US 20170023463A1

(19) United States

(12) Patent Application Publication

(10) Pub. No.: US 2017/0023463 A1

Mayer et al.

(43) Pub. Date: Jan. 26, 2017

(54) **RUGGED TARGET-ANALYTE PERMEATION TESTING INSTRUMENT EMPLOYING A CONSOLIDATING BLOCK MANIFOLD**

(71) Applicant: **MOCON, INC.**, Minneapolis, MN (US)

(72) Inventors: **Daniel W. Mayer**, Wyoming, MN (US); **Slava A. Berezovskiy**, Apple Valley, MN (US)

(73) Assignee: **MOCON, INC.**, Minneapolis, MN (US)

(21) Appl. No.: **15/039,100**

(22) PCT Filed: **Feb. 24, 2015**

(86) PCT No.: **PCT/US15/17193**

§ 371 (c)(1),
(2) Date: **May 25, 2016**

Related U.S. Application Data

(60) Provisional application No. 61/943,772, filed on Feb. 24, 2014.

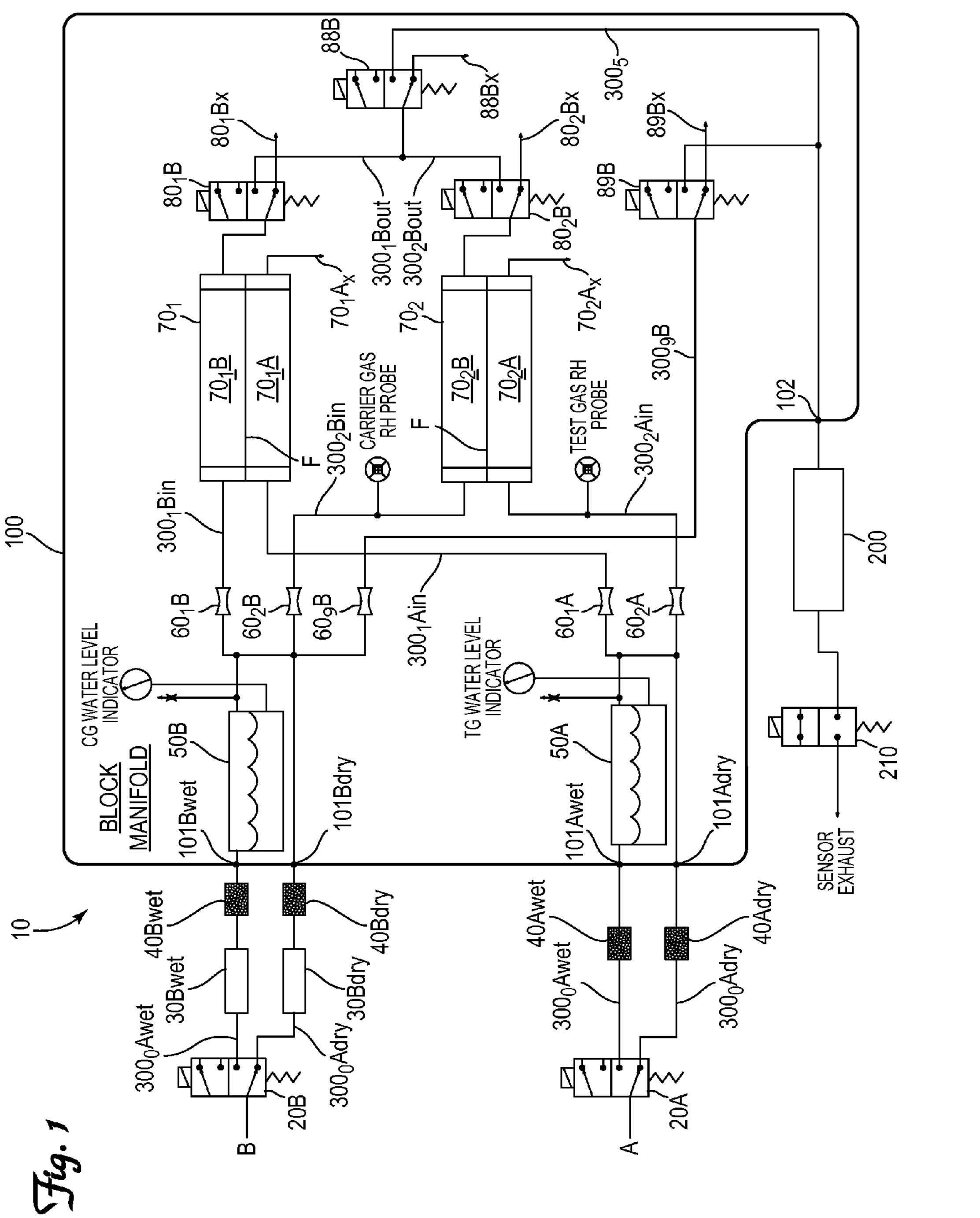
Publication Classification

(51) **Int. Cl.**
G01N 15/08 (2006.01)

(52) **U.S. Cl.**
CPC ***G01N 15/0826*** (2013.01)

(57) **ABSTRACT**

A target-analyte permeation testing instrument (**10**) characterized by a block manifold (**100**) retaining the testing cells (**70***n*) of the instrument (**10**).


10
100
BLOCK MANIFOLD
CG WATER LEVEL INDICATOR
TG WATER LEVEL INDICATOR
CARRIER GAS RH PROBE
TEST GAS RH PROBE
SENSOR EXHAUST
A
B
20A
20B
300_0Awet
300_0Adry
30Bwet
30Bdry
40Awet
40Adry
40Bwet
40Bdry
101Awet
101Adry
101Bwet
101Bdry
50A
50B
60_1A
60_2A
60_1B
60_2B
60_9B
300_1Ain
300_2Ain
300_1Bin
300_2Bin
300_9B
70_1
70_2
70_1B
70_1A
70_2B
70_2A
70_1A_x
70_2A_x
F
80_1B
80_2B
80_1Bx
80_2Bx
300_1Bout
300_2Bout
88B
88Bx
89B
89Bx
300_5
102
200
210
Fig. 1

RUGGED TARGET-ANALYTE PERMEATION TESTING INSTRUMENT EMPLOYING A CONSOLIDATING BLOCK MANIFOLD

BACKGROUND

[0001] Permeation instruments are used to measure the transmission rate of a target analyte, such as oxygen, carbon dioxide or water vapor, through various samples, such as membranes, films, envelopes, bottles, packages, containers, etc. (hereinafter collectively referenced as “test films” for convenience). Typical test films are polymeric packaging films such as those constructed from low density polyethylene (LDPE), high density polyethylene (HDPE), oriented polypropylene (OPP), polyethylene terepthalate (PET), polyvinylidene chrloride (PVTDC), etc. Typically, the film to be tested is positioned within a test chamber to sealing separate the chamber into first and second chambers. The first chamber (commonly referenced as the driving or analyte chamber) is filled with a gas containing a known concentration of the target analyte (commonly referenced as a driving gas). The second chamber (commonly referenced as the sensing chamber) is flushed with an inert gas (commonly referenced as a carrier gas) to remove any target analyte from the cell. A sensor for the target analyte is placed in fluid communication with the sensing chamber for detecting the presence of target analyte that has migrated into the sensing chamber from the driving chamber through the test film. Exemplary permeation instruments for measuring the transmission rate of oxygen (O_2), carbon dioxide (CO_2) and water vapor (H_2O) through test films are commercially available from Mocon, Inc. of Minneapolis, Minn. under the designations OXTRAN, PERMATRAN-C and PERMATRAN-W, respectively.

[0002] Permeation instruments are being used more often to measure ever decreasing concentrations of target-analyte, into the ppm or even ppb range, and are therefore extremely sensitive to even minute atmospheric contamination of the fluids used in the instrument. Permeation instruments employ an extensive network of fluid interconnections with numerous valves to achieve the desired choreographed flow of driving and carrier gas through the instrument, especially when the instrument employs a plurality of testing cells in fluid communication with a single common target-analyte sensor. Each fitting in the fluid transfer system of the instrument is a potential source of contamination as atmospheric oxygen, carbon dioxide and water vapor leak around or permeate through the seals on the fittings, especially as the seals on the fittings loosen over time.

[0003] Accordingly, a substantial need exists for a permeation instrument capable near elimination of atmospheric-induced contamination of the driving and carrier gases flowing through the instrument throughout the lifespan of the instrument.

SUMMARY OF THE INVENTION

[0004] The invention is a target-analyte permeation testing instrument characterized by a block manifold. The instrument has a target-analyte sensor and a plurality of test cells for measuring target-analyte permeation rate of a test film. Each test cell defines a testing chamber and is operable for retaining a test film to sealingly divide the testing chamber into a driving chamber and a sensing chamber. The block manifold is fixed to the plurality of cells and has a plurality of channels in fluid communication with the testing chamber of each cell, a pressurized source of driving gas, a pressurized source of inert gas, and a target-analyte sensor. The plurality of channels are configured and arranged to selectively carry driving gas from the pressurized source of driving gas to the driving chamber of each cell, carry driving gas from the driving chamber of each cell to a driving gas exit port in the manifold, selectively carry inert gas from the pressurized source of inert gas to the sensing chamber of each cell, and selectively carry inert gas from the sensing chamber of each cell to the target-analyte sensor.

[0005] The block manifold can include a refillable first water reservoir in selective fluid communication with the source of driving gas and in fluid communication with the driving chamber of each cell, and a second refillable water reservoir in selective fluid communication with the source of inert gas and in fluid communication with the sensing chamber of each cell.

BRIEF DESCRIPTION OF THE DRAWINGS

[0006] FIG. **1** is a schematic plumbing diagram of one embodiment of the invention.

DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT

[0007]

Nomenclature Table	
10	Target-Analyte Permeation Testing Instrument
20A	Test Gas RH Control Valve
20B	Carrier Gas RH Control Valve
$30B_{wet}$	Catalyst Chamber in Wet Carrier Gas Line
$30B_{dry}$	Catalyst Chamber in Dry Carrier Gas Line
$40A_{wet}$	Particle Filter in Wet Test Gas Line
$40A_{dry}$	Particle Filter in Dry Test Gas Line
$40B_{wet}$	Particle Filter in Wet Carrier Gas Line
$40B_{dry}$	Particle Filter in Dry Carrier Gas Line
50A	Water Reservoir for Test Gas
50B	Water Reservoir for Carrier Gas
60_n	Capillary Restrictors
60_1A	Capillary Restrictor for Test Gas Channel to First Test Cell
60_2A	Capillary Restrictor for Test Gas Channel to Second Test Cell
60_1B	Capillary Restrictor for Carrier Gas Channel to First Test Cell
60_2B	Capillary Restrictor for Carrier Gas Channel to Second Test Cell
60_9B	Capillary Restrictor for Carrier Gas Channel to Rezero Valve
70_n	Testing Cells
70_nA	Driving Chamber of Test Cell n
70_nB	Sensing Chamber of Test Cell n
70_nAx	Exhaust from Driving Chamber of Test Cell n
70_1	First Testing Cells
70_1A	Driving Chamber of First Test Cell
70_1Ax	Exhaust from Driving Chamber of First Test Cell
70_1B	Sensing Chamber of First Test Cell
70_2	Second Testing Cells
70_2A	Driving Chamber of Second Test Cell
70_2Ax	Exhaust from Driving Chamber of Second Test Cell
70_2B	Sensing Chamber of Second Test Cell
80_nB	Carrier Gas Sensing Chamber Exit Valve for Testing Cell n
80_1B	First Test Cell Carrier Gas Exit Valve
80_1Bx	Exhaust from Sensing Chamber of First Test Cell
80_2B	Second Test Cell Carrier Gas Exit Valve
80_2Bx	Exhaust from Sensing Chamber of Second Test Cell
88B	Cell Selector Channel Conditioning Valve
88Bx	Exhaust from Cell Selector Channel Conditioning Valve

-continued

Nomenclature Table	
89B	Rezero Valve
89Bx	Exhaust from Rezero Valve
100	Block Manifold
$101A_{wet}$	Test Gas Water Reservoir Inlet Port
$101A_{dry}$	Test Gas Water Reservoir Bypass Inlet Port
$101B_{wet}$	Carrier Gas Water Reservoir Inlet Port
$101B_{dry}$	Carrier Gas Water Reservoir Bypass Inlet Port
102	Carrier Gas Outlet Port to Sensor
200	Target-Analyte Sensor
210	Sensor Exhaust Valve
300_n	Gas Flow Line n
300_0A_{wet}	Test Gas Water Reservoir Inlet Line
300_0A_{dry}	Test Gas Water Reservoir Bypass Inlet Line
300_0B_{wet}	Carrier Gas Water Reservoir Inlet Line
300_0B_{dry}	Carrier Gas Water Reservoir Bypass Inlet Line
300_1A_{in}	Test Gas First Testing Cell Inlet Line
300_1B_{in}	Carrier Gas First Testing Cell Inlet Line
300_2A_{in}	Test Gas Second Testing Cell Inlet Line
300_2B_{in}	Carrier Gas Second Testing Cell Inlet Line
300_nB_{out}	Carrier Gas Outlet Line for Testing Cell n
300_1B_{out}	Carrier Gas First Testing Cell Outlet Line
300_2B_{out}	Carrier Gas Second Testing Cell Outlet Line
300_5	Shared Carrier Gas Testing Cell Outlet Line
300_9B	Carrier Gas Rezero Line
A	Driving or Test Gas Source
B	Inert or Carrier Gas Source
F	Test Film

DESCRIPTION

[0008] Referring generally to FIG. **1**, the invention is a target-analyte permeation testing instrument **10** characterized by a block manifold **100**, preferably a solid block cast metal manifold **100** into which the appropriate channels and compartments are formed. The instrument **10** has a target-analyte sensor **200** and a plurality of test cells $\mathbf{70}_n$ for measuring target-analyte permeation rate of test films F_n. Each test cell $\mathbf{70}_n$ defines a testing chamber and is operable for retaining a test film F to sealingly divide the testing chamber into a driving chamber $\mathbf{70}_n$A and a sensing chamber $\mathbf{70}_n$B. The cells $\mathbf{70}_n$ are secured to the block manifold **100**. The block manifold **100** has a plurality of channels $\mathbf{300}_n$ in fluid communication with the testing chamber of each cell $\mathbf{70}_n$, a pressurized source of driving gas A, a pressurized source of inert gas B, and a target-analyte sensor **200**. The plurality of channels $\mathbf{300}_n$ are configured and arranged to selectively carry driving gas from the pressurized source of driving gas A to the driving chamber $\mathbf{70}_n$A of each cell $\mathbf{70}_n$, carry driving gas from the driving chamber $\mathbf{70}_n$A of each cell $\mathbf{70}_n$ to a driving gas exit port $\mathbf{70}_n$Ax in the manifold **100**, selectively carry inert gas from the pressurized source of inert gas B to the sensing chamber $\mathbf{70}_n$B of each cell $\mathbf{70}_n$, and selectively carry inert gas from the sensing chamber $\mathbf{70}_n$B of each cell $\mathbf{70}_n$ to a the target-analyte sensor **200**.

[0009] The block manifold **100** can include a refillable first water reservoir **50**A in selective fluid communication with the source of driving gas A and in fluid communication with the driving chamber $\mathbf{70}_n$A of each cell $\mathbf{70}_n$, and a second refillable water reservoir **50**B in selective fluid communication with the source of inert gas B and in fluid communication with the sensing chamber $\mathbf{70}_n$B of each cell $\mathbf{70}_n$.

[0010] An exemplary two-cell embodiment of the invention **10** is depicted in FIG. **1**. The permeation testing instrument **10** preferably includes humidification systems for each of the test gas and carrier gas, such as described in U.S. Pat. Nos. 7,578,208 and 7,908,936, the disclosures of which are hereby incorporated by reference.

[0011] A source of dry test gas A fluidly communicates with a first humidification system that includes a wet line $\mathbf{300}_0A_{wet}$ in fluid communication with a water reservoir **50**A and a dry line $\mathbf{300}_0A_{dry}$ that bypasses the water reservoir **50**A. A test gas RH control valve **20**A controls flow of test gas through the wet line $\mathbf{300}_0A_{wet}$ and dry line $\mathbf{300}_0A_{dry}$ according to a duty cycle for achieving the desired humidification level of the test gas.

[0012] The test gas wet line $\mathbf{300}_0A_{wet}$ enters the block manifold **100** at inlet port $\mathbf{101}A_{wet}$. The test gas dry line $\mathbf{300}_0A_{dry}$ enters the block manifold **100** at inlet port $\mathbf{101}A_{dry}$.

[0013] Upon exiting the water reservoir **50**A, humidified test gas in the wet line $\mathbf{300}_0A_{wet}$ is combined with dry test gas in the dry line $\mathbf{300}_0A_{dry}$ and the combined test gas directed by test gas inlet lines $\mathbf{300}_1$A and $\mathbf{300}_2$A to the driving chambers $\mathbf{70}_1$A and $\mathbf{70}_2$A in the first testing cell $\mathbf{70}_1$ and second testing cell $\mathbf{70}_2$ respectively. Test gas flows through and exits each of the driving chambers $\mathbf{70}_1$A and $\mathbf{70}_2$A through an outlet port (unnumbered) and is vented from the block manifold at vent ports $\mathbf{70}_1$Ax and $\mathbf{70}_2$Ax respectively.

[0014] Particle filters $\mathbf{40}A_{wet}$ and $\mathbf{40}A_{dry}$ are preferably provided in the test gas wet line $\mathbf{300}_0A_{wet}$ and test gas dry line $\mathbf{300}_0A_{dry}$ respectively, for removing any entrained particulate matter from the test gas before it enters the block manifold **100**.

[0015] In a similar fashion, a source of dry carrier gas B fluidly communicates with a second humidification system that includes a wet line $\mathbf{300}_0B_{wet}$ in fluid communication with a water reservoir **50**B and a dry line $\mathbf{300}_0B_{dry}$ that bypasses the water reservoir **50**B. A carrier gas RH control valve **20**B controls flow of carrier gas through the wet line $\mathbf{300}_0B_{wet}$ and dry line $\mathbf{300}_0B_{dry}$ according to a duty cycle for achieving the desired humidification level of the carrier gas.

[0016] The carrier gas wet line $\mathbf{300}_0B_{wet}$ enters the block manifold **100** at inlet port $\mathbf{101}B_{wet}$. The carrier gas dry line $\mathbf{300}_0B_{dry}$ enters the block manifold **100** at inlet port $\mathbf{101}B_{dry}$.

[0017] Upon exiting the water reservoir **50**B, humidified carrier gas in the wet line $\mathbf{300}_0B_{wet}$ is combined with dry carrier gas in the dry line $\mathbf{300}_0B_{dry}$ and the combined carrier gas directed by carrier gas inlet lines $\mathbf{300}_1$B and $\mathbf{300}_2$B to the sensing chambers $\mathbf{70}_1$B and $\mathbf{70}_2$B in the first testing cell $\mathbf{70}_1$ and second testing cell $\mathbf{70}_2$ respectively. Carrier gas flows through and exits each of the sensing chambers $\mathbf{70}_1$B and $\mathbf{70}_2$B through an outlet port (unnumbered) and is directed by dedicated outlet channels $\mathbf{300}_1B_{out}$ and $\mathbf{300}_2B_{out}$ respectively, to a common channel $\mathbf{300}_5$ in fluid communication with a target-analyte sensor **200** located external to the block manifold **100**.

[0018] Common channel $\mathbf{300}_5$ exits the block manifold **100** at outlet port **102**.

[0019] Particle filters $\mathbf{40}B_{wet}$ and $\mathbf{40}B_{dry}$ are preferably provided in the carrier gas wet line $\mathbf{300}_0B_{wet}$ and carrier gas dry line $\mathbf{300}_0B_{dry}$ respectively, for removing any entrained particulate matter from the carrier gas before it enters the block manifold **100**.

[0020] Target-analyte catalytic converters $\mathbf{30}B_{wet}$ and $\mathbf{30}B_{dry}$ are preferably provided in the carrier gas wet line $\mathbf{300}_0B_{wet}$ and carrier gas dry line $\mathbf{300}_0B_{dry}$ respectively, for converting any target-analyte in the carrier gas (e.g., O_2) to a molecular species (e.g., H_2O when the target analyte is O_2) that will not be detected by the target-analyte sensor **200**.

[0021] Capillary restrictors $\mathbf{60}_1$A, $\mathbf{60}_2$A, $\mathbf{60}_1$B and $\mathbf{60}_2$B are preferably provided in the test gas inlet lines $\mathbf{300}_1$A and $\mathbf{300}_2$A, and carrier gas inlet lines $\mathbf{300}_1$B and $\mathbf{300}_2$B respectively, for facilitating a consistent and equal flow of gas into the driving chambers $\mathbf{70}_1$A and $\mathbf{70}_2$A of the testing cells $\mathbf{70}_1$ and $\mathbf{70}_2$, and the sensing chambers $\mathbf{70}_1$B and $\mathbf{70}_2$B of the testing cells $\mathbf{70}_1$ and $\mathbf{70}_2$ respectively. The capillary restrictors $\mathbf{60}_n$ are preferably side mounted onto the block manifold **100**.

[0022] Valves $\mathbf{80}_1$B and $\mathbf{80}_2$B are provided in the dedicated outlet channels $\mathbf{300}_1\mathrm{B}_{out}$ and $\mathbf{300}_2\mathrm{B}_{out}$ respectively, for selectively and mutually exclusively allowing passage of carrier gas, containing any target-analyte that has permeated through the test film F, from each of the sensing chambers $\mathbf{70}_1$B and $\mathbf{70}_2$B into sensing engagement with the sensor **200**. When closed, the valves $\mathbf{80}_1$B and $\mathbf{80}_2$B vent carrier gas, containing any target-analyte that has permeated through the test film F, to atmosphere through vent ports $\mathbf{80}_1$Bx and $\mathbf{80}_2$Bx in the manifold **100**. The valves $\mathbf{80}_n$B are preferably side mounted onto the block manifold **100**.

[0023] The instrument **10** depicted in FIG. **1** includes an optional channel conditioning feature. Permeation testing instruments **10** employ a very low mass flow through rate through the gas flow lines **300***n* of the instrument **10** to limit the creation of any pressure differentials in the instrument **10** that could impact humidification of the test and/or carrier gases or create a pressure-induced driving force across a test film F. This low mass flow rate through the instrument **10** imposes a significant time delay between measurements from different testing cells $\mathbf{70}_n$ as both the "stale" carrier gas contained in the length of the testing cell outlet line $\mathbf{300}_n\mathrm{B}_{out}$ for the upcoming testing cell $\mathbf{70}_n$ to be measured and the "inapplicable" carrier gas contained in the length of the shared outlet line $\mathbf{300}_5$ from the previously measured testing cell $\mathbf{70}_n$ is flushed from the lines and replaced with fresh carrier gas, containing any target-analyte that has permeated through the test film F, from the upcoming testing cell $\mathbf{70}_n$. A channel conditioning feature employs a cell selector channel conditioning valve **88**B in the shared outlet line $\mathbf{300}_5$ for allowing, in coordination with opening and closing of valves $\mathbf{80}_n$B for the upcoming and previous testing cells $\mathbf{70}_n$, for advanced venting of "stale" carrier gas contained in the length of the outlet line $\mathbf{300}_n\mathrm{B}_{out}$ for the upcoming testing cell $\mathbf{70}_n$. The cell selector channel conditioning valve **88**B is operable as between a flow-through state, in which carrier gas is directed to the sensor **200**, and a vent state, in which carrier gas is vented to atmosphere through a vent port **88**Bx in the block manifold **100**. The cell selector channel conditioning valve **88**B is preferably side mounted to the block manifold **100**.

[0024] The instrument **10** depicted in FIG. **1** includes an optional rezero feature. Rezero is a method of measuring residual target-analyte contained in the carrier gas during performance of testing that includes the steps of bypassing the test cell(s) $\mathbf{70}_n$ and directly measuring the carrier gas target-analyte level, which is then subtracted from the measured transmission rate of the target-analyte level for each sample.

[0025] The rezero feature includes a rezero line $\mathbf{300}_9$B upstream from the testing cells $\mathbf{70}_n$ for bypassing the testing cells $\mathbf{70}_n$ and carrying carrier gas directly to the sensor **200**. A rezero valve **89**B is provided in the rezero line $\mathbf{300}_9$B for selectively directing carrier gas to the sensor **200** or venting carrier gas from the block manifold **100** at vent port **89**Bx. The rezero valve **89**B is preferably side mounted to the block manifold **100**.

[0026] A capillary restrictor $\mathbf{60}_9$B is preferably provided in the carrier gas rezero line $\mathbf{300}_9$B for facilitating a consistent and equal flow of carrier gas into the sensing chambers $\mathbf{70}_1$B and $\mathbf{70}_2$B of the testing cells $\mathbf{70}_1$ and $\mathbf{70}_2$ respectively. The capillary restrictor $\mathbf{60}_9$B is, as with the other capillary restrictors, preferably side mounted onto the block manifold **100**.

[0027] The sensor **200** is selected to measure the appropriate target-analyte (e.g., oxygen (O_2), carbon dioxide (CO_2) or water vapor (H_2O)). Selection of a suitable sensor **200** is well within the knowledge and expertise of a person having routine skill in the art. The sensor **200** is preferably a coulox sensor and is equipped with an exhaust valve **210** for preventing atmospheric contamination of the sensor when there is no flow of carrier gas to the sensor **200**.

I claim:

1. A target-analyte permeation testing instrument for measuring target-analyte permeation rate of a test film in test cell, the instrument having a target-analyte sensor and a plurality of test cells each defining a testing chamber with each test cell operable for retaining a test film to sealingly divide the testing chamber into a driving chamber and a sensing chamber, the target-analyte permeation testing instrument characterized by a block manifold (-) fixed to the plurality of cells, and (-) having a plurality of channels in fluid communication with the testing chamber of each cell, a pressurized source of driving gas, a pressurized source of inert gas, and the target-analyte sensor, wherein the plurality of channels are configured and arranged to (i) selectively carry driving gas from the pressurized source of driving gas to the driving chamber of each cell, (ii) carry driving gas from the driving chamber of each cell to a driving gas exit port in the manifold, (iii) selectively carry inert gas from the pressurized source of inert gas to the sensing chamber of each cell, and (iv) selectively carry inert gas from the sensing chamber of each cell to the target-analyte sensor.

2. The target-analyte permeation testing instrument of claim **1** wherein the block manifold further includes a refillable first water reservoir in selective fluid communication with the source of driving gas and in fluid communication with the driving chamber of each cell, and a second refillable water reservoir in selective fluid communication with the source of inert gas and in fluid communication with the sensing chamber of each cell.

3. The target-analyte permeation testing instrument of claim **1** wherein the block manifold is constructed from a single unitary metal block.

4. The target-analyte permeation testing instrument of claim **1** wherein the channels in fluid communication with the pressurized source of driving gas and the driving chamber of each cell are in fluid communication with at least one block mounted valve operable for effecting selective delivery of driving gas to the driving chambers of the cells.

5. The target-analyte permeation testing instrument of claim **1** wherein the channels in fluid communication with the pressurized source of inert gas and the sensing chamber of each cell are in fluid communication with at least one block mounted valve operable for effecting selective delivery of inert gas to the sensing chambers of the cells.

6. The target-analyte permeation testing instrument of claim **1** wherein the channels in fluid communication with the sensing chamber of each cell and the target-analyte sensor are in fluid communication with at least one block mounted valve operable for effecting selective delivery of inert gas from the sensing chambers of the cells to the target-analyte sensor.

* * * * *