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

(19) World Intellectual Property Organization

International Bureau





(10) International Publication Number WO 2015/055743 A1

(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, BN, 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, IR, IS, JP, KE, KG, KN, KP, KR,

KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG,

MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM,

PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, 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.

(43) International Publication Date 23 April 2015 (23.04.2015)

(51) International Patent Classification: G01N 21/3504 (2014.01) G01N 33/14 (2006.01)

(21) International Application Number:

PCT/EP2014/072165

(22) International Filing Date:

15 October 2014 (15.10.2014)

(25) Filing Language:

English

(26) Publication Language:

English

EP

(30) Priority Data:

13188791.1 15 October 2013 (15.10.2013)

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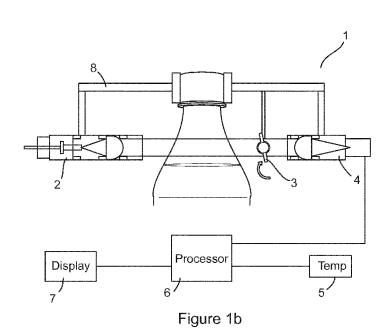
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- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE,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

Published:

with international search report (Art. 21(3))

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(54) Title: SYSTEM AND METHOD FOR DETERMINING THE LEVEL OF CARBON DIOXIDE DISSOLVED IN A LIQUID IN A SEALED CONTAINER



al system and method for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising a light source (2), an optical filter (3), and a detector (4). The detector is configured to receive a first optical transmission measurement at a first wavelength absorbed by CO₂ and a second optical transmission measurement at a second wavelength not affected by CO₂ from light irradiated from the light source through a headspace of the liquid in the sealed container along a single optical path. A processor is configured to process the first and second optical transmission measurements to calculate a ratio of the two transmission values, said ratio being proportional to the partial pressure of CO₂ in the headspace, and to calculate the level of CO₂ dissolved in the liquid from the ratio and an estimated temperature of the liquid.

(57) Abstract: The invention provides an optic-



PCT/EP2014/072165

Title

SYSTEM AND METHOD FOR DETERMINING THE LEVEL OF CARBON DIOXIDE DISSOLVED IN A LIQUID IN A SEALED CONTAINER

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The invention relates to a system and method for calculating carbon dioxide present in the headspace of a liquid in a sealed container.

Background

Carbonation is the process by which carbon dioxide (CO₂) is dissolved in water or an aqueous solution. The most commonplace application is for carbonated drinks including soft drinks and mineral water. Dissolved CO2 may also result from the production of the gas by micro-organisms during fermentation of the beverage, as in beer and champagne. CO₂ has a very high dissolution in water because it reacts with it, producing carbonic acid and thus lowering the pH. There is an equilibrium between the quantities of CO₂ in the surrounding air, those in solution, and the amounts of the various carbonic acid ionic forms (according to their dissociation constants), which depends on pH and therefore on the buffer effect of the beverage that is a function of its composition. The quality of such drinks is affected by the level of dissolved CO2 and the amount of Therefore an important quality control parameter for the carbonic acid. beverage industry is the level of dissolved CO2 in their drinks and therefore measurement of dissolved CO₂ levels are routinely carried out in practice.

25 The measurement of dissolved CO₂, apart from the quality of the beverage itself, is also of great interest to beverage manufacturers in terms of their packaging. PET plastic is the most common format for carbonated beverage containers and is the highest cost part of the beverage manufacturing process. PET is permeable, so CO₂ gradually escapes over time as the bottles are transported and stored on the shelf. As described above the level of dissolved 30 CO₂ is important for beverage quality and customer experience. Companies would ideally like to be able to instantaneously test the level of CO₂ present in the beverage on the shelf without sending it to a central laboratory. This gives

important information on the state of the beverage, as experienced by the customer, as well as the performance of the PET packaging. Such a direct CO₂ measurement system could be deployed by companies to track and potentially reduce the amount of PET used in their packaging, which would result in significant cost savings.

One type of CO₂ beverage measurement system available on the market presently is an optical based system and has been known for a long time. One optical solution is disclosed in US Patent 5,473,161, assigned to Coca Cola Inc., entitled "Method for testing carbonation loss from beverage bottles using IR spectroscopy". The system proposed in US5,473,161 is described as working under very controlled conditions with no temperature measurement where the beverage in bottles is expected to be in equilibrium or the bottles are filled with carbon dioxide only which makes the device inaccurate.

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Another type of measurement system is EP 2 620 761, assigned to Ft System S.r.l, discloses a system and method for measuring the quantity of carbon dioxide dissolved in a liquid contained in a closed container. The system disclosed uses a laser source that operates on a single wavelength measurement and assumes an ambient temperature when measuring the quantity of carbon dioxide dissolved in a liquid. However a problem with this approach is that the measurement system becomes unstable for changes in temperature. As a consequence measurements obtained are inaccurate for temperature variations. Another problem with the Ft System is that the system does not take account of the transparency or thickness of the walls for a particular container. Light path distortion produced by the higher than air refractive index of the container material can therefore make measurements inaccurate. In addition this system is not able to discriminate between different coloured containers with the result that the inaccuracy of the measurements is increased.

German Patent Publication number DE 10 2008 005 572, assigned to smartGAS Mikrosensorik GmbH, discloses a similar system described above.

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The method described in this publication involves designing filter elements according to spectral filtering such that the former filter element filters a radiation source in a spectral region where the presence or concentration of a gas is detected for an inline carbonated liquid or beverage. Two optical paths are provided in the form of single wavelength filters, however a similar problem exists where measurements are only accurate where the temperature is assumed to be ambient.

It is therefore an object to provide an improved system and method for measuring partial pressure of carbon dioxide present in the headspace of a liquid in a sealed container.

Summary

According to the invention there is provided, as set out in the appended claims, an optical system for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising:

a light source; an optical filter; and a detector, wherein the detector receives a first optical transmission measurement at a first wavelength and a second optical transmission measurement at a second wavelength from light irradiated from the light source through a headspace of the liquid in the sealed container along a single optical path;

a processor configured to process the first and second optical transmission measurements to calculate a ratio of the two transmission values, said ratio is proportional to the partial pressure of CO₂ in the headspace; and

said processor calculates the level of CO₂ dissolved in the liquid from the ratio and a measured or estimated temperature of the liquid.

An important aspect of the invention is that the system is configured to provide temperature stabilisation by preventing changes to the emission spectrum of the light source.

The invention provides a system, for example a standalone system or a portable handheld device, that is capable of testing the CO_2 level without opening or piercing the packaging which is in contrast to almost all current systems. The invention works particularly well for PET bottles and works on all colours of PET, in other words the PET does not have to be clear. The headspace can be defined as the gas above the liquid in the sealed container.

In one embodiment the system comprises a thermometer configure to measure the temperature of the liquid.

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In one embodiment the processor uses the calculated partial pressure and the estimated liquid temperature to calculate the level of dissolved CO_2 in units of CO_2 L / Liquid L in the liquid by employing Henry's Gas Law.

In one embodiment the first measurement comprises the response of the system to the level of CO₂ in the light path of the headspace and provides the value as a first voltage value.

In one embodiment the second measurement comprises the response of the system at a CO₂ off-resonance measurement position as a second voltage.

In one embodiment measurement of the level of CO_2 present in the headspace is achieved by recording the system's response on resonance at $2\mu m$ and off resonance at a wavelength which is unaffected by CO_2 .

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In one embodiment there is provided a clamp adapted to secure the container to provide a stable reference point before making any measurement.

In one embodiment the clamp is dimensioned to clamp around the neck of a standard PET drinks bottle.

In one embodiment the clamp uses a bottle cap, neck flange from the container preform or similar physical feature as a physical reference point to always clamp

around the neck of the container in the same location and ensure as a result that the measurement light path is the same for each or all container types.

In one embodiment the filter comprises a narrow bandpass filter.

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In one embodiment the thermometer comprises a thermocouple or IR thermometer.

In one embodiment the detector comprises a photo-detector. In one embodiment the light detector employed is a long wavelength extended InGaAs photodiode which provides linear output voltage response versus received light power. In one embodiment the photo-detector can be temperature stabilised in order to ensure the same linear response for different external temperatures and thereby reduce the level of thermal noise.

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In one embodiment the container comprises PET material or glass material.

In a further embodiment there is provided a method for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising the steps of:

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irradiating light from a light source through a headspace of the liquid in the sealed container and obtaining at a first optical transmission measurement at a first wavelength and a second optical transmission measurement at a second wavelength using a tuneable optical filter along at least one optical path;

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estimating the temperature of the liquid in the container;

processing the two measurement values to calculate ratio of the first and second optical transmission measurements, said ratio is proportional to the partial pressure of CO_2 in the headspace; and

calculating the level of CO₂ dissolved in the liquid from the ratio and the temperature of the liquid.

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In another embodiment there is provided a method for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising the steps of:

irradiating light from a light source through a headspace of the sealed liquid container and obtaining at least two measurement values;

estimating the temperature of the liquid in the container;

processing the two measurement values to calculate the ration of intensities of the two measurements that is proportional to the partial pressure of CO₂ in the headspace; and

calculating the level of CO₂ dissolved in the liquid from the ratio of the shared intensities and the temperature of the liquid.

In another embodiment there is provided an optical system for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising:

a light source; a filter; and a detector, wherein the detector receives at least two measurement values from light irradiated from the light source through a headspace of the liquid in the sealed container;

a thermometer configured to estimate the temperature of the liquid;

a processor configured to process the two measurement values to calculate the ratio of the two measurements that is proportional to the partial pressure of CO_2 in the headspace; and said processor calculates the level of CO_2 dissolved in the liquid.

In another embodiment there is provided a computer implemented system for calculating the level of CO₂ dissolved in a liquid stored in a sealed liquid container comprising:

a processor configured to process the first and second optical transmission measurements to calculate a ratio of the two transmission values, said ratio is proportional to the partial pressure of CO₂ in the headspace; and

said processor calculates the level of CO₂ dissolved in the liquid from the ratio and a measured or estimated temperature of the liquid.

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In another embodiment there is provided an optical system for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising:

a light source; an optical filter; and a detector, wherein the detector receives a first optical transmission measurement at a first wavelength and a second optical transmission measurement at a second wavelength from light irradiated from the light source through a headspace of the liquid in the sealed container along one or more optical paths; a processor configured to process the first and second optical transmission measurements to calculate a ratio of the two transmission values, said ratio is proportional to the partial pressure of CO₂ in the

said processor calculates the level of CO₂ dissolved in the liquid from the ratio and a measured or estimated temperature of the liquid.

There is also provided a computer program comprising program instructions for causing a computer program to carry out the above method which may be embodied on a record medium, carrier signal or read-only memory.

Brief Description of the Drawings

headspace; and

An embodiment will be more clearly understood from the following description of an embodiment thereof, given by way of example only, with reference to the accompanying drawings, in which:-

Figure 1a & 1b illustrates a plan view and side view of the optical system according to one aspect;

Figure 2a & 2b illustrates two flowcharts showing how the level of dissolved CO₂ is calculated according to one embodiment;

Figure 3 compares the measured transmission spectrum (by FT-IR) for an empty bottle with no CO_2 to that with CO_2 (partial pressure of 3bar), where the measured wavelengths for on and off CO_2 resonance are marked; and

Figure 4 shows the calibrated response of the system to increasing levels of CO_2 in a bottle headspace.

Detailed Description of the Drawings

The invention is an optical measurement system, based on the principle of midinfrared spectroscopy, to directly measure the level of dissolved CO_2 in a carbonated beverage, regardless of the beverage composition or alcohol content. The majority of chemical compounds have a response in the midinfrared spectrum (~3-15 μ m) to the fundamental vibrational modes of their molecular structure. Carbon Dioxide (CO_2) exhibits a number of absorption lines in the mid-infrared. The most commonly used absorption is found at 4.26 μ m (2349 cm⁻¹) and this is the wavelength that is typically used to measure gas phase CO_2 in the atmosphere using transmission spectroscopy. However, the aim of this system is to measure high partial pressures (~1-4 bar) within a closed volume. In this scenario, with the level of CO_2 present in the light path, the absorption at 4.26 μ m is so strong that it will not allow light to reach the detection system and hence render it useless.

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As a result another CO_2 absorption line must be used and in one embodiment 2.004µm (4990 cm⁻¹) can be chosen. Although there are other more suitable absorption lines (e.g. 2.779µm) it is the availability of light sources and technology at 2µm that makes it the best candidate for the operating wavelength. Additionally the absorption line at 2µm is very narrow in comparison to some of the other lines so it lends itself easily to making a comparison measurement of on and off resonance which is how the measurement is achieved according to one aspect of the invention.

System Hardware Embodiment

Referring now to Figure 1a &1b the system of the invention according to one embodiment is shown indicated generally by the reference numeral 1. The system comprises a light source2, an optical filter 3 and detector 4. In addition the system comprises a temperature measuring device 5 to estimate the temperature of a liquid in a container, for example a bottle, and a processor 6 to calculate the dissolved CO_2 in the liquid and display on a screen 7 or other means. A standard clamp device 8 is shown to clamp a container in place. In

the example shown a standard Coca Cola bottle containing a liquid is held in place by the clamp device 8. It will be appreciated that the invention can be employed to most containers and is particularly suitable to bottles containing a beverage, for example a carbonated beverage.

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In one realisation the light source is a Light-Emitting Diode (LED) centred at $2\mu m$ with a spectral width of approximately 100nm. Measurement of the level of CO_2 present in the headspace is achieved by recording the system's response on resonance at $2.004\mu m$ and off resonance at a wavelength which is unaffected by CO_2 at $2.004\mu m$. To ensure the fidelity of this measurement these on and off resonance wavelengths must ideally be close together with a similar transparency through PET such that any difference in absorption by the plastic will not unduly affect the measurement. To realise this on and off resonance arrangement an optical filter can be deployed to tune the wavelength of the LED (light source) as seen by the detector. This can be done a number of ways. The optical filter can be preferably a narrow bandpass filter, but it will be appreciated that other filters can be used to achieve the same function.

One realisation utilises two separate optical filters with different passbands which correspond to the two measurement wavelengths. Alternatively a single optical filter can be rotated about a single axis so as the change angle of incidence for the incoming light which will result in a change in the wavelengths transmitted by the filter. Only a single optical path is required in this realisation, although additional optical paths could be contemplated. In this way rotation of the filter allows the system to scan across the wavelength range of interest to make the comparison between on and off resonance. Finally the light is detected using a detector which has response in the 2µm range. A photodetector can be selected which has peak efficiency at approximately 2.2µm, or other suitable wavelength, and so is well suited to the application. The detector is preferably a photo-detector, for example an InGaAs photodiode. The photodetector is also preferably temperature stabilised in order to ensure the same linear response for different external temperatures and thereby reduce the level of thermal noise. It will be appreciated that there are associated electronics

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to drive the light source and detection circuits that are familiar to the skilled person in the art.

The temperature of the beverage must also be measured in order to calculate the level of dissolved CO₂ in the liquid, for example by a thermometer. A thermocouple in contact with the PET bottle surface to estimate the liquid temperature can be used but another approach would be to use any device that could measure the temperature. The contact method assumes that there is thermal equilibrium between the liquid and the PET wall. For example infrared thermometer with specific calibrated emissivity for the PET material can be used to estimate liquid temperature inside the container.

Measurement Method

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The system, as described above and with respect to Figure 1a and 1b, will produce two measurements. A first optical measurement at a first wavelength and a second optical measurement at a second wavelength is measured by the photodetector from light received from the light source through the headspace of the liquid in a sealed container along the same optical path, as shown in Figure 1b. The wavelengths can be set to measure at any suitable wavelength value or range. The thermometer is configured to estimate the temperature of the liquid. A processor is configured to process the first and second optical measurements to calculate a ratio of the two measurements.

The ratio of these two responses is proportional to the partial pressure of CO₂ in the headspace. The level of CO₂ dissolved in the liquid will depend on both this partial pressure and the temperature of the beverage. The system software uses both the calculated partial pressure and the estimated liquid temperature to calculate the level of dissolved CO₂ in units of CO₂ L / Liquid L by employing

Henry's Gas Law:

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At a constant temperature, the amount of a given gas that dissolves in a given type and volume of liquid is directly proportional to the partial pressure of that gas in equilibrium with that liquid.

Figure 2a & 2b illustrates a flowchart for the calculation of the CO_2 level in volumes of CO_2 . The two wavelengths measured by the system are illustrated in the spectra shown in Figure 3. A first optical measurement at a first wavelength and a second optical measurement at a second wavelength are measured and represented as voltage values V1 and V2. The ratio of these two values is calculated to determine the CO_2 partial pressure in the headspace to provide a value P_{CO_2} . The temperature of the liquid, measured by the thermometer, is applied as a function of P_{CO_2} and the dissolved CO_2 is calculated that can be subsequently displayed. The ambient temperature changes are not affecting system as all active optical elements can be temperature stabilised.

The spectra shown in Figure 3 were measured on a Fourier Transform Infra-Red (FT-IR) spectrometer with a PET bottle placed in the measurement path as is achieved with the system itself. As can be seen in Figure 3 the off-resonance response is measured at a wavelength where there is high PET transparency but no absorption by CO₂. The on resonance response is where there is high PET transparency coupled to a strong absorption by CO₂.

The performance of the system is illustrated in Figure 4 where the system measures, and quantifies in volumes of CO₂, the amount of CO₂ present in a sealed PET bottle with levels ranging from 1 to 5 bars with respect to gas pressure within the vessel.

Alternative Embodiment

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It will be appreciated that the combination of the heretofore described elements makes the system insensitive to external temperature variations. It will be appreciated that both LED and detector can be temperature controlled using

Peltier thermoelectric cooler (TEC). This ensures that the colour of the TEC LED source is conserved; the ratio of light intensity between wavelengths off and on CO2 resonance is conserved; the linearity of detector response is conserved and the detector sensitivity is conserved.

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In one embodiment the optical elements: the light source (LED), collimating and focusing lenses, tuneable optical filter and detector are fixed along a single axis (optical pathway). The distance of the optical pathway can be set as appropriate, depending on a number of factors, such as the size of a bottle neck.

It will be further appreciated that the electronics used to measure the detector response for light passing through the bottle at a wavelength off resonance, following this the filter is retuned and again the detector response is measured for light passing through the bottle at the on resonance wavelength. The ratio between the two responses is recalculated to the CO_2 partial pressure according to a specified calibration curve. The electronics also measures the temperature of the bottle below the headspace and sends to a processor, for example a PC, both the partial pressure of CO_2 and the bottle temperature. The PC recalculates these values to the CO_2 carbonation level in the liquid inside the bottle.

The embodiments in the invention described with reference to the drawings comprise a computer apparatus and/or processes performed in a computer apparatus. However, the invention also extends to computer programs, particularly computer programs stored on or in a carrier adapted to bring the invention into practice. The program may be in the form of source code, object code, or a code intermediate source and object code, such as in partially compiled form or in any other form suitable for use in the implementation of the method according to the invention. The carrier may comprise a storage medium such as ROM, e.g. CD ROM, or magnetic recording medium, e.g. a floppy disk or hard disk. The carrier may be an electrical or optical signal which may be transmitted via an electrical or an optical cable or by radio or other means.

In the specification the terms "comprise, comprises, comprised and comprising" or any variation thereof and the terms include, includes, included and including" or any variation thereof are considered to be totally interchangeable and they should all be afforded the widest possible interpretation and vice versa.

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The invention is not limited to the embodiments hereinbefore described but may be varied in both construction and detail.

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Claims

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1. An optical system for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising:

a light source; an optical filter; and a detector, wherein the detector receives a first optical transmission measurement at a first wavelength and a second optical transmission measurement at a second wavelength from light irradiated from the light source through a headspace of the liquid in a sealed liquid container along a single optical path;

a processor configured to process the first and second optical transmission measurements to calculate a ratio of the two transmission values, said ratio is proportional to the partial pressure of CO_2 in the headspace; and

said processor calculates the level of CO₂ dissolved in the liquid from the ratio and a measured temperature of the liquid.

- 2. The system of claim 1 wherein the processor uses a calculated partial pressure from the ratio and the estimated liquid temperature to calculate the level of dissolved CO₂ in units of CO₂ L / Liquid L in the liquid by employing Henry's Gas Law.
- 3. The system of claims 1 or 2 wherein the first optical transmission measurement comprises the response of the system at a CO₂ on resonance wavelength in the light path through the headspace and provides a value as a first voltage value.
- 4. The system of claims 1 to 3 wherein the second optical transmission measurement comprises the response of the system at a CO₂ off resonance wavelength in the light path through the headspace and provides a value as a second voltage value.
- 5. The system of any preceding claim wherein measurement of the level of CO₂ present in the headspace is achieved by recording the system's response on

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resonance at $2.004\mu m$ and off resonance at a wavelength which is unaffected by CO_2 at $2.004\mu m$.

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- 6. The system of any preceding claim comprising a clamp adapted to secure the container to provide a stable reference point before making any measurement.
 - 7. The system of claim 6 wherein the clamp is dimensioned to clamp around the neck of a standard PET drinks bottle.
- 8. The system of claim 6 wherein the clamp uses a bottle cap, neck flange from the container preform or similar physical feature as a physical reference point to always clamp around the neck of the container in the same location and ensure as a result that the measurement light path is the same for each or all container types.
 - 9. The system of any preceding claim wherein the light source comprises a broadband light emitting diode (LED) source
- 10. The system of claim 9 wherein the broadband LED source is configurable to emit light at different wavelengths.
 - 11. The system of any preceding claim wherein the optical filter is tuneable to allow different wavelengths of light to transmit across the same optical path.
 - 12. The system of any preceding claim wherein the optical filter comprises a narrow bandpass filter.
- 13. The system of any preceding claim comprising a thermometer to measure
 the temperature of the liquid wherein the thermometer comprises a
 thermocouple or IR thermometer.
 - 14. The system of any preceding claim wherein the detector comprises a photodetector.

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15. The system of claim 14 wherein the photo-detector comprises an InGaAs photodiode and configured to provide a linear output voltage response versus received light power.

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- 16. The system of any preceding claim wherein, when the system is in use, the sealed liquid container comprises PET material or glass material.
- 17.A hand-held device comprising the optical system of any preceding claim.

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18.A method for calculating the level of CO₂ dissolved in a liquid stored in a sealed container comprising the steps of:

irradiating light from a light source through a headspace of the liquid in a sealed container and obtaining at a first optical transmission measurement at a first wavelength and a second optical transmission measurement at a second wavelength using a tuneable optical filter along a single optical path;

estimating the temperature of the liquid in the container;

temperature of the liquid.

processing the two measurement values to calculate ratio of the first and second optical transmission measurements, said ratio is proportional to the partial pressure of CO_2 in the headspace; and calculating the level of CO_2 dissolved in the liquid from the ratio and the

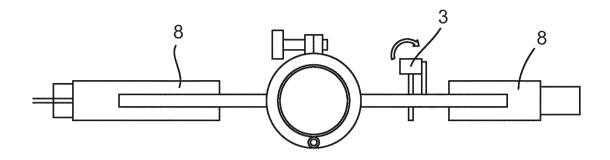


Figure 1a

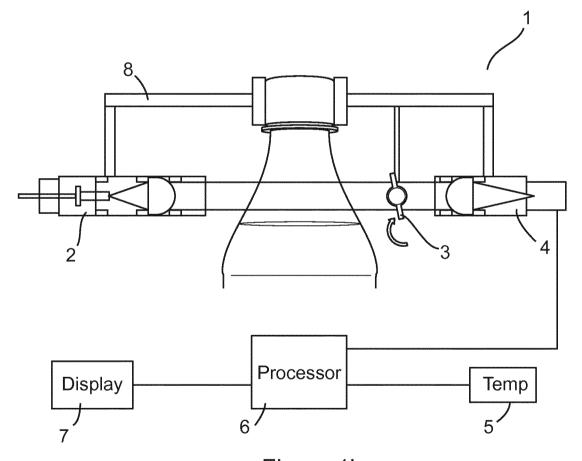


Figure 1b

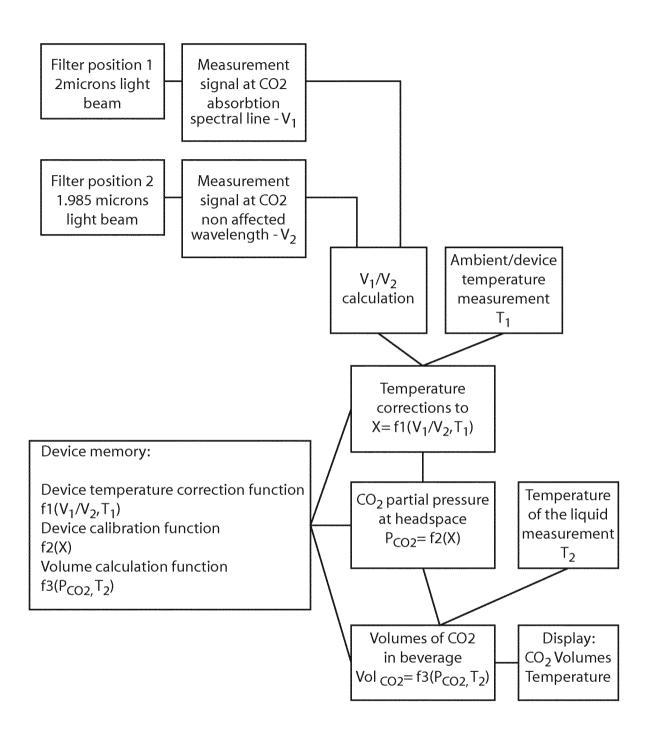


Figure 2a

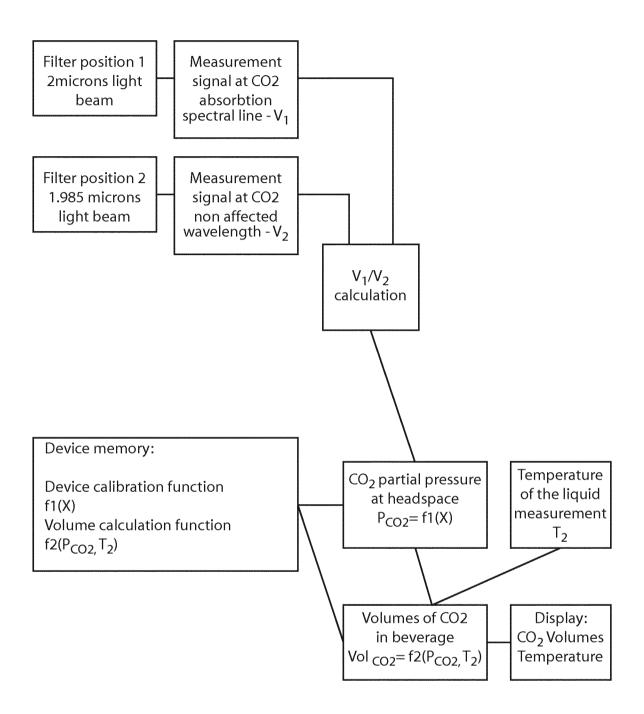


Figure 2b

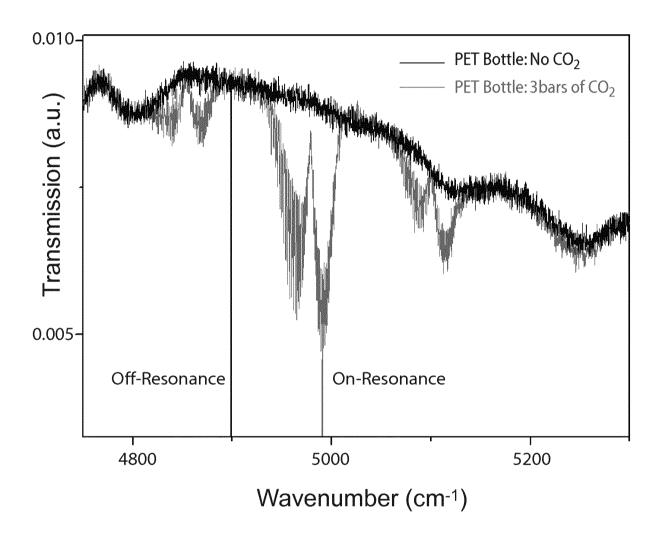


Figure 3

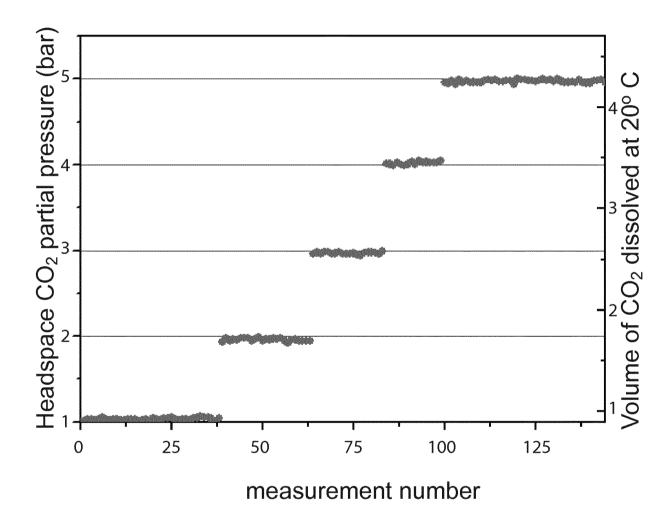


Figure 4

INTERNATIONAL SEARCH REPORT

International application No PCT/EP2014/072165

A. CLASSIFICATION OF SUBJECT MATTER INV. G01N21/3504 G01N33/14 ADD.

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

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

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Υ	DE 10 2004 063667 A1 (OPTOTRANSMITTER UMWELTSCHUTZ T [DE]) 13 July 2006 (2006-07-13) paragraphs [0001] - [0006], [0009] - [0011], [0013], [0022]; figure 6	1-18
Υ	EP 2 522 984 A2 (SCHLUMBERGER TECHNOLOGY BV [NL]; SCHLUMBERGER SERVICES PETROL [FR]; PR) 14 November 2012 (2012-11-14) paragraph [0029]	1-10, 12-17
Y	US 2010/290045 A1 (SAPTARI VIDI A [US]) 18 November 2010 (2010-11-18) paragraph [0044]; figure 3	1-18

X Further documents are listed in the continuation of Box C.	X See patent family annex.		
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filling 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 filling 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 9 December 2014	Date of mailing of the international search report 18/12/2014		
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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2014/072165

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