Subsurface Fluids Screening By An Analytical System Employing A Diffusion-Limited And Implantable Sampling Module Deployable With A Cone Penetrometer

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Daniel P. Lucero², Ralph H. Ilgner¹, Rob R. Smith¹, and Roger A. Jenkins¹ ¹Chemical and Analytical Sciences Division, Oak Ridge National Laboratory*, P. O. Box 2008, Mail Stop 6120. Oak Ridge TN 37831-6120 Phone (423)574-4905 FAX (423)576-7956 IGN@ornl.gov ²Consulting Thermodynamicist, 18421 Cedar Drive, Triangle, VA 22172

ABSTRACT

An analytical system employing a diffusion-limited sampling module and a direct sampling ion trap for quantitative assessment of subsurface fluids was developed and field tested. The sampling module is deployable with a cone penetrometer. It can be retrieved and/or remain as an implant for an indefinite time period. The device geometry, comprised of two planar membranes enclosing a diffusion cell, provides good implant ruggedness and reliable service in the field. Also, the sampling module is protected within a push pipe housing to extend implant service life. Subsurface volatile organic compound (VOC) vapors, in nanoliter amounts, diffuse through the sampler membrane wall by a diffusion-limited process that is independent of the soil permeability. Sample vapors are transported to the surface for analysis by direct sampling ion trap, or other analytical devices. Metered pressurized or reduced pressure transport (carrier) gas is utilized for sample transport to the surface. The vapors obtained are a function only of the fluid partial pressure and the vapor conductance of the sampler. Thus, quantitative analytical data is obtained regardless of soil conditions. The sampling module was deployed in the field at Dover Air Force Base at depths of 5 to 8.5 feet by the US Army Site Characterization and Analysis Penetrometer System (SCAPS). Relatively small 1.75 inch diameter push pipe and the relatively small vapor samples extracted cause minimal soil disturbance which preserves the integrity of the sampler subsurface surroundings. Analytical results were obtained for the system sampler operating in real time and as an implant where equilibrium was obtained between sampler interior and the external surroundings. Results in the real time and implant mode correlated to within 10%. The utility of the entire analytical system, deployed with a US Army cone penetrometer, was satisfactorily demonstrated. No plugging of the membranes, calibration gas ports, or transport gas ports was noted during field implantations. More field applications are required to more conclusively demonstrate implant utility for more widespread applications including ground water analysis.

INTRODUCTION

A major problem in the cleanup process or assessment of sites contaminated by hazardous and other wastes stems from the lack of information regarding the sites subsurface characteristics. A substantial amount of information can be obtained from a general prospecting assay by means of a soil gas screening survey 1,2,3 . This information is used to:

1) examine subsurface contamination with volatile compounds:

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- 2) develop a general properties survey of site hazardous fluids; and,
- 3) characterize the soil contamination plume in terms of its volumetric and aerial extent, mobility and composition. ę.

The screening is performed with sampling and analytical tools. Generally, a representative sample of the soil gas is obtained and introduced into an analytical instrument that responds to the particular soil gas species. "The submitted manuscript has been authored

The basic requirements for a soil gas survey system are that it:

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- 1) obtain a representative soil gas sample for analysis;
- 2) disturb minimally the surrounding subsurface soil; and,
- 3) is a reasonably low cost operation.

A soil gas sample is obtained by collecting a sample of the soil itself or extracting a sample of soil gas downhole and transporting it to the surface for immediate onsite or subsequent analysis. A more ideal approach is that of performing the sampling and analysis downhole if reliable equipment can be deployed downhole at a reasonable cost with minimal soil disturbance. There are many aspects of a survey system which lead to meeting these requirements for individual applications ^{4,5}.

The soil gas survey system described in this work comprises two distinct modules for the onsite analytical processes: a special purpose downhole sampling module and a direct sampling ion trap mass spectrometer (DSITMS)^{6,7}. This system was successfully tested in the field and met the requirements listed above.

SOIL GAS SURVEY SYSTEM

A schematic illustration of the deployed system is given in Figure 1. A passive dual planar membrane organics monitor (PDPMOM), the sampling module, is in the subsurface zone and the analyzer, a DSITMS, is shown at the surface. The sampling module is connected to the surface by four pneumatic lines, the carrier or transport gas, calibration gas and return lines. Soil gas enters PDPMOM by a molecular diffusion process and is lifted to the surface through the return line. Transport gas from the surface flows through the transport line, the sampling module and the return line to the analyzer. Its flow rate is controlled and can be induced by either a pressurized or reduced pressure technique. Any analytical instrument that responds satisfactorily to the soil gas species in terms of its lower detection limit, specificity and time rate of response requirements can be used to perform the analysis.

Membrane Tests

A brief discussion of membrane testing provides some device development history, which may be relevant to calibration protocols. Membrane characterizations were conducted in the laboratory in an effort to find a membrane material with optimum properties for this water or soil gas monitoring device. The experimental apparatus consisted of a membrane test cell configured to simulate realistic field conditions as closely as was practical in the laboratory. The laboratory test cell was challenged with standard volatile organic compounds (VOC) to simulate soil gases surrounding an implanted device. Experimental results with the best membrane material obtained to date, challenged with typical hazardous soil gases, are given in Table 1.

Membrane Configuration

Desirable membrane properties include low permeability coefficients (P_m) for typical soil gases so that soil gas species mass transfer rates remain low relative to mass transfer rates through any soil, thus limiting sampling mass and sample depletion in the soil. This describes the most defining principal of PDPMOM⁸ or its predecessor, TerraTrog^{9,10,11}. These devices place a limit on sampling mass with a suitable membrane system ensuring quantitative soil gas measurements in any soil or aqueous matrix regardless of matrix permeability to hazardous soil gas species. The membrane is the limiting factor, this differentiates these from other sampling systems.

Membranes with low permeability coefficients, P_m , to vapor phase organic compounds, were selected for further tests. Fast membrane response rate is another desirable membrane property ensuring rapid soil gas quantitations in the field. Membrane time constants (θ_c), measured in the laboratory, for given organic compounds are good experimental indicators of membrane response rates under field conditions, see Table 1. A membrane time constant, θ_c , is defined as the time required to reach 0.63 of maximum dynamic mode signal, obtained with continuous transport gas flow, measured from the time a membrane was first challenged with a VOC concentration step change outside the diffusion cell. See the

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portion of Figure 2 illustrating typical dynamic mode response. Membranes were sought with short time constants for typical VOC. A thin film polyimide membrane proved to be the best membrane system tested to date. The most important feature that sets PDPMOM apart from TerraTrog is the less complicated membrane configuration. The device geometry, with the capability of reliably sealing two planar membranes, thus enclosing a diffusion cell, is an important advance which should give better implant ruggedness and reliable service in the field. An additional benefit of planar construction is simplified membrane "engineering". The often mutually exclusive properties of fast rate of response and low sample mass transfer rates, high membrane impedance, require more sophisticated fabrication techniques. This is minimized through the planar design. These high technology membranes may not be suitable for configurations in more complex shapes requiring manufacturing processes such as heat sealing.

Another important feature is that PDPMOM is protected within a hardened push pipe housing which should protect the more delicate membrane systems during implantation and extend implant service life, while allowing rapid soil gas entry into the implant space outside the membrane cell. In contrast, TerraTrog is designed to be implanted directly.

PDPMOM

A cross-sectional illustration of the PDPMOM diffusion chamber and an exploded view of the device is shown in Figures 3 and 4 respectively. It comprises a segmented rectangular chamber approximately 20 cm long, 1 cm wide and 0.3 cm thick. Pneumatic fittings are attached at one end for the transport and return lines. The flat side walls comprise thin semipermeable pin-hole free membranes, an assembled view of the sampling module is shown in Figure 5. Soil gas enters the chamber by diffusion, mixes with the incoming transport gas and flows to the surface where the soil gas sample flows into an analyzer. The soil gas flow rate is diffusion limited by membrane impedance to VOC which is orders of magnitude greater than soil impedance ¹². This feature enables the sampler to obtain a quantitative amount of soil gas that is proportional to the partial pressure of the soil gas. Thus, the soil gas concentration measurement of the transport gas is related directly and quantitatively to the soil gas pressure ^{13,14}.

Two modes of sampling the soil gas are available with the use of PDPMOM, the dynamic mode and the static, Figure 2. The dynamic mode is the sample obtained from the condition where the soil gas flow rate into the chamber has reached a steady state level with the transport gas flowing through the system. The static mode is the sample obtained from the condition where soil gas has ceased flowing into the chamber without transport gas flowing through the system, i.e., that sample obtained by implanting the sampler in the soil.

At dynamic equilibrium, the soil gas pressure is related to soil gas concentration measured in the transport gas stream:

$$\mathbf{P}_{sg} = (\mathbf{Q}_t / \mathbf{KP}_a) \mathbf{P}_t$$

where

 $P_{sg} = soil gas pressure, ppb (v/v);$

 P_t = soil gas pressure measured in the transport gas stream, ppb (v/v);

 $Q_t = \text{transport gas stream flow rate, mL/min.};$

K = soil gas species diffusion conductance of PDPMOM chamber membrane walls, ml/min-torr; and, (1)

 P_a = ambient total pressure, torr.

The time required to reach dynamic equilibrium after a step change in soil gas pressure or concentration is:

where

$$T_{de}$$
 = time for the sampler to attain dynamic equilibrium, min.;

t = membrane wall thickness, cm; and,

D = soil gas species membrane material diffusion coefficient, cm²/s.

For carbon tetrachloride, a sampler with a membrane conductance of 2.5×10^{-4} ml/min-torr operating with a 20 mL/min. transport gas flow rate at one atmosphere pressure will attain dynamic equilibrium in 34 minutes. The carbon tetrachloride concentration or pressure measured in the transport stream will be 0.0095 that in the soil sampled.

The time required to reach static equilibrium after the sampler is implanted in the soil is:

$$T_{se} = 5V/KP_a$$

where

 T_{se} = time required for the sampler to attain static equilibrium, hr;

V = sampler chamber volume, cm³,

- K = soil gas species diffusion conductance of sampler chamber membrane walls, mL/min.-torr; and,
- P_a = ambient total pressure, torr.

At static equilibrium, the soil gas pressure in the sampler chamber is equal to the soil gas pressure itself. Thus, the transport gas soil gas pressure or concentration measured at the surface is that of the soil gas pressure at the subsurface level. For carbon tetrachloride, a sampler with the membrane conductance cited above and a 12 cm³ chamber volume will attain equilibrium in approximately 5.3 hours. The sampler operates in an identical fashion when immersed in liquids to measure the partial pressure of dissolved liquids or gases or the vapor pressure of liquids 9,10,11.

As mentioned above, PDPMOM has the capability of operation in two modes, static or dynamic. Operation in the static mode occurs when the diffusion chamber between the two planar membranes is not purged with transport gas. This would be the normal mode of operation when the device is left implanted within a contaminated area. Soil gas diffusion into the membrane diffusion cell will continue until equilibrium is reached and the partial pressures of soil gas contaminants within the cell are equal to their partial pressures in the soil strata. This would apply also when PDPMOM is implanted below the water table. Concentrations inside the membrane cell are directly proportional to VOC concentrations in the water. Periodically the implanted device membrane cell would be purged with transport gas bringing VOC to an analytical instrument at the surface for quantitation. The second mode of operation, the dynamic mode, occurs during continuous purging of the membrane cell. A steady state or dynamic equilibrium condition is maintained in this case with a constant VOC mass transport through the membranes into the purged cell. A more detailed and rigorous treatment of these principles is described in the following literature ^{9,10,11}.

Calibration

The PDPMOM requires a calibration independent from any analytical instrument or monitoring device located at the surface. A calibration for an individual PDPMOM would involve:

- 1. Laboratory calibration with a standard gas
- 2. Laboratory or field calibration with targeted hazardous soil gas species of interest
- 3. Periodic in situ calibrations, after field implantation, with the standard gas

(2)

(3)

This procedure identifies drifts in implant response or conductance (K) and provides a test of implant seal and membrane integrity. The response, K, of each soil gas species is unique. One might expect that new soil gas species will occasionally be encountered during site characterizations, for which no previous calibration has been performed. Therefore, if monitoring data quality requirements can tolerate slight dimensional variations between individual implant devices, then it is only necessary to determine the permeability coefficient, P_m , of the soil gas species in question. This coefficient may be determined by measurement of the implant response for the species in question relative to a known soil gas species or the standard gas mentioned above. More stringent monitoring and analysis protocols would require that each individual implant be calibrated for each specific soil gas species prior to implantation. This would not be practical.

An alternative approach would be to use a calibration procedure that relates the conductance, K, to a standard gas of each implanted PDPMOM unit to the standard gas conductance, K_r, of a reference PDPMOM unit maintained in a laboratory. Thus, the response ratio, K/K_r, using a standard gas is determined for each PDPMOM prior to field deployment. Then, the relative response of a deployed PDPMOM for any soil gas species, K_{sg}, is determined from a measurement of the reference PDPMOM response, K_{ne}, for the same soil gas species:

$$K_{sg} = (K/K_{T})K_{ng}$$

where

- K_{sg} = the relative response of a deployed PDPMOM for any soil gas species,
- K = standard gas diffusion conductance of PDPMOM chamber membrane walls, ml/min-torr,
- K_r = standard gas conductance of a reference PDPMOM unit maintained in a laboratory, ml/min-torr; and,

 K_{ng} = the relative response of a reference PDPMOM to any soil gas species measured with an implanted device.

Equation (4) applies whether implant soil gas measurements are made in static or dynamic modes. Static and dynamic mode responses, K_{sg} , to trichloroethane (TCA), an industrial degreasing solvent contaminating the soil near an aircraft maintenance facility, are illustrated in Figure 6. This equation would be utilized to calculate the solvent vapor concentration in the soil, if this was a soil gas species for which the implant had not been previously calibrated. Once the unknown species is identified, then a standard response curve, K_{ng} , of the component is generated with a reference PDPMOM so the true soil gas concentration of the previously unknown contaminant may be determined.

With this procedure, rapid field measurements may be taken without the cost and time required to do extensive implant calibrations in the field for every contaminant encountered with every implant. Soil gas contaminant mapping efforts at a large site may require many implants. This also eliminates the complication of introducing the species one is measuring into the environment. Periodic in situ field and lab reference calibrations with standard gas would be necessary to identify drift in implant and reference unit response and update changes in response ratio, K/K_r. In situ calibration, with dual calibration lines, provides a means of transferring surface located calibration gas down hole to inject into the region outside of the membrane cell. Additionally, these lines may be used for soil gas sampling directly, however; this could lead to quantitative errors due to soil gas depletion in the region near the implant.

ION TRAP MASS SPECTROMETER

Soil gas measurements in the field were made with a DSITMS equipped with a helium and air blending device. The mixing ratio of these gases was adjusted for optimum ion trap performance. Air transport gas flow rate through PDPMOM was also optimized with this arrangement, and was typically controlled to approximately 20 mL/min. The capability of pulling ambient air transport gas through the

(4)

sampling module facilitated making field soil gas measurements.

SYSTEM RESPONSE CHARACTERISTICS

The system response characteristics to a soil gas sample from an implanted sampler that has attained static equilibrium is illustrated in Figure 2. After time zero, where time zero is the time the transport gas begins to flow, there is a lag time during which the analyzer shows only baseline response. Subsequently, a rise in the response to a peak is observed followed by a decrease to a steady level above baseline and approximately 0.0095 that of the peak level.

Static Response:

- For a 20 mL/min. transport gas flow rate and a < 1 mL analyzer detector chamber volume:
- δt_1 lag time of transport gas and sample soil gas in the pneumatic return line or time for sample to be lifted from the module to the surface or analyzer, ≈ 0.8 min., for 50 feet of 0.125 inch O.D. standard wall return line.
- δt_2 time required (rise time) to fill analyzer detector chamber with transport gas and sample soil gas, ≈ 0.05 min.
- δt_3 time required to fill and empty the analyzer detector chamber of transport gas and sample soil gas, ≈ 0.25 min.
- R_d analyzer response to dynamic sample; and,
- R_s analyzer response to static sample.
- $R_s/R_d \approx 105$, for carbon tetrachloride

The peak response is the analyzer response to the static sample and the steady low level response is the analyzer response to the dynamic sample. It is noted that only one static response can be obtained every 6 hours providing the transport gas stream is zero during this period. However, a dynamic sample response can be obtained indefinitely.

Field Trials

Field deployment of PDPMOM at Dover Air Force Base resulted in several successful implantations and soil gas analyses. The implants were only deployed to a depth of 5 to 7 feet because the water table was very shallow at this site, approximately 10 feet. The intent was to perform initial field tests of PDPMOM with soil gas only. Therefore, implantation depth was limited to the soil vadose zone in an attempt to keep the sampling device above tidal fluctuations of the water table. Soil gas analyses were performed using ion trap mass spectrometers with modified ion trap air sampling interfaces with the capability of optimizing implant sampling flow rate. Static and dynamic mode responses, K_{sep} to trichloroethane (TCA), an industrial degreasing solvent contaminating the soil near an aircraft maintenance facility at the air base, are illustrated in Figure 6. During these soil gas measurements, it became apparent that TCA rather than trichloroethylene (TCE) was the major soil contaminant. As a result, several field measurements were made before standards of authentic TCA could be obtained. Initial measurements were made relative to TCE, a minor contaminant at this site. Later in the laboratory, relative response ratios of these two compounds were calculated in an attempt to estimate soil gas concentrations obtained during the repeated field implantations. Not every implantation resulted with soil gas analyses. Often no soil gases were detected indicating very localized contamination at this site. At the end of the first week of field trials, PDPMOM was left implanted at a depth of 5 feet.

Field soil gas measurements resumed three weeks later. Two soil gas measurements consisting of a calibration followed by static and dynamic measurements were obtained during each week of tests for a total of four complete runs, shown in Table 2. An ion chromatogram resulting from a quantitative soil gas measurement taken during the final week of tests is shown in Figure 6. A permeability coefficient, P_m , for TCA was calculated to be 8.99 x 10⁻¹⁰ ml/min-cm²-torr/cm, based on this data (final entry in Table 2). This value, obtained from data taken during the final field test, generally agrees with other VOC

permeability coefficients previously observed in the laboratory with the 2.0 μ m polyimide membrane, Table 1. It is worthy to note that the variation in the observed and calculated soil gas concentrations given in Table 2, for TCA may be due to factors unrelated to the actual PDPMOM operation. Most important is the fact that only the final soil gas measurement was quantitative for TCA. In this case only, TCA was in the standard mix used for calibration. As a result, the previous three runs were estimates of TCA concentrations at this site. When PDPMOM technology becomes available for more extensive site characterizations and contaminant mapping efforts, this variability should be accounted for by utilizing the calibration procedure described above. This would allow more quantitative determinations of analytes not previously encountered in the field.

CONCLUSION

The sampling module was shown to be a rugged fieldable device. The integrity of the membrane seals were not compromised during field tests. Best rate of response was achieved with ultra thin polyimide membranes. Teflon® membranes proved to be more durable based on laboratory tests only. However, these membranes would likely be more durable during long term field implantations provided that ultra fast rate of response was not a critical analytical requirement. PDPMOM performed quite well in the field. No plugging of the membranes, calibration gas ports, or transport gas ports was noted during the field implantations. After approximately three weeks in the ground, no visible effects were noted with the sampling module even after a significant amount of surface water due to a ruptured water line deluged the area. The endcap adapter plate, mounting and sealing PDPMOM within the hardened housing, gave an effective surface to subsurface seal. Two implanted sampling modules performed exceptionally well in these initial field trials. Interface of PDPMOM to the US Army Site Characterization and Penetrometer System was successful with no major problems noted.

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Table 1. Laboratory test results with selected VOC diffusing through a 2 μ m polyimide membrane coated on a porous inert substrate.

Volatile Organic Compounds (VOC)	VOC Transport Gas ¹ Conc. (ppbv)	VOC Standard ² Conc. (ppmv)	³ θ _c (min.)	⁴ D (cm ² /s)	⁵ P _m (mL/mincm ² -torr/cm)
Trichloroethylene	165	20	50	1.30x10 ⁻¹¹	9.70x10 ⁻¹⁰
Carbon Tetrachloride	123	17	8	8.00x10 ⁻¹¹	8.46x10 ⁻¹⁰
Benzene	349	34	29	2.30x10 ⁻¹¹	1.22x10 ⁻⁹
Toluene	298	29	71	9.40x10 ⁻¹²	1.22x10 ⁻⁹
Perchloroethylene	143	16	33	2.00x10 ⁻¹¹	1.01x10 ⁻⁹
Chloroform	225	22	25	2.67x10 ⁻¹¹	1.15x10 ⁻⁹
meta-Xylene	346	25	96	6.96x10 ⁻¹²	1.57x10 ⁻⁹

¹ Laboratory simulated soil gas measurements at dynamic equilibrium.

² Simulated soil gas concentrations.

³ Time constants, time required to reach 0.63 maximum dynamic mode signal level.

⁴ Diffusion coefficients, determined empirically.

⁵ Permeability coefficients, determined empirically.

Table 2.	Subsurface contaminants;	PDPMOM field soil gas measurements at a site on Dover Air
	Force Base, May 1995. ¹	

Field Sampling Tests	TCA (ppmv)		TCE (ppmv)		
	Static Equilibrium Conc.	Dynamic Equilibrium Conc. ²	Static Equilibrium Conc.	Dynamic Equilibrium Conc. ³	
First Week	2009	2152	10.9	ND ⁴	
First Week	2163	3674	11.2	ND	
Final Week	1441	1497	9.9	ND	
Final Week	1719	1594	7.8	ND	

¹ All values obtained from a cone penetrometer push designated as, hole #4, sampling module was left implanted for 3 weeks.
² Calculated value obtained by multiplying dynamic equilibrium response by the ratio of maximum static equilibrium response divided by dynamic equilibrium response, ratio determined empirically for each analyte.
³ No calculated value reported because dynamic equilibrium TCE response ≅ baseline noise.
⁴ Dynamic equilibrium TCE response ≅ baseline noise.

Deployed PDPMOM



Figure 1. Implanted sampling module.







Figure 3. Cross sectional view of PDPMOM membrane diffusion chamber.



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Figure 4. Exploded view of sampling module components.

Passive Dual Planar Membrane Organics Monitor (PDPMOM)



Figure 5. Assembled sampling module.



Figure 6. An ion chromatogram illustrating typical static and dynamic mode soil gas measurements obtained at Dover Air Force Base.

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