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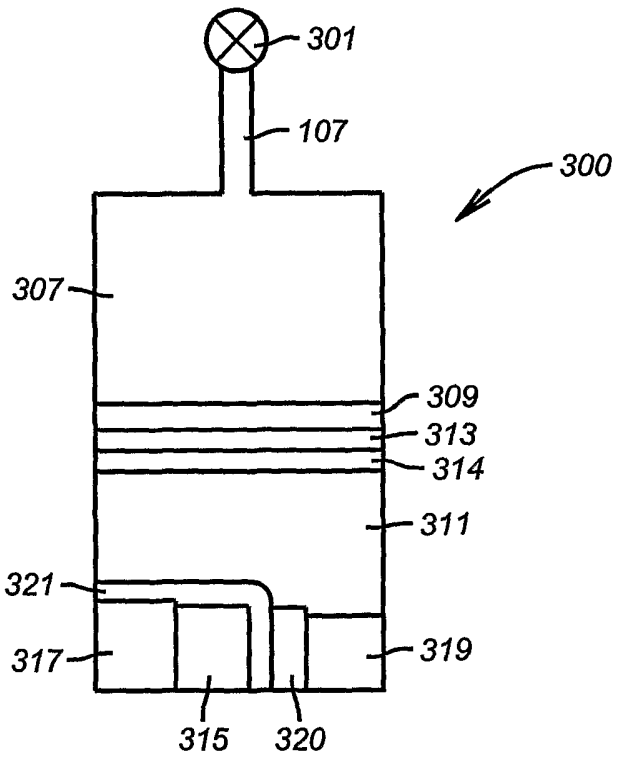
[Continued on next page]

(54) Title: A METHOD AND APPARATUS FOR DOWNHOLE DETECTION OF CO2 AND H2S USING RESONATORS COATED WITH CO2 AND H2S SORBENTS



(57) Abstract: A formation fluid sample is exposed to a rigidly-supported semi-permeable membrane such as silicone rubber to permit diffusion of gases and vapors from the formation fluid into a vacuum chamber, while at the same time, blocking the passage of any liquids. The membrane-transmitted gas is analyzed in the vacuum chamber by a resonator that reacts with it. The resulting change in resonant frequency of the resonator indicates the presence of a gas that reacts with it. An ion pump or sorbent is associated with the evacuated chamber to maintain the vacuum. The ion pump or sorbent removes gases and vapors from the low-pressure chamber, which have diffused into it from the reservoir sample that is on the opposite (high-pressure) side of the semi-permeable membrane.

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

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A METHOD AND APPARATUS FOR DOWNHOLE DETECTION OF CO2 AND H2S USING RESONATORS COATED WITH CO2 AND H2S SORBENTS

INVENTOR: ROCCO DIFOGGIO

Background of the Invention

Field of the Invention

[0001] The present invention relates generally to downhole reservoir characterization and in particular to a method and apparatus for real time identification of CO2 and H2S gases diffused out of a formation fluid sample. Formation fluid samples are obtained and gases are allowed to diffuse from these fluid samples through a silicone rubber layer backed by a sintered metal filter and perforated backing plate acting as semi-permeable membrane into an evacuated chamber. The gases are analyzed in the evacuated chamber by a resonator coated with a CO2 or H2S sorbent and a processor, which identifies gases such as CO2 and H2S and other gases or vapors extracted from a downhole reservoir fluid or sample.

Summary of the Related Art

[0002] To obtain hydrocarbons such as oil and gas, boreholes are drilled into the earth by rotating a drill bit attached at to the end of a drill string. Modern directional drilling systems generally employ a drill string having a bottom hole assembly (BHA) and a drill bit at an end thereof that is rotated by a drill motor (mud motor) and/or by rotating the drill string. A number of downhole devices placed in close proximity to the drill bit measure certain downhole operating parameters associated with the drill string. Such devices typically include sensors for measuring downhole temperature and pressure, azimuth and inclination measuring devices and a resistivity-measuring device to determine the presence of hydrocarbons and water. Additional downhole instruments, known as logging-while-drilling (LWD) tools, are frequently attached to

the drill string to determine the formation geology and formation fluid conditions during the drilling operations.

[0003] Commercial development of hydrocarbon fields requires significant amounts of capital. Before field development begins, operators desire to have as much data as possible regarding the nature of the hydrocarbon formation, in order to evaluate the reservoir for commercial viability. Despite the advances in data acquisition during drilling using the MWD systems and wireline analysis applications, it is often desirable to conduct further testing of the hydrocarbon reservoirs in order to obtain additional data. Therefore, after the well has been drilled, the hydrocarbon zones are often tested with other test equipment such as wireline tools, which are used to further analyze and monitor the formation.

[0004] One type of post-drilling test involves producing fluid from the reservoir and collecting such fluid samples downhole in tanks for transport to surface laboratories where Pressure-Volume-Temperature (PVT) studies and fluid properties such as density, viscosity and composition are measured. Also, one can measure the downhole fluid pressure at several depths and, from this pressure gradient, calculate the fluid's density.

[0005] Fluid samples extracted downhole are typically analyzed weeks to months later in a surface laboratory to identify and quantify gases present in the fluid. It is time consuming to retrieve fluid samples downhole and send them to a surface lab for analysis of gas content. Moreover, surface analysis requires removal of the fluid sample and the tool from the borehole for testing the sample before additional exploration and/or production activities occur. Thus, there is a need for a real-time

downhole method and apparatus for detection, distinction and quantification of gases in the formation.

SUMMARY OF THE INVENTION

[0006] The present invention provides a method and apparatus for real-time downhole detection, distinction and quantification of gases such as CO2 and H2S and other gases and vapors present in a formation fluid sample. The present invention exposes downhole high-temperature and high-pressure formation fluids to a silicone rubber filter backed by a sintered metal filter backed by a perforated metal plate, forming semi-permeable membrane, which blocks liquids but allows passage of certain gases and vapors. This membrane is mechanically supported by a rigid but porous and permeable structure such as a perforated metal plate. The perforated metal plate is capable of withstanding the pressure difference between vacuum and downhole pressures. The semi-permeable membrane is made of a material such as silicone rubber, which permits the diffusion of gases and certain vapors from the formation fluid sample, through the membrane and into a vacuum chamber adjacent the semi-permeable membrane.

[0008] The vacuum chamber forms a gas analysis chamber containing a resonator coated with a CO2 or H2S sorbent. A formation fluid sample is captured in a downhole tool and filtered by a semi-permeable membrane such as silicone rubber to permit diffusion of gases from the formation fluid into a vacuum chamber. The gases diffuse out of the formation fluid and analyzed by sorbent coated resonator situated in the evacuated portion of a gas analysis chamber.

[0009] An ion pump is associated with the evacuated gas analysis chamber to maintain a vacuum in the chamber. The ion pump removes gases, which have diffused from the formation fluid sample into the evacuated chamber on the opposite

side of the semi-permeable membrane filter. The ion pump can be turned off during the time that a gas concentration measurement is being made by the resonator. Alternatively, the ion pump can be left on but either physically positioned so as to restrict its pumping ability or electronically controlled so as to pump slow enough that the resonator has an opportunity to sense the gas before the pump has brought the vacuum back to its baseline value. In place of an ion pump, activated charcoal or some other sorbent could be used to prevent the gases that have diffused into the vacuum from lingering there too long and interfering with the measurement of subsequent gases that have evolved from the next sample.

BRIEF DESCRIPTION OF THE FIGURES

[0010] The novel features of this invention, as well as the invention itself, will be best understood from the attached drawings, taken along with the following description, in which similar reference characters refer to similar parts, and in which:

[0011] FIG. 1 is an illustration of an exemplary embodiment of the present invention as deployed in a borehole from a wireline;

[0012] FIG. 2 is an illustration of an exemplary embodiment of the present invention as deployed in a borehole from a drill string;

[0013] FIG. 3 is an illustration of the components comprising the current example of the invention;

[0014] FIG. 4 illustrates the semi-permeable membrane, sintered metal filter and metal plate with small hole having scoring of fact of plate between the holes;

[0015] FIG. 5 is a flow chart of functions performed in an example of the present invention;

[0016] FIG. 6 is a table showing some examples of gas diffusion rates through a suitable semi-permeable membrane for use with the present invention; and

[0017] FIG. 7 illustrates an alternative embodiment having a filter and capillary tube input.

DESCRIPTION OF AN EXEMPLARY EMBODIMENT

[0019] The present invention provides a method and apparatus for real-time downhole detection, classification and quantification of gases present in a representative formation fluid sample. Gases such as H2S and CO2 other gases and vapors present in a formation fluid sample are quantified by the present invention. The present invention exposes downhole high-temperature high-pressure formation fluid to a semi-permeable membrane such as silicone rubber to permit diffusion of gases from the formation fluid sample into a vacuum chamber containing a sorbent coated resonator.

[0020] The present invention analyzes high-temperature, high-pressure reservoir fluids by extracting and submitting a gaseous fraction of a formation fluid or fluid sample to a sorbent coated resonator. A formation fluid sample is acquired or captured and filtered through a semi-permeable membrane, such as silicone rubber to permit diffusion of gases from the formation fluid sample past the filter into a gas analysis chamber. In the present example of the invention the gas analysis chamber is evacuated to facilitate diffusion of gases from the formation or wellbore fluid into an evacuated gas analysis chamber. The diffused gas is analyzed by a sorbent coated resonator situated in the evacuated gas analysis chamber opposite the formation fluid chamber on the other side of the semi-permeable membrane. In the present example of the invention, an ion pump is associated with the evacuated gas analysis chamber to help establish and to maintain a vacuum in the chamber and to facilitate diffusion

of gases from the fluid to the gas analysis chamber by maintaining a lower concentration of gas on the vacuum side of the membrane than there is in the fluid on the high pressure side of the membrane. Diffusion of a gas through a membrane is proportional to the concentration gradient of that gas across the membrane. The ion pump removes gases from the evacuated chamber, which have diffused into the evacuated chamber from the formation fluid sample located on the opposite side of the semi-permeable membrane filter.

[0021] The first function is to evacuate a vacuum chamber containing a resonator and processor to analyze gases. The vacuum chamber also is provided with an ion pump to maintain the vacuum. A semi-permeable membrane (such as silicone rubber) is placed at the inlet to the vacuum chamber to allow gases to diffuse into the vacuum chamber, while at the same time preventing liquids from entering the evacuated chamber.

[0022] Turning now to **FIG. 1**, **FIG. 1** illustrates an example of the current invention deployed from a wireline **102** in a borehole **104** drilled in a formation **100**. An extensible probe **101** extracts fluid from the formation **100**. The extracted formation fluid flow through flow line **105** where the gas analysis chamber **300** of the present invention determines the gas content of the formation fluid sample. Stablizers **103** hold the tool **50** and extensible probe **101** in place during extraction of a formation fluid sample. The results of the gas analysis performed by a resonator **317** in gas analysis chamber **300** and processor **102**, can be acted on by processor **102** or the analysis results can be sent to the surface **51** to acted on by the surface processor and control unit **1000.** A well bore fluid can also be analyzed by extracting fluid from the well bore instead of the formation.

[0023] Turning now to **FIG. 2**, another example of the current invention is shown deployed from a drill string **201**. Straddle packers **203** hold the tool **50** in place during the entry of fluid through flow path **105** to the gas analysis chamber **300** of the present invention. The fluid can come from the annulus **105** between the tool **50** and the well bore **104** or from the formation **100**. Fluid can be routed to the sample tank **111** or back to the well bore annulus **105** as desired based on the results of the density determination performed in the gas analysis chamber **300** of the present invention. The results of the gas analysis chamber are acted on by the processor **102**, or the results can be sent to the surface **51** to acted on by surface processor and control **1000.** A well bore fluid can also be analyzed by extracting fluid from the well bore instead of the formation.

[0024] Turning now to **FIG. 3**, a more detailed schematic of the gas analysis chamber **300** of the present invention is shown. A sorbent coated resonator **317**, ion pump **319**, semi-permeable membrane **309**, fluid containment chamber **307** and processor **315** are shown in schematic form in **FIG. 3**. A sorption-cooling unit **321** is provided to maintain processor and resonator control electronics within their operating and/or survival temperature range. The formation fluid containment chamber **307** is separated from the evacuated gas analysis chamber **311** by the semi-permeable membrane **309.** Thus, the formation fluid containment chamber **307** is positioned on one side of the semi-permeable membrane **309** and an evacuated gas analysis chamber **311** on the other side of the semi-permeable membrane **309**. The gases present in the formation fluid sample diffuse across the semi-permeable membrane into the evacuated gas analysis chamber for analysis. Activated charcoal or other gas sorbent **320** is placed in the gas analysis chamber to adsorb gases to prevent them from lingering in the gas analysis chamber too long.

[0025] Formation fluid is extracted from the formation **100** or the well bore when the probe is not in contact with the well bore wall and enters into the fluid containment chamber **307** via flow line **107** and valve **301**. Gases diffuse from the formation fluid or well bore fluid on the fluid side of the semi-permeable membrane, through the semi-permeable membrane and into the evacuated chamber **311**. The gas analysis module equipment, resonator **317** and processor/control electronics **315** are located in the evacuated gas analysis chamber **311**. The gas is exposed to and analyzed by the resonator **317** and processor **102**. The processor **102** and resonator electronics control and conduct the analysis. The processor **102** reports the analytical results to the surface via the wireline or other means of downhole communication. The processor **102** can act on the analysis results without reporting the results to the surface. **FIG. 4** illustrates the semi-permeable membrane **309**, sintered metal filter **313** and metal plate **314** with small hole having scoring of fact of plate between the holes.

[0026] Turning now to **FIG. 5**, some of the functions performed by the present invention are illustrated. As shown in block **401**, the present invention captures a formation fluid sample from the formation. The formation fluid enters the tool **50** via a flow line in fluid communication with the formation. In block **403**, the gas analysis chamber is evacuated. The evacuation of the gas analysis chamber enables gases present in the formation fluid sample to diffuse from the fluid as gas into the evacuated chamber through the semi-permeable membrane. In block **405** the semi-permeable membrane between the fluid and the evacuated chamber allows gases from the fluid to diffuse through the semi-permeable membrane into an evacuated gas analysis chamber. In block **407**, the resonator and processor of the present invention monitors the gases to detect, identify and quantify the gases and distinguish between them. In block **409**, the ion pump removes excess or residual diffused gases from the

evacuated side of the chamber to maintain the vacuum in preparation for the next measurement.

[0027] Sensors for CO2 and H2S are usually meant to operate in air or in vacuum. It is difficult to detect these gases while they are dissolved in crude oil. Thus a silicone rubber layer or other polymeric separation membrane, followed by a sintered stainless steel filter and a steel plant with a few holes in it covering a vacuum chamber are provided to separate gas and fluid. Thus, the gases can diffuse out of the crude oil and into a vacuum chamber containing the resonator. The chamber also contains activated charcoal **320** or other sorbent to prevent the gases from lingering too long in the chamber. When mass from diffused gas is deposited on the resonator, such as a tuning fork as used in the present example of the invention, the resonant frequency for the resonator is lowered. Thus, by coating a resonator with a material that selectively reacts with a particular gas, the presence of the particular gas can be detected by monitoring the resonate frequency of the resonator. In one embodiment the resonator is coated with silver, which reacts with H2S to produce black silver sulfide or tarnish and makes the resonator heavier, thus lowering it resonant frequency. In this case, a drop in the silver-coated resonator resonant frequency would indicate the presence of H2S. In another embodiment, the resonator is coated with copper or zinc or some other H2S reactive metal. In another embodiment, a resonator is coated with a sodium oxide to produce Na2CO3 when exposed to CO2, thus making the resonator heavier. In this case a reduction in the resonator resonant frequency would indicate the presence of CO2. A coating of tetramethylammonium fluoride tetrahydrate (TMAF) is useful for the detection of CO2 although TMAF also has some sensitivity to H2S.

[0028] Suitable semi-permeable membranes, activated charcoal sorbents and ion pumps are commercially available suitable for use with the present invention are discussed herein. Furthermore, membranes can be specially designed to be selective to the transmission of one gas instead of transmitting many gases as silicone membranes do. The invention enables diffusion and separation of CO2 and H2S from crude oil at high pressure and temperature and letting this gas diffuse through a semi-permeable membrane filter. In the high pressures of the downhole environment, membranes must be supported, thus, the present invention provides a sintered stainless steel filter analogous to sandstone with the sand particles replaced with steel particles. The sintered metal filter is porous and permeable. The sintered metal filter is backed by a perforated steel plate for rigid stability against the pressure of the fluid from which gases diffuse.

[0029] Separation membrane technology is discussed by Sandra Young of the School of Polymers at The University of Southern Mississippi, (see, e.g., http://www.psrc.usm.edu/mauritz/diffuse.html), which states:

> Aromatic polyimides that contain -C(CF3)2- groups tend to have higher preference for CO2 relative to CH4. Introduction of -C(CF3)2- groups is believed to increase chain stiffness which reduces intrasegmental mobility, and reduce and limit the degree of chain packing by increasing the free volume, serving as molecular spacers and chain stiffeners in the polymer (Stern, S.A. J. Membrane Sci., 1994, 94, 1-65 and Kim, T.H.; Koros, W.J.; Husk, G.R.; O'Brien, K.C. J. Membrane Sci., 1988,37, 45-62).
>
> Polysulfones have been used for years as perm-selective membranes, starting in 1977 when Monsanto utilized asymmetric hollow fiber coated with a thin layer of silicone rubber for H2 separations. Asymmetric cellulose acetate membranes are used for the removal of CO2 and H2S from natural gas. CO2 and H2S have high solubility in cellulose acetate, which induces pseudo-plasticization, causing the polymer to swell with disruption of the polymer matrix, which increases the mobility of the polymer chains. In the area of rubbery polymers, the only systems currently under investigation are the poly(organosiloxanes). Poly(organosiloxanes) have been studied in detail because of the vast utility of polydimethylsiloxane (PDMS) as a pre-formed

membrane that can then be used as a template for IPN formation in gas or liquid separation processes. PDMS possesses one of the greatest permeability coefficients of any polymer, due to its large free volume, and low selectivity. Through copolymerization, properties have the potential to be tailored to suit specific separation needs. Porosity control in materials used for separation processes is essential due to the potential variability of gases or liquids through the membrane. Sol-gel polymerizations can be manipulated to adjust the shrinkage of a network for the development of controlled porosity inorganic materials.

[0030] John J. Pellegrino of National Institute of Standards and Technology states:

http://membranes.nist.gov/publication_abstracts/Pell_Ko_Nass_Eine.html

> CO2 and H2S can be selectively separated from each other and from non-polar gases, such as H2, CO and CH4 using chemically reactive carriers immobilized in a membrane phase. Ion-exchange membranes made from polyperfluorosulfonic acid (PFSA) have been modified to form a gel for use as the support for the solvent and carrier. The membrane contains hydrophilic regions into which a solvent, containing the desired chemical complexing agent, may be imbibed. In experiments performed at ambient conditions selectivities for CO2 versus H2 are 20 to 30 with CO2 permeabilities of 1000-2000 barrer. [1 barrer = 10^{-10} $cm^2 \cdot s^{-1} \cdot cmHg^{-1}$] Higher selectivities and H2S permeabilities are obtained for the H2S - H2 separation. Our studies include characterization of this membrane with a variety of amine carriers and polar solvents at ambient temperatures and pressure. This paper presents a summary of the acid gas permeation rates and selectivities for the acid gases versus H2 and CO. Preliminary economic evaluations indicate that composite membranes with PFSA coated films 5 to 1 μm thick, would have capital costs lower than standard amine-absorber technology.

[0031] **FIG. 6** is a tabular listing and specification for some gases through a representative semi-permeable membrane, which is suitable for use with the present invention. The specifications for some small commercially available resonators and small ion pumps are discussed below. **FIG. 7** illustrates an alternative embodiment having a filter **316** and capillary tube **318** input to evacuated gas analysis chamber **311**. There is typically a tradeoff between the speed of response to gases in a fluid and the thickness of the semi-permeable membrane.

[0032] In another embodiment of the present invention, the method of the present invention is implemented as a set computer executable of instructions on a computer

readable medium, comprising ROM, RAM, CD ROM, Flash or any other computer readable medium, now known or unknown that when executed cause a computer to implement the method of the present invention.

[0033] While the foregoing disclosure is directed to the preferred embodiments of the invention various modifications will be apparent to those skilled in the art. It is intended that all variations within the scope of the appended claims be embraced by the foregoing disclosure. Examples of the more important features of the invention have been summarized rather broadly in order that the detailed description thereof that follows may be better understood, and in order that the contributions to the art may be appreciated.

What is claimed is

1. A method for monitoring the presence of a particular gas in a fluid downhole comprising:

diffusing gas from the fluid downhole into a gas analysis chamber;

exposing the gas to a resonator that adsorbs with the particular gas;

monitoring the resonant frequency of the resonator to detect a change in the resonant frequency after being exposed to the gas; and

estimating the presence of the particular gas in the fluid downhole based on the change in the resonant frequency.

2. The method of claim 1, further comprising:

coating the resonator with a sorbent that adsorbs the particular gas.

3. The method of claim 1, wherein:

the gas is diffused through a semi-permeable membrane.

4. The method of claim 3, wherein:

backing the semi-permeable membrane is backed with a sintered metal.

5. The method of claim 3, wherein:

backing the semi-permeable membrane is backed with a perforated backing plate.

6. The method of claim 2, wherein the particular gas is CO2.

7. The method of claim 2, wherein the particular gas is H2S.

8. The method of claim 3, wherein the semi permeable membrane comprises a gas selectable semi permeable membrane

9. The method of claim 1, further comprising:

removing gases from the gas analysis chamber.

10. The method of claim 9, wherein removing is performed by:

pumping gases from the gas analysis chamber.

11. An apparatus for monitoring the presence of a particular gas in a downhole fluid comprising:

a membrane for diffusing gas from the downhole fluid into a gas analysis chamber;

a resonator within the gas chamber; and

a processor for monitoring the resonant frequency of the resonator to detect a change in the resonate frequency after being exposed to the gas and estimating the presence of the particular gas in the fluid downhole based on the change in the resonate frequency.

12. The apparatus of claim 11, wherein the resonator further comprises:

a sorbent that adsorbs the particular gas wherein the processor monitors the resonant frequency of the coated resonator to detect the presence of the particular gas in the fluid downhole.

13. The apparatus of claim 11 wherein the membrane is semi-permeable.

14. The apparatus of claim 13, wherein the membrane further comprises:

a sintered metal backing the semi-permeable membrane.

15. The apparatus of claim 13, wherein the membrane further comprises:

a perforated backing plate backing the semi-permeable membrane.

16. The apparatus of claim 12, wherein the resonator further comprises:

a CO2 sorbent for coating the resonator to detect the presence of CO2.

17. The apparatus of claim 12, wherein the further comprises:

a H2S sorbent that coats the resonator to detect the presence of H2S.

18. The apparatus of claim 13, wherein the filter only allows certain gases to diffuse from the fluid.

19. The apparatus of claim 11, further comprising:

charcoal in the gas analysis chamber for adsorbing gases from the gas analysis chamber to prevent the gases from lingering in the gas analysis chamber.

20. The apparatus of claim 11, further comprising:

an ion pump for pumping gases from the gas analysis chamber to prevent the gases from lingering in the gas analysis chamber.

21. A system for monitoring the presence of a gas in a downhole fluid comprising:

a downhole tool;

a membrane for diffusing gas from a fluid downhole into a gas analysis chamber in the downhole tool;

a resonator for detecting the presence of a gas in the fluid downhole; and

a processor for monitoring the resonant frequency of the resonator to detect a change in the resonate frequency after being exposed to the gas and detecting the presence of a gas based on the change in the resonate frequency.

22. The system of claim 21, wherein the membrane comprises a semi permeable membrane.

23. The system of claim 21, wherein the membrane comprises a gas selective membrane.

24. The system of claim 21, wherein the resonator is coated with a sorbent to detect the presence of a gas.

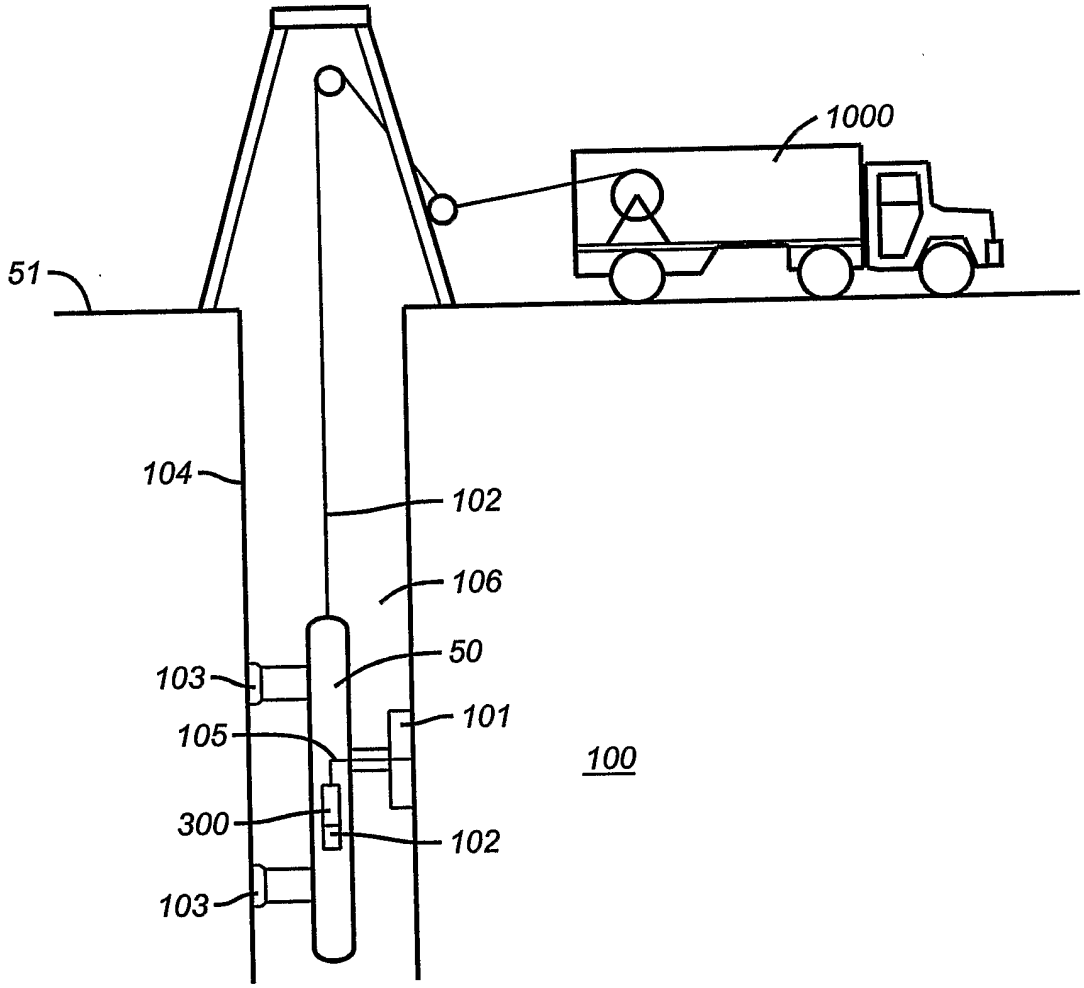

FIG. 1

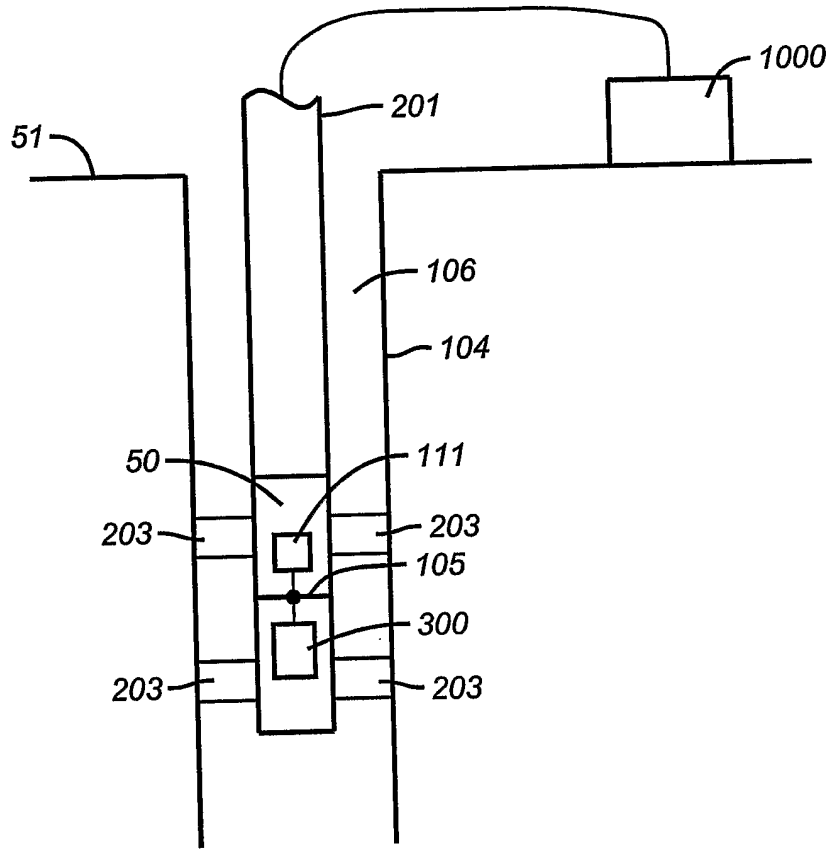

FIG. 2

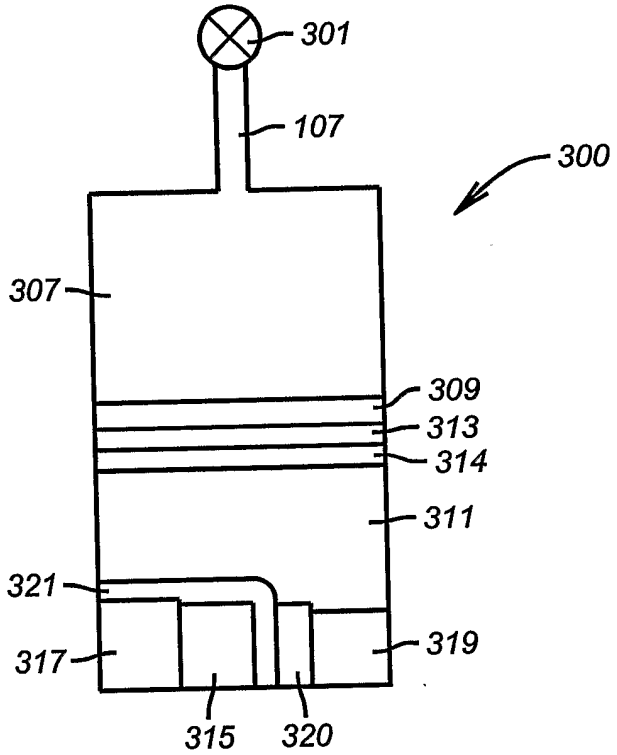

FIG. 3

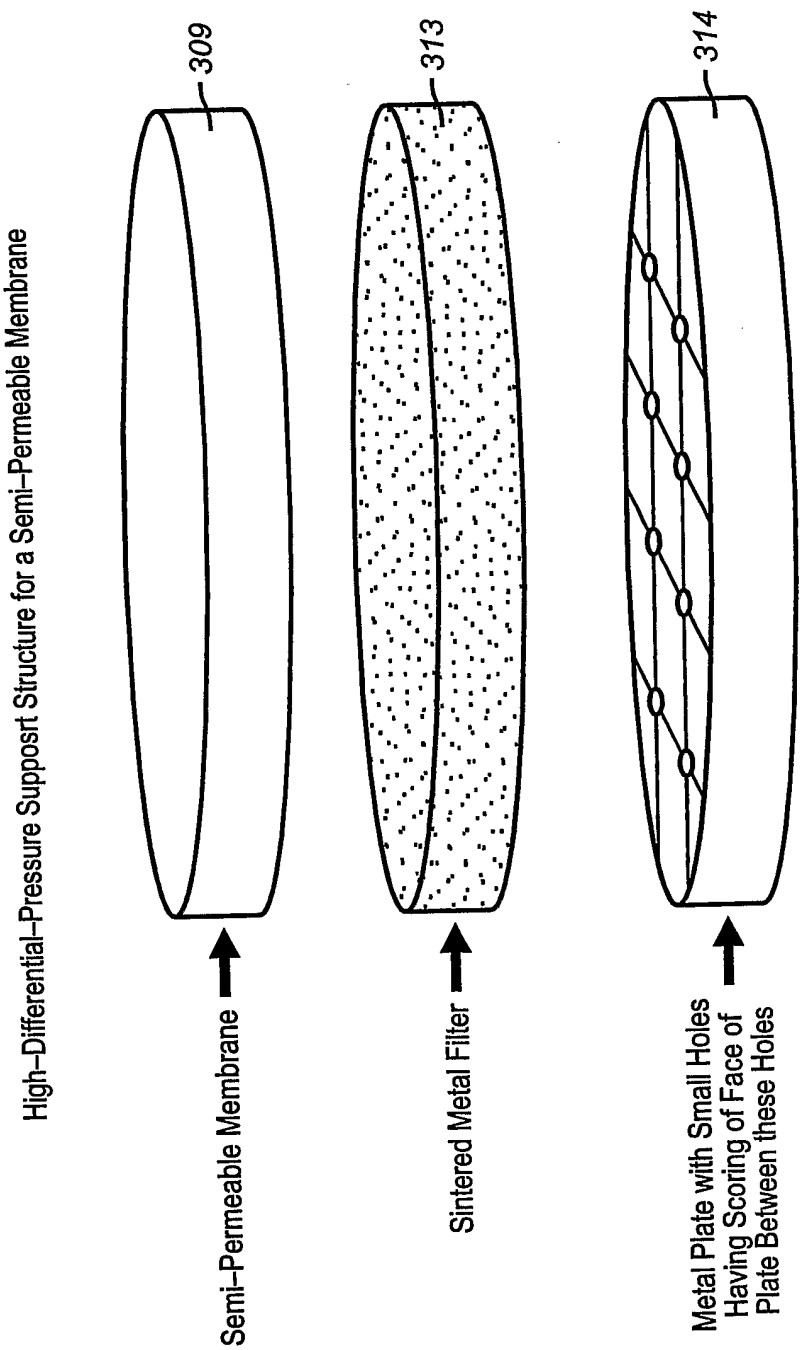
High–Differential–Pressure Supposrt Structure for a Semi–Permeable Membrane
309
313
314
Semi–Permeable Membrane
Sintered Metal Filter
Metal Plate with Small Holes Having Scoring of Face of Plate Between these Holes

FIG. 4

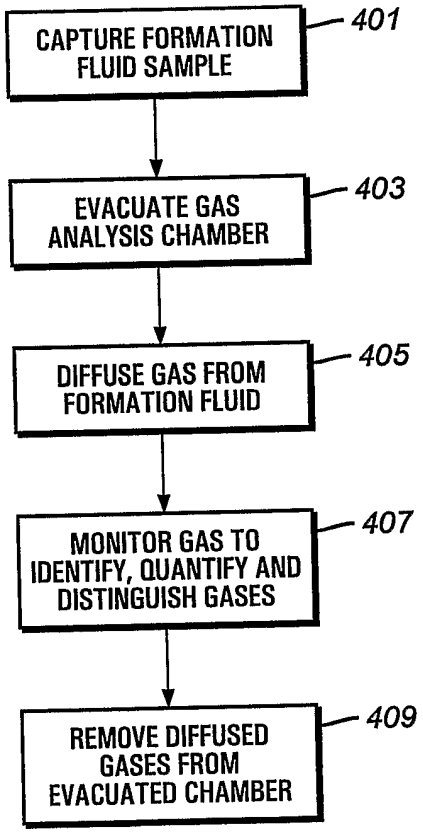
CAPTURE FORMATION FLUID SAMPLE
401
EVACUATE GAS ANALYSIS CHAMBER
403
DIFFUSE GAS FROM FORMATION FLUID
405
MONITOR GAS TO IDENTIFY, QUANTIFY AND DISTINGUISH GASES
407
REMOVE DIFFUSED GASES FROM EVACUATED CHAMBER
409

FIG. 5

Gas Permeabilities 10^{-9} cc gas (RTP) cmsec cm^2cm Hg DP

GAS	SSP-M100	GAS	SSP-M100
H_2	55	n-$C_{10}H_{22}$	360
He	30	HCHO	925
NH_3	500	CH_3OH	1160
H_2O	3000	$COCL_2$	1250
CO	30	Acetone	490
N_2	25	Pyridine	1595
NO	50	Benzene	900
O_2	50	Phenol	1750
H_2S	840	Toluene	760
Ar	50	Xe	171
CO_2	270	CCl_4	5835
N_2O	365	CH_2O	925
NO_2	635	Freon 11	1290
SO_2	1250	Freon 12	107
CS_2	7500	Freon 22	382
CH_4	80	Freon 114	211
C_2H_6	210	Freon 115	51
C_2H_4	115		
C_2H_2	2200		
C_3H_8	340	GAS	SSP-M213**
n-C_4H_{10}	750	CO_2	97
n-C_5H_{12}	1670	H_2	21
n-C_6H_{14}	785	O_2	16
n-C_8H_{18}	715	N_2	7

FIG. 6

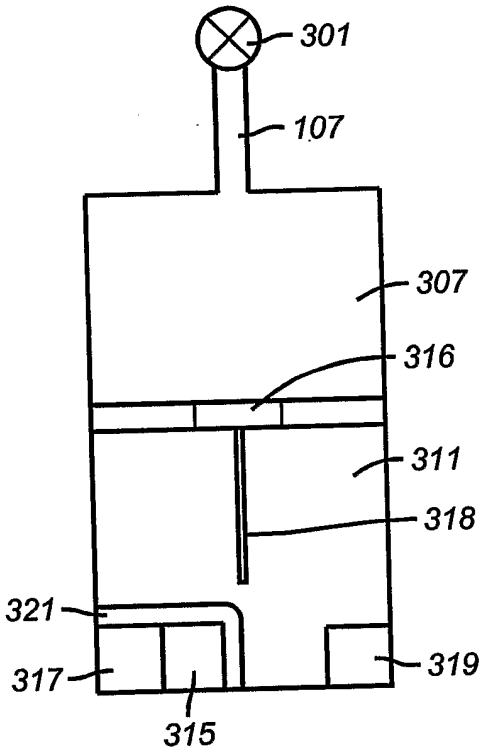

FIG. 7