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(71) Applicant *(for all designated States except US)*: **THE BOARD OF REGENTS FOR OKLAHOMA STATE UNIVERSITY** [US/US]; 203 Whitehurst, Oklahoma State University, Stillwater, OK 74078 (US).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: **MATERER, Nicholas** [US/US]; 1810 W. 4th Street, Stillwater, OK 74074 (US). **APBLETT, Allen** [US/US]; 2413 Coyote Run, Stillwater, OK 74075 (US). **SCOTT, Dane** [US/US]; 1807 N. Hartford Street, Apt. 522, Stillwater, OK 74075 (US).

(74) Agent: **WEEKS, Alan, R.**; Fellers, Snider, Blankenship, Bailey & Tippens, PC, 321 South Boston, Suite 800, Tulsa, OK 74103-3318 (US).

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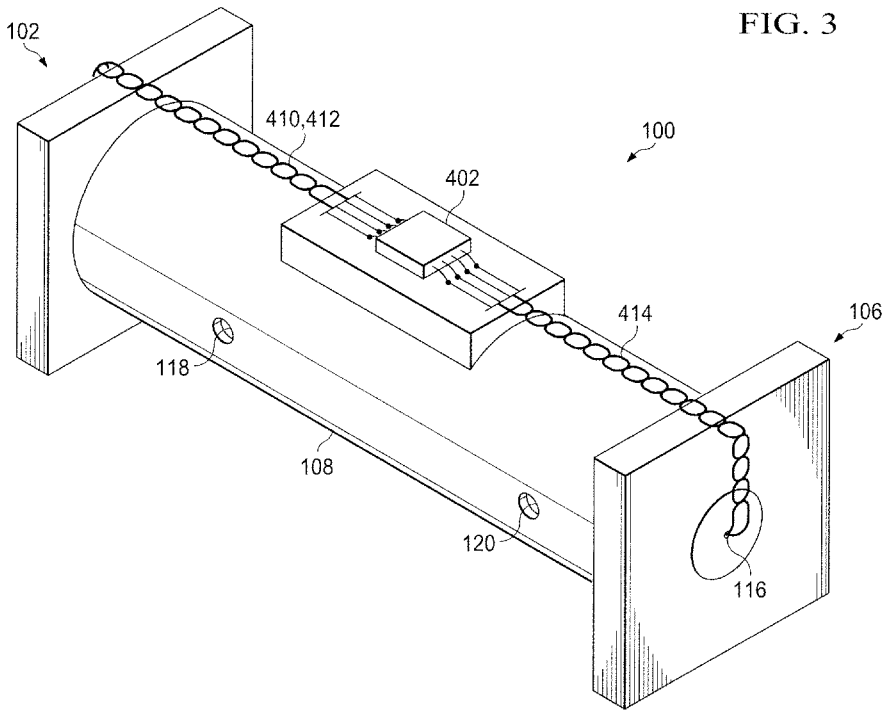
[Continued on next page]

(54) Title: CHLORINE DIOXIDE SENSOR

FIG. 3



(57) Abstract: A chemical sensor is disclosed. The sensor has a test chamber for receiving chemicals in a gaseous state, the test chamber having two substantially transparent windows at first and second ends of the test chamber. The sensor uses a pulse operated ultraviolet light emitting diode at the first end of the test chamber emitting at a wavelength close to a maximum in the absorption band of a test chemical, and an electromagnetic sensor at a second end of the test chamber, the sensor being sensitive to the light emitted by the light emitting diode.

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Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

Published:

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

CHLORINE DIOXIDE SENSOR

FIELD OF THE INVENTION

This disclosure relates to the field of noxious gas detection, and more specifically, to detection of gaseous chlorine dioxide.

BACKGROUND OF THE INVENTION

Currently available commercial sensors exist for monitoring or measuring the concentration of chlorine dioxide. However, many of these sensors are developed for regulatory applications which require measurements at very low concentration levels (e.g., parts per billion). At higher concentrations and long exposure durations, existing electrochemical based sensors can be damaged such that they will become inaccurate or inoperative. Additionally, for some applications, the sensor needs to be able to operate accurately at high relative humidity.

What is needed is a system and method for addressing the above, and related, issues.

SUMMARY OF THE INVENTION

The invention of the present disclosure, in one aspect thereof, comprises a chemical sensor. The sensor has a test chamber for receiving chemicals in a gaseous state, the test chamber having two substantially transparent windows at first and second ends of the test chamber. A pulse operated ultraviolet light emitting diode is at the first end of the test chamber, emitting at a wavelength close to a maximum in the absorption band of a test chemical. An electromagnetic sensor is at a second end of the test chamber, the sensor being sensitive to the light emitted by the light emitting diode.

In one embodiment, the pulsed light emitting diode is modulated at about 1 kilohertz to increase the signal-to-noise ratio at the electromagnetic sensor. The electromagnetic sensor may be a photodiode. In one embodiment, the photodiode is sensitive to radiation at a wavelength of about 370 nanometers.

The windows may comprise a material that is resistant to degradation in the presence of chlorine dioxide. The window may comprise a material that is resistant to degradation in the presence of chlorine dioxide at relative humidities above 70%. In

one embodiment, the windows material is polyethylene terephthalate (PET). In another embodiment, the windows comprise fluorinated ethylene propylene (FEP).

The pulsed ultraviolet light emitting diode may operate in an on state for about 50 milliseconds per pulse and may remain in an off state for about 5 seconds between pulses.

The test chamber may be elongated and have an inlet port and an outlet port. In some embodiments, a beam splitter interposes the pulsed ultraviolet light emitting diode and the window at the first end of the test chamber. The beam splitter directs a portion of the electromagnetic radiation from the ultraviolet light emitting diode to a reference diode and passes a remainder of the radiation into the test chamber.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a cross-sectional view of one embodiment of a chlorine dioxide sensor according to aspects of the present disclosure.

FIG. 2 is a schematic view of the chlorine dioxide sensor of FIG. 1 with attached control and detection circuitry.

FIG. 3 is a perspective view of the chlorine dioxide sensor of FIG. 1 with attached control and detection circuitry.

FIG. 4 is a perspective view of the chlorine dioxide sensor of FIG. 1 with an opaque enclosure.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to FIG. 1, a cross-sectional view of one embodiment of a chlorine dioxide (ClO_2) sensor according to aspects of the present disclosure is shown. The chlorine dioxide sensor **100** is designed to detect chlorine dioxide in a gaseous state. Quantification may be achieved for minute (parts per billion) quantities up to very high concentrations. A test chamber **104** is defined by one or more test chamber walls **108**. Although the view of FIG. 1 is a cross-sectional view, it will be appreciated that there may be four walls **108** providing a rectangular test chamber **104**. In other embodiments, the test chamber **104** may be tubular and the walls **108** may represent a single tubular circumferential wall surrounding the test chamber **104**. At opposite ends of the test chamber **104** are a source end **102** and a detector end **106**.

In the present embodiment, an ultraviolet light emitting diode (UV LED) is provided for energizing the test chamber **104**. The presence of chlorine dioxide and/or the concentration thereof is determined by spectrographic analysis of the sample in the test chamber **104**. In the present embodiment, the UV LED **110** provides a beam of electromagnetic energy in the ultraviolet wavelength region. The beam is split by a beam splitter **112** that may comprise a quartz plate. A portion of the energy from the UV LED **110** may be directed by the beam splitter **112** to a reference diode **114**. The remainder of the energy from the UV LED **110** that is not reflected to the reference diode **114** will pass from the source end **102** through the test chamber **104** to the detector end **106**. The path of the beam is illustrated by line **115.** The detector end **106** is equipped with a detector diode **116**.

In operation, the UV LED **110** will be active or illuminated in a pulsed fashion. Illuminating the test chamber **104** in a pulsed or periodic fashion will minimize photochemical reactions with chlorine dioxide within the test chamber **104**. In one embodiment, the UV LED **110** will be periodically activated. For example, activation for about 50 milliseconds approximately every 5 seconds. To increase the signal-to-noise of the detected radiation, the UV LED **110** can also be modulated at frequencies in the kilohertz range (e.g., about 1 to about 10 kilohertz).

The UV LED **110** may provide electromagnetic radiation generally toward the ultraviolet portion of the spectrum but other wavelengths of light could be present in the beam as well. In the particular embodiment shown, the UV LED **110** will at least transmit around the wavelength of 370 nanometers. Both the reference diode **114** and the detector diode **116** will be sensitive to radiation at this wavelength. In this manner, the reference diode **114** may be activated to indicate that the UV LED **110** is active. Furthermore, the reference diode **114** may be used to compensate for any intensity variations in the output of the UV LED **110**.

Due in part to the corrosive and photochemical effects that may occur with chlorine dioxide, the test chamber walls **108**, as well as any other components of the sensor **100** that come in contact with chlorine dioxide, will need to be made of a material that is suitably resilient to the effects of chlorine dioxide. The materials may also be resistant to the effects of chlorine dioxide in a high humidity environment (e.g., above

70% relative humidity). In one embodiment, the components may comprise polyethylene terephthalate (PET). In another embodiment, the components may comprise fluorinated ethylene propylene (FEP). PET and FEP are two examples of materials that would be suitable to construct the sensor **100**.

It is contemplated that the sensor **100** may be installed and used more or less continuously for an indeterminate period of time. In the embodiment shown, one or more of the test chamber walls **108** will define opening **118**, **120** for introduction of test gases into the test chamber **104** and for removal of the tested sample. In one embodiment, it is contemplated that gas flow through the test chamber will be more or less continuous

In one embodiment, the source end **102** comprises the UV LED **110**, the beam splitter **112**, and the reference diode **114**. The construction of the beam splitter **112** in combination with the UV LED **110** and reference diode **114** may attach to an end plate **202**. The end plate **202** may define a first end of the test chamber **104**. The end plate **202** in the present embodiment is comprised of aluminum.

The end plate **202** may define a passage **203** to allow passage of the beam from the UV LED **110**. A film holder **204**, which may comprise PET, attaches against the end plate **202**. The film holder also defines a passage **205**. Retained by the film holder **204** is a thin film **208**. In the present embodiment, the thin film **208** comprises PET. The thin film **208** may comprise high-purity PET having a thickness of about 0.004 inches. A high purity film of PET that is sufficiently thin will not interfere substantially with the optics and operation of the sensor **100**. The thin film **208** provides a substantially transparent window into the test chamber **104**. As described, the components of the sensor **100** coming in contact with the sample, which may include chlorine dioxide, must be made from a resilient material. Thus, the end plate **202** is protected by the film **208** and the film holder **204**.

An O-ring **206** may be provided to seal the end of the test chamber **104**. The O-ring may comprise a fluorocarbon elastomer such as Viton®. Viton® will be less resilient against the corrosive effects of the chlorine dioxide than those components comprising PET. However, the Viton® will adequately seal the test chamber **104** and will be somewhat protected from the chlorine dioxide by the configuration of the end **102** of the sensor **100**. It can be seen that the end plate **202** and film holder **204** project

inward to the chamber **104** against the walls **108**. This will prevent the O-ring **206** from being in the primary gas flow during operation and will serve to decrease the corrosive effects of the chlorine dioxide.

The detector end **106** comprises another end plate **302**, which also positions the detector diode **116** in the center of the beam path **115.** The end plate **302** may comprise aluminum. Attached on the outside of the end plate **302** is the detector diode **116**. An end cap **304** attaches to the inside of the end plate **302**. The end cap **304** may comprise PET and define a passage **305.** The end cap also protects the end plate **302** from coming in contact with the sample, which may include chlorine dioxide. The passage **305** allows for maximum transmission of the test beam from the test chamber **104** to the detector diode **116**. In some embodiments, the end cap **304** retains another thin film **308** against the end plate **302** and the photodiode **116**. This film **308** may also comprise high purity PET having thickness of about .004 inches. This will allow the beam to exit the test chamber **104** and reach the detector diode **116** substantially unaffected. The thin film **308** provides a substantially transparent window into the test chamber **104**.

As before, a Viton® O-ring **306** is provided to seal the end **106** of the sensor **100**. The configuration of the second end **106** of the sensor **100** will keep the O-ring **306** out of the main gas flow and serve as some protection against the corrosive effects of the chlorine dioxide on the O-ring.

The detector diode **116** is configured to detect absorption around the ultraviolet absorbance maximum of the chlorine dioxide spectra. Thus, the LED beam traveling from the UV LED **110** through the test chamber **104** and striking the detector diode **116** will have been altered in the presence of chlorine dioxide gas. This alteration may present itself in the form of a loss in intensity of the test beam.

As can be seen in FIG. 1, various plastic screws may be used to hold the major components of the sensor **100** in place. In one embodiment, the assembly screws will be nylon. However, in other embodiments, the components may be assembled and epoxied or glued in place. For example, the walls **108** may be epoxied together and against the end plates **202, 302**. The film holder **204** and end cap **304** may also be epoxied in place against the insides of the end plates **202**, **302**, respectively.

Referring now to FIG. 2, a schematic diagram of the chlorine dioxide sensor of FIG. 1 with attached control and detection circuitry **402** is shown. The control and detection circuitry **402** provides power and control to the UV LED **110** as needed. The control and detection circuitry **402** connects to the UV LED **110** via wire leads **410**. The control and detection circuitry **402** connects to the reference diode **114** and the detector diode **116** via wire leads **412** and **414**, respectively.

The detection circuitry **402** may read and/or compare the output from the LEDs **114**, **116** to determine whether there is a significant absorption that would be indicative of chlorine dioxide within the test chamber. The concentration of chlorine dioxide in the test chamber **104** may also be discernable. In one embodiment, detection and control circuitry may comprise one or more integrated circuits and/or discrete analog or digital components.

The functionality of the circuitry **402** could also be provided in software operating on a general purpose computer or integrated circuit. In addition, the circuitry **402** may provide the necessary signal conditioning and amplification to ensure adequate and usable readings from the diodes **114**, **116**. The output from the circuitry **402** may be an analog voltage or a digital reading. In some embodiments, the circuitry **402** will be equipped to log or record readings for later retrieval.

In one embodiment, the photocurrent of the detector diode **116** will be measured by the circuitry **402**. A change in photocurrent of the detector diode **116** may correspond to the presence and/or concentration of chlorine dioxide. Enhanced signal-to-noise ratio may be obtained at the detector circuit **402** by modulating the UV LED **110** at several kilohertz. In some embodiments, the reference diode **114** will measure the output of the UV LED **110** to correct for possible variations in the output of the UV LED **110**. The photocurrent of the reference diode **114** will be proportional to the output of the UV LED **110** regardless of the presence of chlorine dioxide because the UV light incident on the reference diode **114** will not pass through the test chamber.

In FIG. 2, environmental gas is shown as a cloud **404**. The environmental gas **402** may be ambient atmosphere that is to be tested for chlorine dioxide. The gas **402** may also be the output or input of a specific process for which monitoring is desired. In some embodiments, a conduit or input port **406** may be provided for delivering the test

sample to the sensor **100**. An outlet conduit or port **408** may be provided for exhausting the tested sample. In some embodiments, the ports **406**, **408** provide a continuously refreshed test sample into the openings **118**, **120** (FIG. 1) of the test chamber. Positive pressure and/or vacuum may be used depending upon the needs of the user.

Referring now to FIG. 3, a perspective view of the chlorine dioxide sensor of FIG. 1 with attached control and detection circuitry is shown. Here, the overall physical shape of the sensor **100** can be seen. In the embodiment shown, the test chamber is defined in part by a single cylindrical wall **108**. The detection and control circuitry **402** has been mounted to the wall **108** outside the test chamber. The leads **414** connecting the control circuitry **402** to the detector diode **116** can be seen leading to the detector end **106** of the sensor **100**. Similarly, the leads **410**, **412** connecting the circuitry **402** to the UV LED **110** and the reference diode **114**, respectively, can also be seen leading to the source end **102**.

Referring now to FIG. 4, a perspective view of the chlorine dioxide sensor of FIG. 1 with an opaque enclosure **420** is shown. Due to the detrimental photochemical reactions that are possible with chlorine dioxide, the test chamber **104** may need to be shielded from ambient light. Depending upon the wavelength of the ambient light, the diodes **114**, **116** may also benefit from shielding. The detection electronics may also benefit from shielding to reduce possible electromagnetic interferences. In the present embodiment, an opaque casing **420** has been fitted around the entire sensor **100**. In order to provide test samples to the sensors **100**, ports **406**, **408** are provided in communication with the openings **118**, **120** of the wall **108** of the test chamber **104**. Using positive pressure and/or vacuum, a continuous or intermittent test sample may be provided. In order to power the circuitry **402** and to read the output or results of the test, a communication and power port **412** may be provided. The enclosure **420** itself may comprise an appropriate metal, plastic, or other material of suitable opacity and resiliency to the environment in which the sensor **100** will be used.

* * * *

Thus, the present invention is well adapted to carry out the objectives and attain the ends and advantages mentioned above as well as those inherent therein. While presently preferred embodiments have been described for purposes of this disclosure,

numerous changes and modifications will be apparent to those of ordinary skill in the art. Such changes and modifications are encompassed within the spirit of this invention as defined by the claims.

CLAIMS

What is claimed is:

1. A chemical sensor comprising:

a test chamber for receiving chemicals in a gaseous state, the test chamber having two substantially transparent windows at first and second ends of the test chamber;

a pulse operated ultraviolet light emitting diode at the first end of the test chamber emitting at a wavelength close to a maximum in the absorption band of a test chemical; and

an electromagnetic sensor at a second end of the test chamber, the sensor being sensitive to the light emitted by the light emitting diode.

2. The chemical sensor of claim 1, wherein the pulsed light emitting diode is modulated at about 1 kilohertz to increase the signal-to-noise ratio at the electromagnetic sensor.

3. The chemical sensor of claim 1, wherein the windows comprise a material that is resistant to degradation in the presence of chlorine dioxide.

4. The chemical sensor of claim 1, wherein the window comprises a material that is resistant to degradation in the presence of chlorine dioxide at relative humidities above 70%.

5. The chemical sensor of claim 1, wherein the windows comprise polyethylene terephthalate (PET).

6. The chemical sensor of claim 1, wherein the windows comprise fluorinated ethylene propylene (FEP).

7. The chemical sensor of claim 1, wherein the pulsed ultraviolet light emitting diode operates in an on state for about 50 milliseconds per pulse.

8. The chemical sensor of claim 7, wherein the pulsed ultraviolet light emitting diode remains in an off state for about 5 seconds between pulses.

9. The chemical sensor of claim 1, wherein the test chamber is elongated and has an inlet port and an outlet port.

10. The chemical sensor of claim 1, further comprising:

a beam splitter interposing the pulsed ultraviolet light emitting diode and the window at the first end of the test chamber; and

a reference photodiode;

wherein the beam splitter directs a portion of the electromagnetic radiation from the ultraviolet light emitting diode to the reference diode and passes a remainder of the radiation into the test chamber.

11. The chemical sensor of claim 1, wherein the electromagnetic sensor at the second end of the test chamber is a photodiode.

12. The chemical sensor of claim 1, wherein the electromagnetic sensor at the second end of the photodiode is sensitive to radiation at a wavelength of about 370 nanometers.

13. A chemical sensor for detecting the presence of chlorine dioxide in a gaseous state, comprising:

a test chamber with first and second ends, the first and second end each having a polyethylene terephthalate (PET) window;

an ultraviolet light emitting diode (UV LED) proximate the first end window;

a test diode proximate the second end window, the test diode sensitive to light of about 370 nanometers in wavelength; and

a control system for activating the UV LED intermittently to provide ultra violet light into the test chamber;

detecting circuitry that reads a signal from the test diode to determine the degree of absorption at about 370 nanometers and thereby determines a concentration of gaseous chlorine dioxide in the test chamber.

14. The chemical sensor of claim 13, further comprising:

a reference diode; and

a beam splitter interposing the UV LED and the first window, the beam splitter directing a portion of the UV LED light to the reference diode and passing a remainder of the light into the test chamber;

wherein the detecting circuitry reads a reference signal from the reference diode to detect operation of the chemical sensor and normalize the measured absorbance.

15. The chemical sensor of claim 13, further comprising an inlet port and an outlet port on the test chamber, the inlet port receiving gases to be tested, the outlet port providing gases post testing.

16. The chemical sensor of claim 13, wherein the control system pulses the UV LED for lengths of time less than that necessary to significantly degrade chlorine dioxide gas in the test chamber due to photochemical effects.

17. The chemical sensor of claim 13, wherein the control circuitry pulses the UV LED on for about 50 milliseconds approximately every 5 seconds.

18. A method of detecting chlorine dioxide in a gaseous state, comprising:

containing a test sample within a test chamber having first and second windows comprising a material resistant to degradation by chlorine dioxide;

illuminating the test sample with ultraviolet light through the first test window for a period of time insufficient to cause any substantial degradation of the sample due to photochemical effects on the chlorine dioxide; and

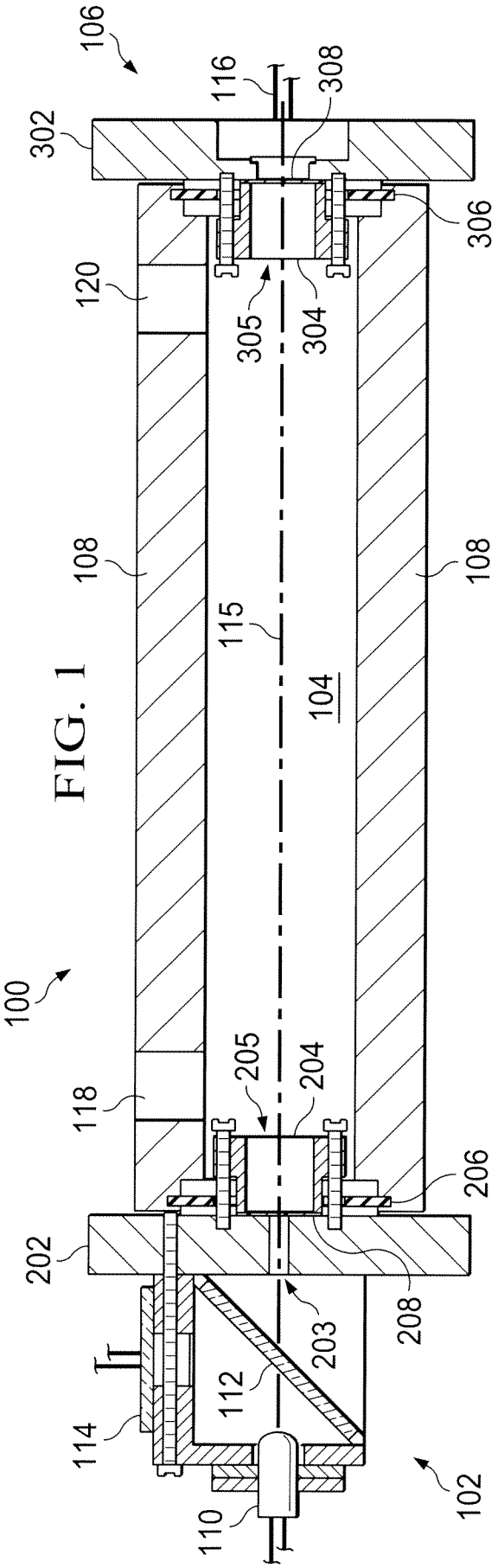
testing the absorption of the sample at the second window.

19. The method of claim 18, wherein testing the absorption of the sample further comprises testing absorption of the sample at about 370 nanometers.

20. The method of claim 18, further comprising removing the test sample from an outlet port in the test chamber while introducing a new test sample into an inlet port of in the test chamber.

21. The method of claim 18, wherein illuminating the test sample further comprises illuminating the sample for about 50 milliseconds approximately every 5 seconds.

22. The method of claim 18, wherein containing a test sample within a test chamber having first and second windows comprising a material resistant to degradation by chlorine dioxide further comprises providing windows comprising of polyethylene terephthalate (PET).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302
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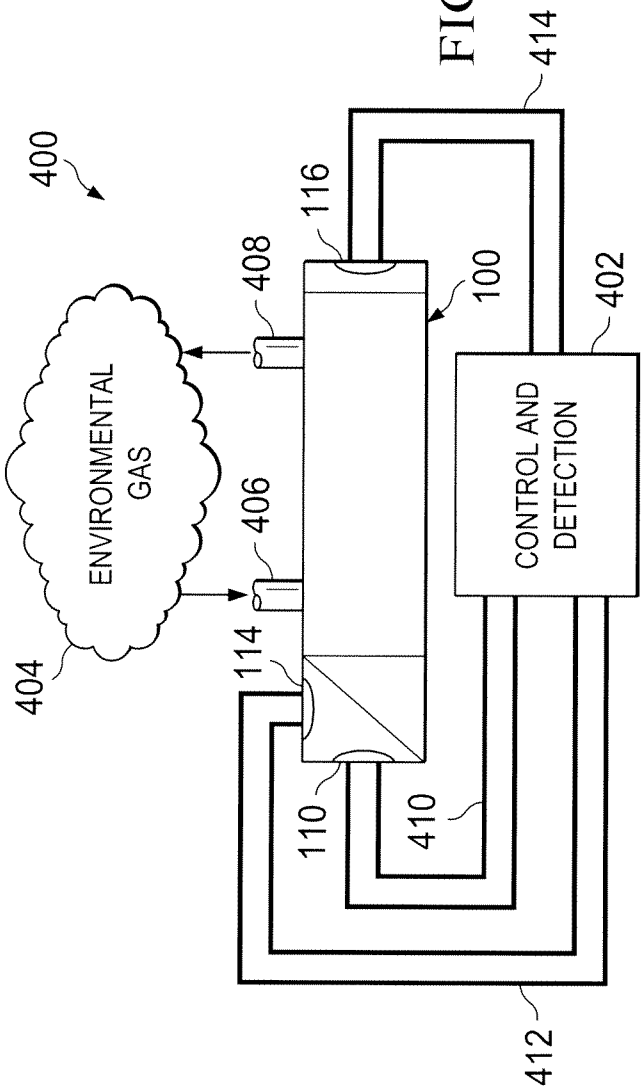
114
110
102

FIG. 1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100
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ENVIRONMENTAL GAS
406
CONTROL AND DETECTION
404
114
110
410
412

FIG. 2

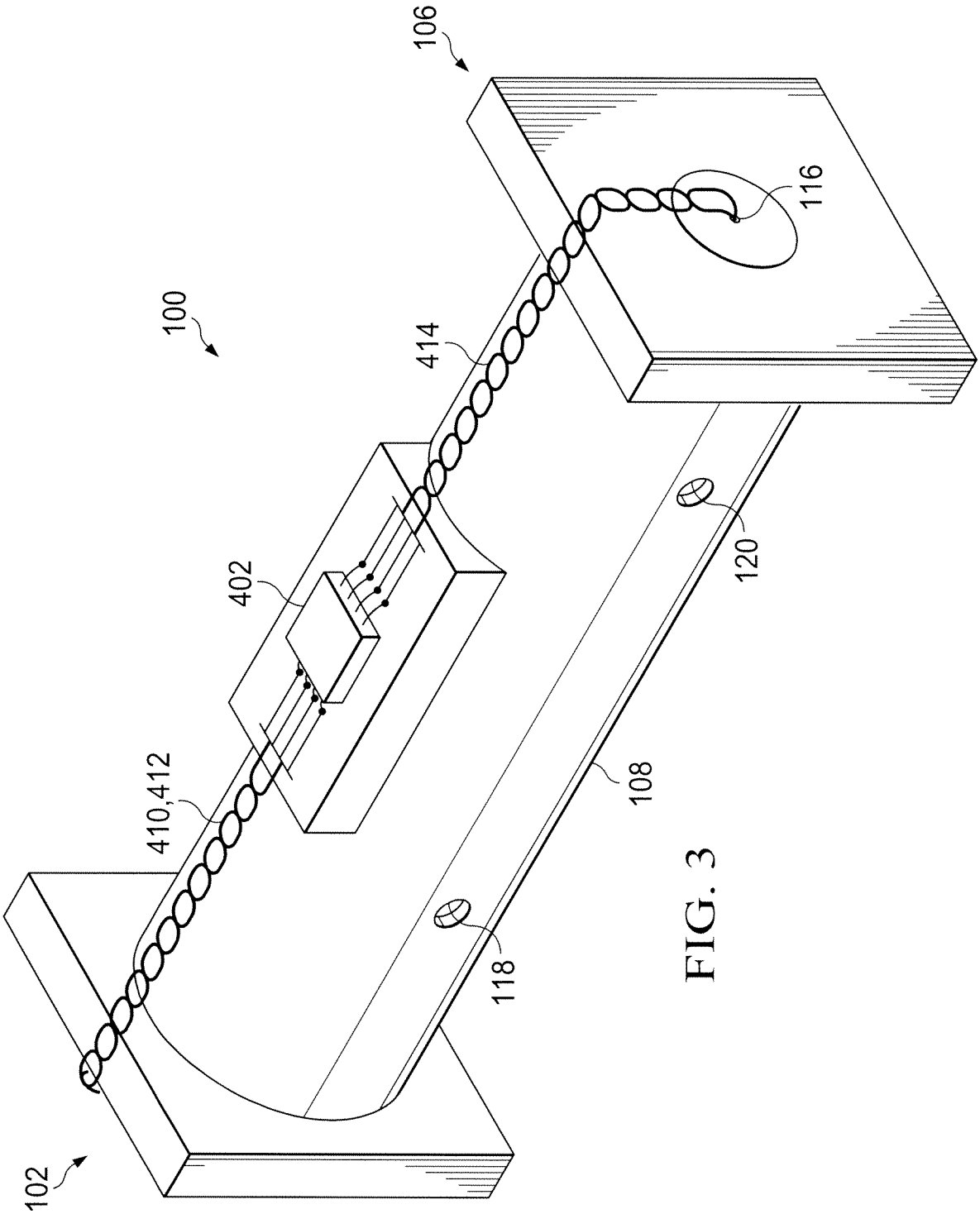

FIG. 3

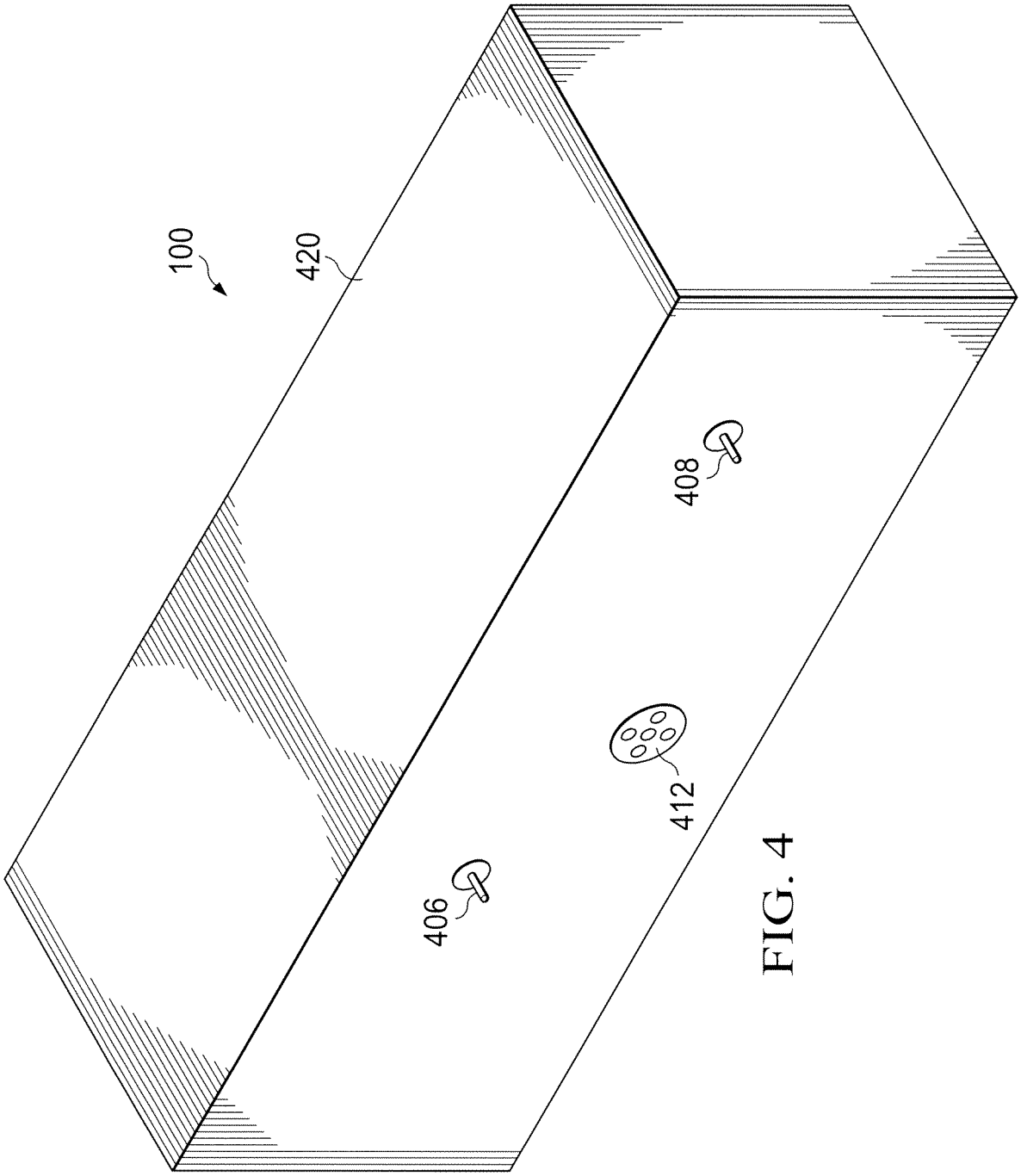

FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2009/043594

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.

G01N 21/33 (2006.01)

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)

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, WPI- IPC & KEYWORDS - LIGHT EMITTING DIODE, LED, INTENSITY, LUMINATION, CONCENTRATION, MEASURE, RECORD, ANALYSE, ULTRA VIOLET, UV, CHLORINE DIOXIDE, CLO AND OTHER LIKE TERMS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 1999/053297 (INSTRUMENTARIUM CORPORATION) 21 October 1999 Whole document	
A	DE 60216988T (BOUNAIX FABRICE MARCEL S) 18 October 2007 Abstract	
A	US 2004062683 A1 (WING-WAH, WAI-KIN) 1 April 2004 Whole document	
A	JP 2008157874 A (MASAHRIO, HIDEKI) 10 July 2008 Whole document	

☐ 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

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"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	Date of mailing of the international search report
07 September 2009	10 SEP 2009
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No. +61 2 6283 7999	Authorized officer **PATRICK ROBERTS** AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No :

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/US2009/043594

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
WO	9953297	EP	0988521	US	6791689		
DE	60216988	NONE					
US	2004062683	AU	2003271509	CN	1701228	EP	1546686
		WO	2004029597				
JP	2008157874	NONE					

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX