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(54) **AUTOMATED CLAMP-ON SAMPLE CHAMBER FOR FLOW POROMETRY AND A METHOD OF USING SAME**

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(57) **ABSTRACT**

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A sample chamber includes a movable upper chamber. The movable upper chamber includes a center bore opening to a bottom of the chamber, at least one port for introduction of gas under pressure to the center bore, and a first annular seal around the center bore. A stationary lower seat opposing the upper chamber has a center bore aligned with the upper chamber and includes an exhaust and a second annular seal around the center bore. A test material is placed between the upper chamber and the lower seat. An actuator moves the upper chamber. When the upper chamber is moved down with the first annular seal in contact with an upper surface of a sample of the material and the second annular seal in contact with a lower surface of the sample, gas introduced to the upper chamber goes through the upper chamber and out through the exhaust.

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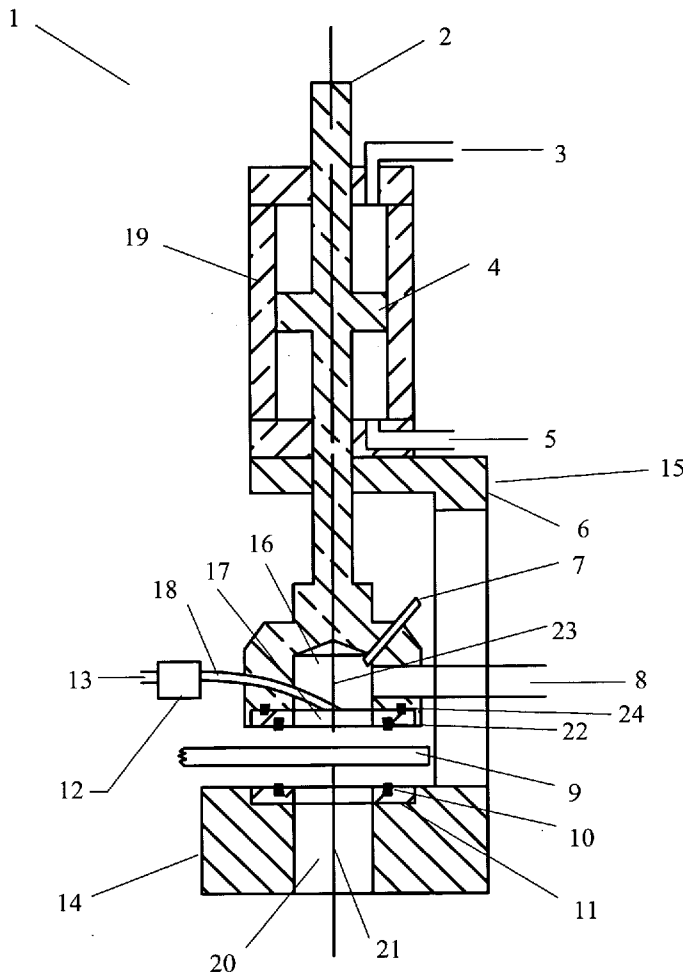


Fig. 1

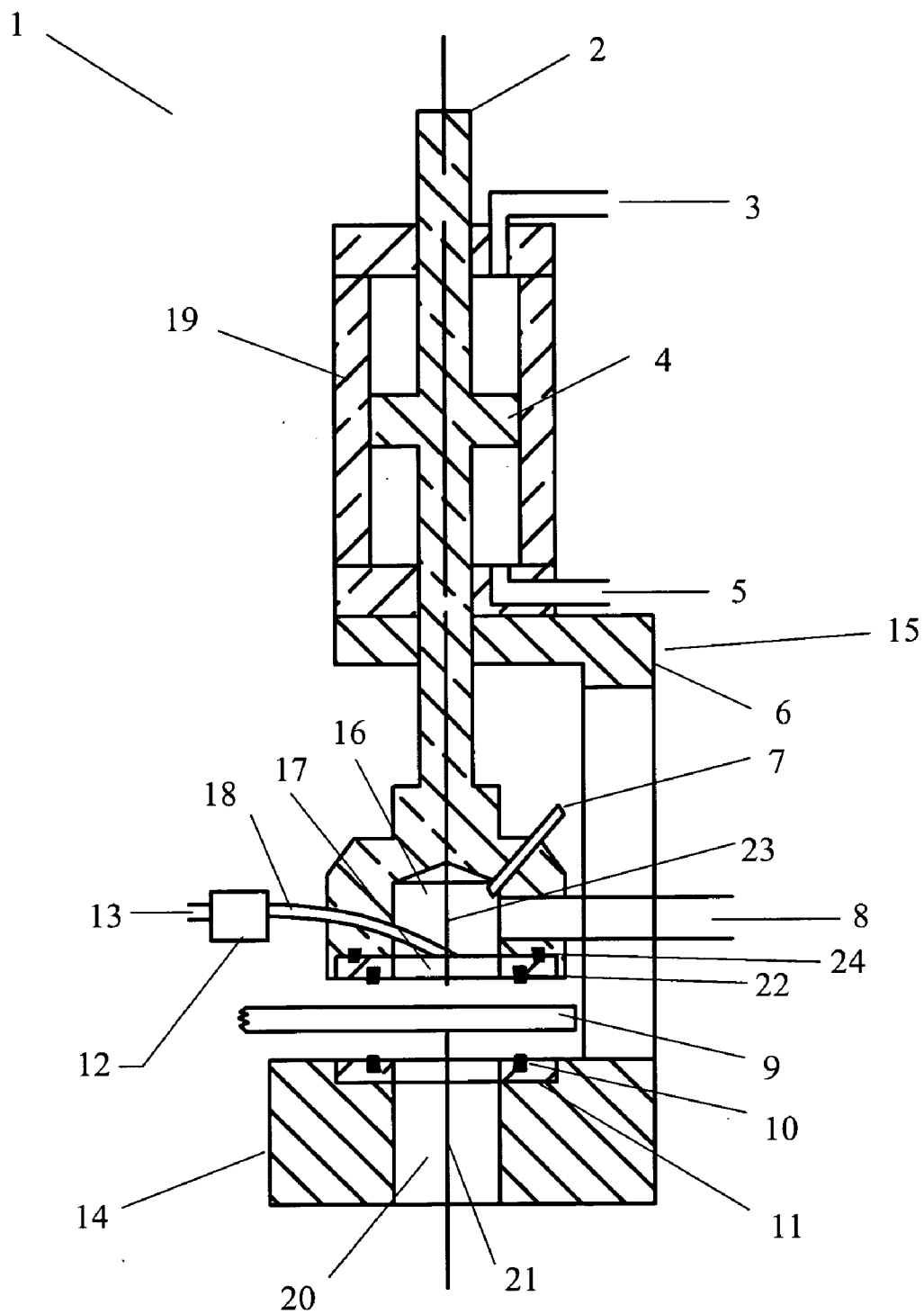
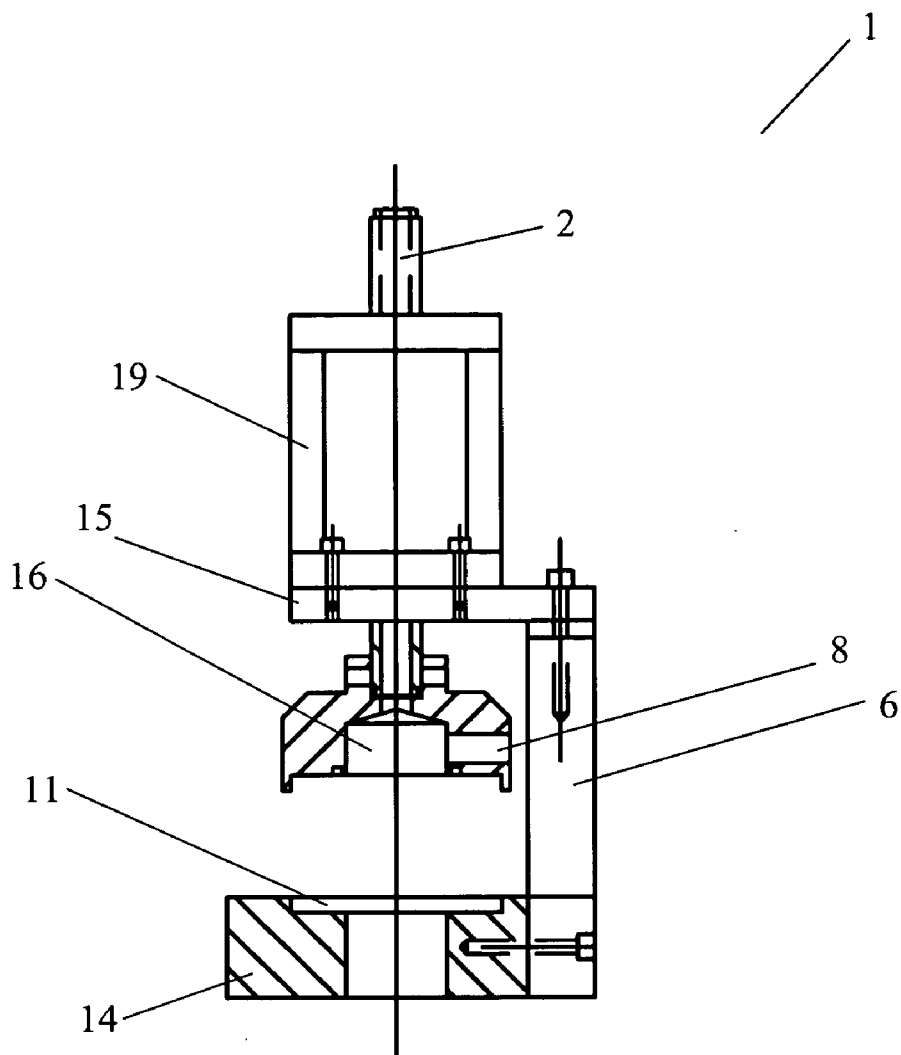


Fig. 2



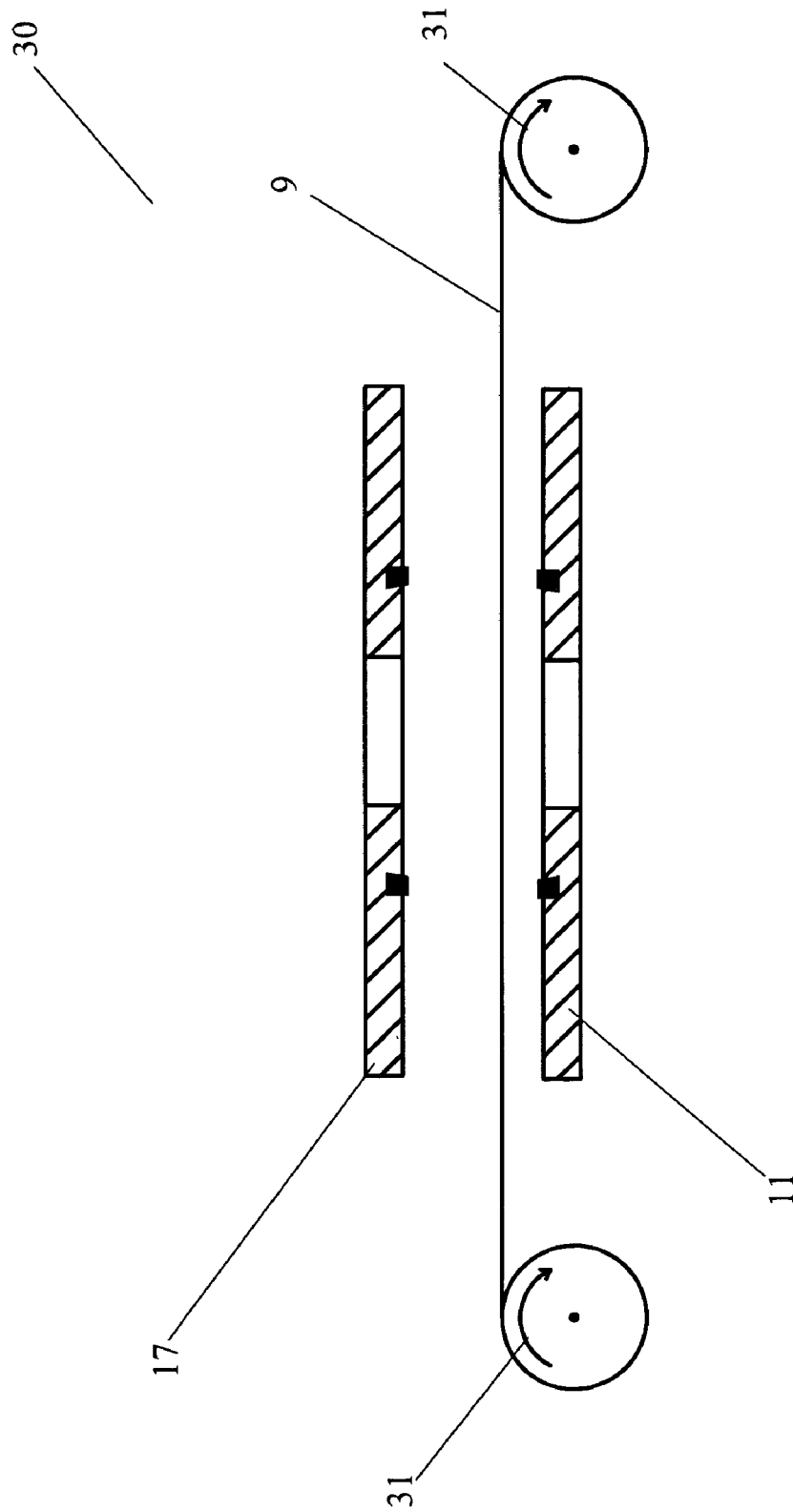


Fig. 3

Fig. 4

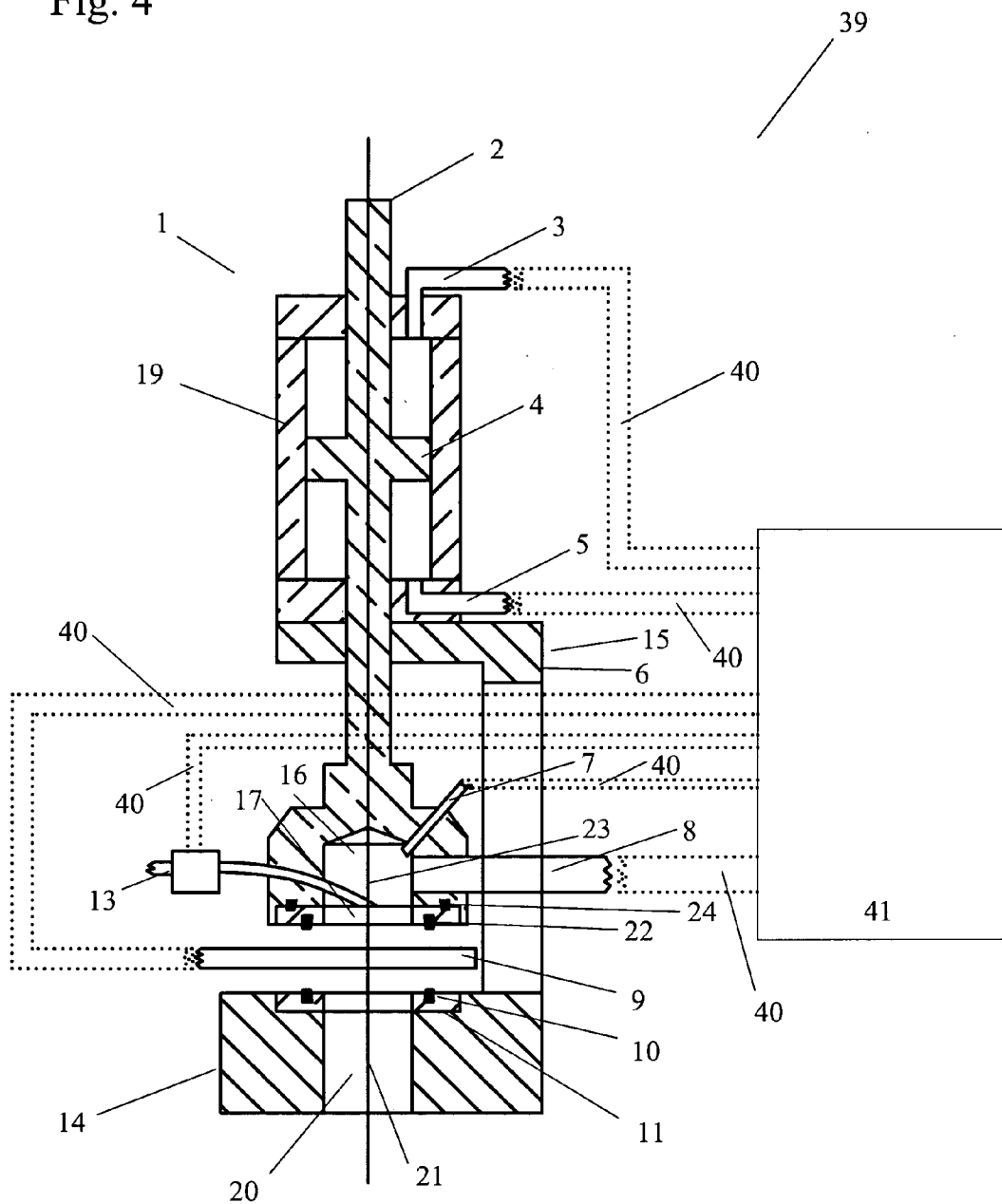
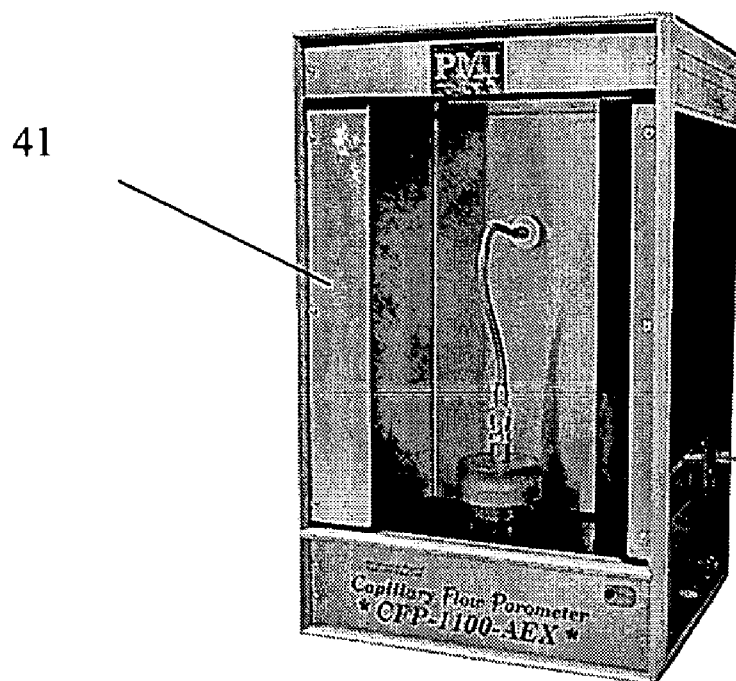


Fig. 5



AUTOMATED CLAMP-ON SAMPLE CHAMBER FOR FLOW POROMETRY AND A METHOD OF USING SAME

BACKGROUND OF THE INVENTION

[0001] 1. Field of the Invention

[0002] The invention pertains to the field of flow porometry. More particularly, the invention pertains to a sample chamber for flow porometry.

[0003] 2. Description of Related Art

[0004] Flow Porometry is a technique for measurement of pore diameter, pore distribution and gas permeability. In this technique, a wetting liquid is allowed to spontaneously fill the pores of the sample. The pressure of a non-reacting gas on one side of the sample is gradually increased to empty the pores of the sample and allow gas flow through the empty pores. The differential pressure required to remove liquid from a pore is given by:

$$p=4\gamma \cos \theta/D$$

[0005] where p is differential pressure, γ is surface tension of the wetting liquid, θ is the contact angle of the wetting liquid on pore surface and D is pore diameter. The differential pressure of the gas on the sample and the gas flow rates are measured. The differential pressure and gas flow rates through the dry sample with all of its pores open are also measured. The differential pressure yields the pore diameter. The differential pressure and gas flow rates through wet and dry samples yield the largest pore diameter, the mean flow pore diameter, the distribution of gas flow rate over pore diameter and gas permeability. This technique is used in industry for characterization of through pores, which are very important for various kinds of filtration applications in many industries, including chemical technology, biotechnology, food technology and nonwovens.

[0006] The PMI Capillary flow porometer (41), shown in FIG. 5, is a flow porometer. It regulates gas pressure, increases gas pressure on samples in small increments, accurately measures pressures and flow rates, automatically executes all operations, and acquires, stores and displays data in many formats. This instrument, with state-of-the-art components, many innovative design features and complete automation is capable of giving highly accurate, reliable and reproducible data.

[0007] However, to test a material in a flow porometer, samples are normally cut from the bulk material, which results in damage to the material. This also restricts the number of tests that can be performed on the material. The test involves loading the sample in the sample holder, sealing the sample to prevent leakage, measuring differential pressure and gas flow rate through the dry sample, wetting the sample, measuring differential pressure and gas flow rate through the wet sample and unloading the sample. Consequently, test time is often considerable and operator skill is important.

[0008] Therefore, there is the need in the art for a sample holder that overcomes the shortcomings of the prior art. More specifically, there is a need in the art for a fully automated sample chamber, which allows a sample to be loaded without cutting the bulk test material, and also allows testing to be repeated without removing the bulk material.

SUMMARY OF THE INVENTION

[0009] A sample chamber includes a movable upper chamber. The upper chamber includes a center bore opening to a bottom of the chamber, at least one port for introduction of gas under pressure to the center bore, a port for a pressure transducer, a tube for adding wetting liquid, and a first annular seal around the center bore. A stationary lower seat opposes the upper chamber and has a center bore aligned with the upper chamber. The lower seat includes an exhaust and a second annular seal around the center bore. A material to be tested is placed between the upper chamber and the lower seat. An actuator moves the upper chamber. When the upper chamber is moved down with the first annular seal in contact with an upper surface of a sample of the material and the second annular seal in contact with a lower surface of the sample, gas introduced to the upper chamber is constrained to go through the upper chamber and out through the exhaust.

[0010] A method of performing flow porometry uses the sample chamber, and includes the steps of placing a sheet of material between the upper chamber and the lower seat, moving the upper chamber down, applying pressure with the actuator until the first annular seal and the second annular seal create a gas-tight seal around the sample, and performing at least one flow porometry test by introducing gas through the port.

[0011] An apparatus for performing flow porometry includes a sample chamber. The sample chamber includes a movable upper chamber. The upper chamber includes a center bore opening to a bottom of the chamber, at least one port for introduction of gas under pressure to the center bore and a first annular seal around the center bore. A stationary lower seat opposes the upper chamber and has a center bore aligned with the upper chamber. The lower seat includes an exhaust and a second annular seal around the center bore. A material to be tested is placed between the upper chamber and the lower seat. An actuator moves the upper chamber. When the upper chamber is moved down with the first annular seal in contact with an upper surface of a sample of the material and the second annular seal in contact with a lower surface of the sample, gas introduced to the upper chamber is constrained to go through the upper chamber and out through the exhaust. The apparatus also includes a plurality of sensors for measuring flow and differential pressure, and a source of gas.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] FIG. 1 shows a sample chamber in an embodiment of the present invention.

[0013] FIG. 2 shows a schematic of the sample chamber of FIG. 1.

[0014] FIG. 3 shows testing of different locations by automatic feeding of material in the sample chamber.

[0015] FIG. 4 shows an apparatus for flow porometry using the clamp-on sample chamber in an embodiment of the present invention.

[0016] FIG. 5 shows a PMI flow porometer, used in an embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0017] The sample holder or sample chamber (1) of the present invention is illustrated in FIGS. 1 and 2. A sub-

stantially C-shaped support (6) preferably has as a part of its bottom (14) an adapter plate (11), which is preferably flat, containing an annular seal (10), which is preferably an O-ring seal or a gasket seal, and an opening (20) for the air to escape. The adapter plates are replaceable. Adapters containing different size O-rings allow the sample chamber (1) to test different sample areas. High porosity materials may require smaller areas to be tested. Also, homogeneity of a material could be evaluated by testing different areas. The bottom (14) preferably forms the stationary lower seat of the sample chamber (1). The test material (9), also known as the bulk material, to be tested is placed on the adapter (11). It is not necessary to cut a sample out of the test material (9) before the test material (9) is placed in the chamber (1).

[0018] The top part (15) of the support (6) allows an actuator, which is shown as a piston rod (2) in the figures, to move up or down. Other actuators include, but are not limited to, a rack and pinion, or a motor. Other mechanical and electrical means of moving the upper part of the sample chamber can alternatively be used. Because pressurized air is used in flow porometry, the air pressure-cylinder system shown in the figures is preferred. At the bottom of the piston rod (2) is a hollow chamber (16) and an adapter (17) similar to the one on the bottom part of the support (6). The hollow chamber (16) has a center bore (23). An annular seal (22), which is preferably an O-ring seal or a gasket seal, is also present on the bottom side of adapter. Another annular seal (24), which is preferably an O-ring seal or a gasket seal, is present on the topside of the adapter to prevent any leak between the adapter and the piston. Other seals for any of the annular seals (20), (22) or (24) include, but are not limited to, knife-edges and glue. O-ring seals and gasket seals are preferred, because application of pressure on flexible O-rings and gaskets can effectively seal many materials without damaging them.

[0019] The hollow chamber (16) and the annular seals (22) and (24) preferably form the upper movable chamber of the sample chamber. The bottom (14) of the C-shaped support opposes the hollow chamber (16) with a center bore (21) aligned with the chamber (16). The exhaust opening (20) is preferably at least as wide as the hollow chamber (16). When the upper movable chamber is moved down until the annular seal (10) is in contact with an upper surface of a sample and the second annular seal is in contact with a lower surface of the sample, gas introduced to the upper chamber is constrained to go through the upper chamber and out through the exhaust (20).

[0020] A flexible tube (8) is connected to the hollow chamber (16) to introduce gas that can flow through the sample. A pressure transducer (7) is preferably connected to the hollow chamber (16) to measure pressure close to the sample so that errors due to pressure drops in the flexible tube (8) are avoided. The exhaust (20) is always at atmospheric pressure. In order to minimize errors due to fluctuations in atmospheric pressure, differential pressure transducers with sensors both below and above the sample are alternatively or additionally used.

[0021] The hollow chamber (16) contains a small tube (18) through which wetting liquid (13) is added to the sample. A valve (12) connected to the tube (18) allows measured quantities of wetting liquid (13) to be discharged. The piston rod (2) passes through a housing, which is shown

as a cylinder (19) in the figures, and contains a piston (4) that moves up or down inside the cylinder (19). Openings on the top (3) and bottom (5) of the cylinder (19) are used for introducing pressurized gas. The pressurized gas inlet at the top (3) is used to move the piston (4) down, and the pressurized gas inlet at the bottom (5), is used to move the piston (4) up. The pressure of the gas introduced through the gas inlet (3) is preset to obtain a desired pressure on the O-rings and prevent sideways flow. The sample chamber (1) is preferably connected to a flow porometer, like the PMI Capillary Flow Porometer (41) shown in FIG. 5, which introduces the pressurized gas, as well as measuring and recording differential pressures and flow rates through the sample, by flexible tubes and wires.

[0022] An automatic feeding mechanism is preferably used in conjunction with the sample chamber (1) such that after each test the next test location is brought forward and moved to the top of the bottom adapter plate for testing. One such mechanism (30) is shown in FIG. 3.

[0023] An apparatus for performing flow porometry using the clamp-on sample chamber is shown in FIG. 4. The apparatus (39) includes the sample chamber (1) discussed above. A gas supply source supplies gas to the porometer (41). A flow sensor is preferably included in the porometer (41) to regulate the flow of gas into the sample chamber (1). Wetting liquid (13) is also introduced into the sample chamber (1). A pressure sensor (7) senses the pressure above the sample. Although not shown, in an alternative embodiment, a differential pressure sensor replaces the pressure sensor (7). The differential pressure sensor senses both the pressure above the sample and the pressure below the sample to determine a differential pressure. The sensors and the sample chamber are all preferably operatively connected to the porometer (41), which controls the apparatus. These connections (40) are shown by dotted lines in FIG. 4. The porometer (41) fully automates the apparatus.

[0024] A method of the present invention uses the novel sample chamber (1) described above, to perform the following steps:

- [0025] (1) Loading the test material without cutting out a sample such that the test location is on the adapter;
- [0026] (2) Sealing the material by introducing adequately pressurized gas at the top of the cylinder;
- [0027] (3) Measuring differential pressure and flow rate through the dry sample;
- [0028] (4) Wetting the sample;
- [0029] (5) Measuring the differential pressure and flow rate through the wet sample;
- [0030] (6) Unloading the sample; and
- [0031] (7) Repeating the procedure for the next test.

[0032] The method includes the steps of placing a sheet of material between the upper chamber and the lower seat, moving the upper chamber down, and applying pressure with the actuator until the first annular seal and the second annular seal create a gas-tight seal around the sample, and performing at least one flow porometry test by introducing gas through the port.

[0033] More specifically, the material (9) to be tested is first placed on the top of the adapter (11) on the bottom (14) of the support (6). The porometer (41) introduces pressurized gas (3) to the top of the cylinder (19). The piston (4) and the piston rod (2) are forced to come down. The adapter (17) at the bottom of the piston (4) presses on the O-ring (10) below the sample and seals the sample for sideways gas flow. The pressure on the O-ring (10) for obtaining a good seal is controlled by adjusting the pressure of the gas (3) introduced to the top of the cylinder (19). The porometer (41) then allows gas to be introduced (8) to the hollow chamber (16) in the piston rod (2). The gas flows through the sample and escapes through the opening at the bottom part (14) of the support (6). The porometer (41) measures and records differential pressures and flow rates through the dry sample.

[0034] Once this part of the test is done, the porometer (41) reduces gas pressure in the sample chamber to zero differential pressure and adds a predetermined amount of wetting liquid (13) to the sample. The porometer (41) then increases gas pressure and measures differential pressures and flow rates through the wet sample. Once the test is done, the porometer (41) releases pressure on the sample and then introduces pressurized gas at the bottom (5) of the cylinder (19) to raise the piston (4) and the piston rod (2). The piston rod (4) lifts the adapter (17) off of the test material (9). The material (9) is moved for the test to be performed at another location of the test material (9).

[0035] An automatic feeding mechanism (30), shown in FIG. 3, is preferably used in conjunction with the sample chamber (1) such that after each test the next test location is brought forward and moved to the top of the bottom adapter plate for testing. Once a test is complete, the porometer (41) automatically activates the feeding mechanism (30) to move (31) the next test location to proper positions and continues with the test procedure at the new location. In this example, the feeding mechanism moves (31) a roll of test material (9). After the actuator lifts the adapter off the test material, an electrical switch is turned on to activate the motor that drives the roll of bulk material. The motor is programmed to unroll the product and advance it by an amount necessary to move the next test location to test position. Thus, a large number of tests can be automatically performed.

[0036] The chamber of the present invention is fully automated. The feeding mechanism used in conjunction with the chamber can completely eliminate the need for human intervention. Results are objective, accurate and reliable. The time taken for a test is small. Operator involvement and the required operator skills are minimal. Large volume of testing could be automatically, quickly and efficiently performed in a cost effective manner without damaging or wasting any material.

[0037] Accordingly, it is to be understood that the embodiments of the invention herein described are merely illustrative of the application of the principles of the invention. Reference herein to details of the illustrated embodiments is not intended to limit the scope of the claims, which themselves recite those features regarded as essential to the invention.

What is claimed is:

1. A sample chamber for flow porometry, comprising:
 - a movable upper chamber comprising a center bore opening to a bottom of the chamber, at least one port for introduction of gas under pressure to the center bore and a first annular seal around the center bore;
 - a stationary lower seat opposing the upper chamber with a center bore aligned with the upper chamber, comprising an exhaust and a second annular seal around the center bore, wherein a material to be tested is placed between the upper chamber and the lower seat; and
 - an actuator for moving the upper chamber, wherein when the upper chamber is moved down with the first annular seal in contact with an upper surface of a sample of the material and the second annular seal in contact with a lower surface of the sample, gas introduced to the upper chamber is constrained to go through the upper chamber and out through the exhaust.
2. The sample chamber of claim 1, wherein the actuator is selected from the group consisting of a piston; a rack and pinion; and a motor.
3. The sample chamber of claim 1, further comprising a pressure transducer, connected to the upper chamber, which measures pressure close to the sample.
4. The sample chamber of claim 1, wherein the at least one port comprises a wetting port for introducing wetting liquid to the sample chamber.
5. The sample chamber of claim 4, further comprising a valve connected to the wetting port, which regulates a quantity of wetting liquid being discharged into the sample chamber.
6. The sample chamber of claim 1, wherein the at least one port comprises a pressure port for introducing gas under pressure.
7. The sample chamber of claim 1, further comprising a feeding mechanism, which moves the material in the sample chamber after flow porometry has been performed at a location on the sample.
8. The sample chamber of claim 1, wherein the first annular seal comprises an O-ring.
9. The sample of claim 1, wherein the second annular seal comprises an O-ring.
10. The sample chamber of claim 1, wherein the upper chamber further comprises an adapter plate located at a bottom of the upper chamber.
11. The sample chamber of claim 1, wherein the lower seat further comprises an adapter plate upon which the material to be tested is placed.
12. A method of performing flow porometry, using a sample chamber comprising a movable upper chamber comprising a center bore opening to a bottom of the chamber, at least one port for introduction of gas under pressure to the center bore and a first annular seal around the center bore, a stationary lower seat opposing the upper chamber with a center bore aligned with the upper chamber, comprising an exhaust and a second annular seal around the center bore, wherein a material to be tested is placed between the upper chamber and the lower seat, and an actuator for moving the upper chamber, wherein when the upper chamber is moved down with the first annular seal in contact with an upper surface of a sample of the material and the second annular seal in contact with a lower surface of the sample, gas

introduced to the upper chamber is constrained to go through the upper chamber and out through the exhaust, comprising the steps of:

- a) placing a sheet of material between the upper chamber and the lower seat;
- b) moving the upper chamber down, and applying pressure with the actuator until the first annular seal and the second annular seal create a gas-tight seal around the sample; and
- c) performing at least one flow porometry test by introducing gas through the port.

13. The method of claim 12, further comprising the steps of:

- d) measuring a first differential pressure and a first flow rate through the sample;
- e) reducing the first differential pressure to zero;
- f) wetting the sample;
- g) measuring a second differential pressure and a second flow rate through the sample; and
- h) unloading the sample.

14. The method of claim 13, wherein step d) comprises the substeps of:

- i) introducing gas into the upper chamber;
- ii) allowing the gas to flow through the sample and out the exhaust; and
- iii) measuring the first differential pressure and the first flow rate.

15. The method of claim 13, wherein step g) comprises the substeps of:

- i) introducing gas into the hollow chamber; and
- ii) allowing the gas to flow through the sample and out the exhaust;
- iii) measuring the second differential pressure and the second flow rate.

16. The method of claim 13, further comprising the step of repeating steps (a) through (h).

17. The method of claim 12, further comprising the step of d) moving the material to perform a test on another part of the material.

18. The method of claim 17, wherein step d) is performed using a feeding mechanism.

19. An apparatus for performing flow porometry using a clamp-on sample chamber, comprising:

- a) a sample chamber, comprising:
 - a movable upper chamber comprising a center bore opening to a bottom of the chamber, at least one port for introduction of gas under pressure to the center bore and a first annular seal around the center bore;
 - a stationary lower seat opposing the upper chamber with a center bore aligned with the upper chamber, comprising an exhaust and a second annular seal around the center bore, wherein a material to be tested is placed between the upper chamber and the lower seat; and

an actuator for moving the upper chamber, wherein when the upper chamber is moved down with the first annular seal in contact with an upper surface of a sample of the material and the second annular seal in contact with a lower surface of the sample, gas introduced to the upper chamber is constrained to go through the upper chamber and out through the exhaust;

- b) a plurality of sensors for measuring flow and differential pressure; and
- c) a source of gas.

20. The apparatus of claim 19, further comprising a flow porometer coupled to the sample chamber, wherein the porometer includes at least one of the sensors and a plurality of controllers, for automating the apparatus.

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