanol). The system described herein allows these conditions

(temperature and relative humidity) to be controlled, thus controlling the maturation

 One particular use for this system will be in the event that testing during the maturation process reveals some issue with the whisky that can be remedied by varying the atmospheric conditions. For example, if it was deemed that increased water loss was required, relative humidity could be reduced. In this way, a specific ethanol content can be targeted, particularly during the final stages of the maturation process. This may, for example, be used to increase alcohol content of whisky or to reduce the alcohol content of wine in the final product – as is often required in the industry.

A further application of the present invention is to detect leaks in a cask, whereby the cask is inserted into the vessel and leakage into the vessel monitored as a function of time to determine the presence of a leak and quantify the extent of the leak. This is preferably carried out before filling with whisky – for example by filling with a test gas. It is envisaged that imaging systems (such as the applicant's active infrared hyperspectral imaging system) may be employed to identify the location and/or the size of any leaks in the cask.

The invention provides an apparatus and a method that reduces fluid loss from a cask during a maturation process by sealably enclosing the cask in a vessel that provides an expansion volume to receive fluid vapour from the cask, a monitoring system and a method that monitors fluid loss from a cask during a maturation process using a light source and a detector to determine the presence of fluid vapour in the vicinity of the cask, a corresponding system for controlling a maturation process in which environmental conditions are controlled, and a cask leak testing system and method making use of the above.

Various modifications may be made within the scope of the invention as herein intended, and embodiments of the invention may include combinations of features other than those expressly claimed. For example, and as stated above, while specific examples are described in relation to the maturation of whisky in casks, similar apparatus, methods and systems may be employed in the maturation of bourbon and other spirits, wines, other alcoholic beverages, and other fluids (e.g. balsamic vinegar) that are matured in casks.

14

WO 2013/008003 PCT/GB2012/051621

1	C	lai	m	S

2

5

6

9

10

An apparatus to reduce fluid loss from a cask during a maturation process, the
 apparatus comprising:

a vessel to sealably enclose the cask and provide an expansion volume to receive fluid vapour from the cask; and

7 a monitoring system arranged to monitor the presence of the fluid vapour within the 8 vessel;

wherein the monitoring system comprises a light source and a detector, the detector arranged to receive light from the light source and the monitoring system configured to determine a relative transmission of the light through the vessel.

11 12

The apparatus according to claim 1, wherein the monitoring system is configured to monitor fluid vapour leak rate as a function of time.

15 16

3. The apparatus according to claim 1 or claim 2, wherein the apparatus comprises at least one aperture in a wall of the vessel, the light source and detector arranged on opposite sides of the aperture.

18 19 20

21

17

4. The apparatus according to claim 1 or claim 2, wherein the apparatus comprises two apertures located in walls of the vessel and defining an optical path through the vessel intersecting the light source and the detector.

222324

25

5. The apparatus according to claim 1 or claim 2, wherein the apparatus further comprises a mirror arranged to receive and reflect light from the light source to the detector via a same aperture.

2627

28 6. The apparatus according to any of claims 3 to 5, wherein the at least one aperture comprises a window.

30 31

7. The apparatus according claim 6, wherein the window comprises CaF<sub>2</sub>.

32

33 8. The apparatus according to any preceding claim, wherein the light source comprises a laser source.

35

36 9. The apparatus according to any preceding claim, wherein the light source comprises an infrared laser source.

WO 2013/008003

PCT/GB2012/051621

1 2 10. The apparatus according to any preceding claim, wherein the light source comprises 3 an optical parametric oscillator mid-infrared source. 4 5 11. The apparatus according to any of claims 1 to 9, wherein the light source comprises a quantum cascade laser source. 6 7 8 12. The apparatus according to any preceding claim, wherein the light source is tuned to 9 an absorption line or absorption band of ethanol. 10 13. The apparatus according to any preceding claim, wherein the monitoring system 11 12 comprises an active infrared hyperspectral imaging system. 13 14 14. The apparatus according to any preceding claim, wherein the vessel comprises a lid. 15 The apparatus according to claim 14, wherein the lid and/or main body of the vessel 16 15. 17 is provided with a rubber gasket or O-ring to provide a seal there between. 18 16. The apparatus according to any preceding claim, wherein the vessel is rectangular. 19 20 21 17. The apparatus according to any of claims 1 to 15, wherein the vessel is cylindrical or 22 cask-shaped. 23 24 18. The apparatus according to any preceding claim, wherein the vessel is sized to 25 receive a plurality of casks. 26 27 19. The apparatus according to any preceding claim, wherein the vessel is in fluid 28 communication with one or more like vessels with or without respective monitoring 29 systems. 30 31 A method of reducing fluid loss from a cask during a maturation process, the method 20. 32 comprising: 33 sealing the cask within a vessel having an expansion volume, and receiving fluid 34 vapour from the cask in the expansion volume of the vessel; monitoring the presence of the fluid vapour within the vessel using a light source and 35 a detector, the detector arranged to receive light from the light source; and 36 37 determining a relative transmission of the light through the vessel.

38

1 21. The method according to claim 20, further comprising monitoring the presence of ethanol within the sealed vessel.

16

3

The method according to claim 20 or claim 21, further comprising monitoring the presence of water vapour within the sealed vessel.

6

7 23. The method according to any of claims 20 to 22, further comprising obtaining a background measurement of the presence of ethanol within the sealed vessel.

9

10 24. The method according to any of claims 20 to 23, further comprising recording the presence of ethanol within the sealed vessel as a function of time.

12

13 25. A system for controlling a maturation process, the system comprising the apparatus 14 of any of claims 1 to 19 and a control system configured to control environmental 15 conditions within the sealed vessel.

16

17 26. The system according to claim 25, wherein the vessel is sized to provide sufficient 18 atmospheric oxygen for at least a portion of the maturation process.

19

27. The system according to claim 25 or claim 26, wherein the vessel is sized to provide sufficient water vapour for at least a portion of the maturation process.

22

23 28. The system according to any of claims 25 to 27, wherein the system comprises at least one pump operable to control an ambient pressure within the vessel.

25

26 29. The system according to claim 28, wherein the pump is operable to maintain a target ambient pressure within the vessel.

28

30. The system according to any of claims 25 to 29, wherein the system comprises one or more gas sources in fluid communication with the vessel, operable to control the presence of one or more gases within the vessel.

32

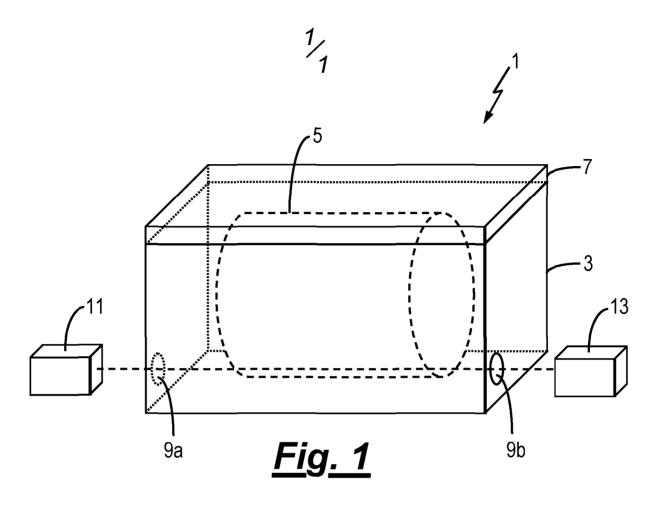
31. The system according to any of claims 25 to 30, wherein the system further comprises one or more heating and/or cooling devices configured to control a temperature of the vessel.

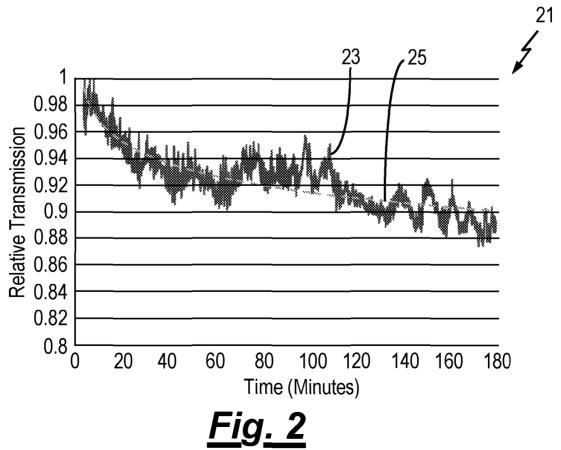
1	32.	A method of controlling a cask maturation process, comprising sealing the cask
2		within a vessel, controlling environmental conditions within the sealed vessel, and
3		monitoring the presence of the fluid vapour within the vessel using a light source and
4		a detector, the detector arranged to receive light from the light source; and
5		determining a relative transmission of the light through the vessel.
6		
7	33.	The method according to claim 32, further comprising controlling the temperature
8		within the vessel.
9	24	The method according to plaim 22 or plaim 22 further comprising controlling the
10 11	34.	The method according to claim 32 or claim 33, further comprising controlling the atmospheric pressure within the vessel.
12		aunospheric pressure within the vessel.
13	35.	The method according to any of claims 32 to 34, further comprising adding one or
14		more gases to the vessel.
15		
16	36.	The method according to any of claims 32 to 35, further comprising adding one or
17		more substances selected to simulate a geographic location during maturation.
18		
19	37.	The method according to any of claims 32 to 36, further comprising maintaining a
20		positive atmospheric pressure within the vessel.
21	00	TI
22	38.	The method according to any of claims 32 to 37, further comprising controlling a
23		partial pressure of water vapour within the vessel to control the relative loss of water
24		versus ethanol from the cask.
25 26	39.	The method according to any of claims 32 to 38, further comprising periodically
27	00.	purging the vessel.
28		parging the vessel.
29	40.	A cask leak testing system, comprising the apparatus of any of claims 1 to 19, or the
30		system of any of claims 25 to 31.
31		
32	41.	The cask leak testing system according to claim 40, further comprising imaging
33		means configured to obtain one or more images of the cask to identify the location
34		and size of one or more leaks in the cask.
35		

1 42. The cask leak testing system according to claim 40, wherein the imaging means comprises an active infrared hyperspectral imaging system.

4 43. A method of testing a cask for leaks, comprising sealing the cask within a vessel of any of claims 1 to 19.

44. The method according to claim 43, further comprising filling the cask with a test gas, and detecting the presence of the test gas within the expansion volume of the vessel using the monitoring system.





SUBSTITUTE SHEET (RULE 26)

### INTERNATIONAL SEARCH REPORT

International application No PCT/GB2012/051621

A. CLASSIFICATION OF SUBJECT MATTER INV. C12H1/22 G01N21/35

ADD.

G01N33/14

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)

C12H G01N

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)

EPO-Internal, WPI Data, BIOSIS, FSTA, COMPENDEX

C. DOCUM	ENTS CONSIDERED TO BE RELEVANT	
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 001 877 A (SHAPIRO ZALMAN M) 26 September 1961 (1961-09-26) the whole document	1-44
A	FR 2 571 382 A1 (COGAT PIERRE OLIVIER [FR]) 11 April 1986 (1986-04-11) the whole document	1-44
A	US 2 943 940 A (WIEDEMANN CLAUS M) 5 July 1960 (1960-07-05) the whole document	1-44
А	US 2 865 770 A (NICKOL GORDON B) 23 December 1958 (1958-12-23) the whole document	1-44
	-/	

X Further documents are listed in the continuation of Box C.	X See patent family annex.
* Special categories of cited documents :	"T" later document published after the international filing date or priority
"A" document defining the general state of the art which is not considered to be of particular relevance	date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive
"L" document which may throw doubts on priority claim(s) or which is	step when the document is taken alone
cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is
"O" document referring to an oral disclosure, use, exhibition or other means	combined with one or more other such documents, such combination being obvious to a person skilled in the art
"P" document published prior to the international filing date but later than the priority date claimed	"&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
	05.440.40040
28 November 2012	05/12/2012
Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Koch, Jürgen

# **INTERNATIONAL SEARCH REPORT**

International application No
PCT/GB2012/051621

C(Continua	tion). DOCUMENTS CONSIDERED TO BE RELEVANT	
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Α	EP 0 305 973 A1 (YU QI HAI) 8 March 1989 (1989-03-08) page 3, line 26 - line 45	1-44
Α	EP 1 355 147 A2 (BIOTECHNOLOGIE KEMPE GMBH [DE]) 22 October 2003 (2003-10-22) the whole document	1-44
Α	US 5 473 161 A (NIX JOHN A [US] ET AL) 5 December 1995 (1995-12-05) abstract	1-44
A	EP 0 884 584 A1 (LAB OF MOLECULAR BIOPHOTONICS [JP]) 16 December 1998 (1998-12-16) abstract	1-44

# **INTERNATIONAL SEARCH REPORT**

Information on patent family members

International application No
PCT/GB2012/051621

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3001877 A	26-09-1961	NONE	
FR 2571382 A1	11-04-1986	NONE	
US 2943940 A	05-07-1960	NONE	
US 2865770 A	23-12-1958	NONE	
EP 0305973 A1	08-03-1989	CN 1031716 A EP 0305973 A1 US 4994289 A	15-03-1989 08-03-1989 19-02-1991
EP 1355147 A2	22-10-2003	AT 306664 T DE 10216653 A1 EP 1355147 A2 US 2003192368 A1	15-10-2005 06-11-2003 22-10-2003 16-10-2003
US 5473161 A	05-12-1995	NONE	
EP 0884584 A1	16-12-1998	EP 0884584 A1 JP 3090692 B2 US 5905030 A WO 9732198 A1	16-12-1998 25-09-2000 18-05-1999 04-09-1997