

Novel technologies for sniffing soil and ground water contaminants

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The presence of hydrocarbons, dense non-aqueous phase liquids, heavy metals and radioactive wastes in soil and ground water represents a serious threat to health and safety. Detecting and delineating these contaminants in the subsurface is a challenging task. Traditional site characterization methods involving drilling and sampling (soil, groundwater, and soil gas), followed by laboratory analysis are time-consuming, laborious, expensive and expose personnel to contaminated media. Recent emphasis has been placed on developing on-site field characterization techniques based on direct push technologies (DPTs) such as the cone penetration test (CPT). A variety of samplers have been deployed in DPTs for collecting soil gas, groundwater and soil samples from desired depths of interest. A variety of *in situ* sensors have been deployed inside cone penetrometer probes, including load cells, pressure transducers, miniature camera, electrical resistivity, temperature, pH and oxidation reduction potential, laser induced fluorescence, infrared spectroscopy and Raman spectroscopy. This article also describes a novel electronic nose technology that was integrated with an *in situ* vapour sampling membrane interface probe for sniffing subsurface volatile organic contaminants. This technology has been recently miniaturized and integrated with CPT sensors for comprehensive geotechnical and geoenvironmental investigations.

Keywords: Contaminants, electronic nose, groundwater, site characterization, soil, sensors.

Introduction

GEOENVIRONMENTAL site characterization aims to determine subsurface soil stratigraphy and hydrogeologic conditions, and detect and delineate the extent and magnitude of chemical contamination. Traditional methods for detecting soil and groundwater contaminants involve drilling and sampling (soil, groundwater and soil gas), followed by laboratory analysis. Because these methods are time-consuming, laborious, expensive and expose personnel to contaminated media, recent emphasis has been placed on developing on-site field characterization techniques.

In recent years, many specialized sensors have been integrated with direct push technologies such as the cone penetration test (CPT) for detecting soil and groundwater contaminants¹. These sensors fall into one of two categories: either non-sampling sensors or sampling sensors. A non-sampling sensor generally performs qualitative measurements on the sensor surface, while a sampling sensor draws a water or vapour specimen either into the cone body or through transmission lines to the ground surface for quantitative chemical analysis. Some of the sensors deployed in direct push technologies (DPTs) are described here.

Groundwater sampling by DPTs

A variety of techniques are available for sampling groundwater. Most of these techniques involve installing a filter from which samples can be taken. The groundwater sampled by these techniques is transferred to air tight containers or are purged *in situ* and collected on sorbent cartridges. The samples may be analysed on-site by purge-and-trap gas chromatography (GC) or they may be sent to the laboratory for analysis. The samples collected on the sorbent cartridges are thermally desorbed and analysed by GC. Some of the groundwater sampling devices may also carry sensors to measure temperature, pH, redox potential, electrical conductivity, dissolved oxygen and other gases.

Bailer

The HydroCone groundwater sampler has a PVC screen with a sacrificial cone tip. This allows samples to be taken at multiple depth intervals within the same sounding location. The groundwater sampler operates by advancing hollow push rods with the filter tip in a closed configuration to the desired sampling depth. Once at the desired sample depth, the push rods are retracted; exposing the encased filter screen and allowing groundwater to enter. A small diameter bailer is lowered through the push rods into the screen section for sample collection. After collecting the sample, the push rods and sampler, with the exception of the PVC screen and the sacrificial cone tip are retrieved to the ground surface, decontaminated and prepared for the next sampling event.

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Hydropunch

The Hydropunch groundwater sampler a self-contained probe made of stainless steel and Teflon has a retrievable stainless steel or disposable plastic intake filter with a sacrificial cone tip. The Teflon sample chamber (500 ml) is connected to the intake filter and two ball check valves at either end of the chamber prevent the sample from leaking. The groundwater sampler operate by advancing hollow push rods with the filter tip in a closed configuration to the desired sampling depth. The push rods are retracted, once the desired sampling depth is reached. This exposes the inlet screen and allows groundwater to infiltrate hydrostatically from the formation through the inlet screen into the sample chamber. As the HydroPunch is retrieved, the hydrostatic pressure differential between the inside of the sample chamber and the surrounding aquifer causes the ball check valves to close and leakage from the sample chamber is thereby prevented during transit of the sample to the surface.

BAT enviroprobe

The BAT enviroprobe sampling system consists of a wellpoint that is internally sealed with a septum². After pushing the wellpoint to the desired depth, an evacuated 35 or 70 ml vial, also sealed with a septum is wired down the casing. A double-ended hypodermic needle, mounted in an adapter below the vial pierces both the wellpoint and the sample vial septa and allows fluids to flow into the vial. As the sample vial is retrieved to the surface, the septa seal, maintaining the sample at *in situ* pressure conditions. The BAT EnviroProbe is considered a more accurate way to obtain samples that contain volatile organic compounds (VOCs) because virtually no exposure of the sample to air occurs during the sampling process.

ConeSipper

The ConeSipper developed by Westinghouse Savannah River Corporation and Applied Research Associates, Inc. is a module that can be attached directly behind a standard cone penetrometer to obtain gas or water samples as the CPT probe is advanced³. This sampling device contains a two stage stainless steel filtration system, an integrated pneumatic valving system, and an 80 ml stainless steel sample chamber. The sample is drawn into the chamber through the filter and a check valve prevents backflow from the chamber. Once the chamber is full, gas pressure is applied through one of the two pneumatic control tubes and the fluid is pushed up to the surface through the second tube. A small pneumatic control box on the surface meters the flow of inert gas to the module, controls the rate of sample collection and allows the ConeSipper to be purged and decontaminated down-hole.

Envirocone and the chemicone

The envirocone samples both groundwater and soil vapour⁴. At the surface, groundwater samples retrieved by the envirocone are analysed for pH, redox potential, dissolved oxygen, electrical conductivity and temperature; vapour samples retrieved by the envirocone are analysed for dissolved oxygen, total VOCs, combustible gas, hydrogen sulfide (H₂S), and carbon dioxide (CO₂). The chemicone is a probe that is instrumented with temperature, pH, and oxidation–reduction potential (ORP) sensors. The chemicone is useful for acquiring data for bio-characterization of contaminated sites⁵.

Soil gas sampling by DPTs

Active soil-gas sampling has evolved and become more cost effective through the application of DPT. Soil gas sampling by DPT is commonly used to take samples of VOCs from the vadose zone⁶. Soil gas can be collected from specific depths in several ways. One method involves driving commercially available hollow soil gas sampling probes to the desired depth using a power hammer (e.g., Geoprobe Direct Push Machine). When they are at the desired depth, the drive point is retracted or detached by pulling back the tooling, exposing an inlet section with holes or a screen. Samples can be drawn directly from the probe rods, after purging (evacuating) a sufficient volume of air from the probe rods. Another method involves collecting a sample through Teflon tubing inserted through the probe and attached by an adaptor to the bottom probe rod section.

The gas samples may be collected in Tedlar bags, Summa canisters or on to sorbent cartridges. When a Summa canister is used, sampling is achieved by soil gas equilibration with the evacuated Summa canister (air sampling pump is not required). On-site analysis may be performed using field analytical equipments or mobile laboratories equipped with photoionization detectors (PID) and flame ionization detectors (FID), portable gas chromatographs or mass spectrometers. Mobile laboratories provide quantitative results with rapid turnaround time and do not require storing, packaging and shipping of samples.

Cone penetrometer multiport sampler

A multiport sampler attached to a cone penetrometer for obtaining multiple soil vapour and/or liquid samples during a single penetration was designed and developed by the Waterways Experiment Station (WES), Site Characterization and Analysis Penetrometer System (SCAPS) Program⁷. The multiport sampler has a series of 12 vertically stacked sampling modules that can be independently opened for sampling at desired depths. At a given sam-

pling depth, a known volume of gas is sampled through the port in the side of the push rod. Analytes are transferred to the surface through separate transfer lines for either collection onto sorbent cartridges or analysed by field analytical instruments. Because of the efficient sealing and control of the inlet ports, cross-contamination between samples is minimized. This technology is reliable, rapid and economical since multiple soil vapour and/or liquid samples are collected during a single penetration.

The membrane interface probe

The membrane interface probe (MIP) is an *in situ* tool (Figure 1) that thermally extracts VOCs from the subsurface and transports the vapour through 30.5 m of Teflon tubing (trunk-line) to various detectors on the surface⁸. The probe is 38 mm in diameter and 320 mm in length with a tapered end and a conical tip. It has a replaceable semi-permeable thin film membrane (20 mm in diameter) impregnated into a stainless steel screen mounted along the face. The membrane is heated to 120°C by a heater block. Heating the soil around the probe not only releases the VOCs, but also accelerates the diffusion of vapour across the membrane. Diffusion occurs because of a concentration gradient between the soil-gas and the clean carrier gas behind the membrane. The carrier gas (ultra zero air) sweeps the vapour through the trunk-line and delivers it to GC detectors. The probe is also equipped with a soil conductivity device to measure the electrical conductivity of the soil. The MIP is robust and can be advanced at a steady rate, or driven into the subsurface, using Geoprobe percussion soil probing equipment. A

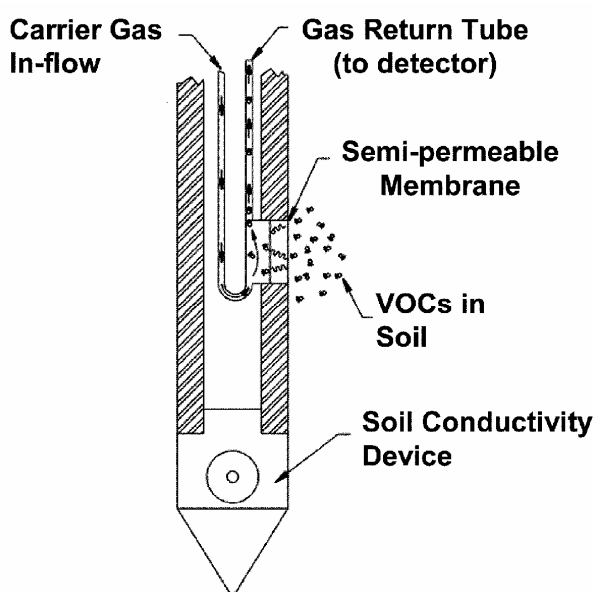


Figure 1. Membrane interface probe.

depth encoder is used for measuring the rate of advancement and the depth of the probe. An MIP controller box, equipped with a pressure regulator and a mass flow controller, regulates the flow rate of the carrier gas which is normally set to 40 ml/minute. The controller box has a temperature measurement and control system that regulates the power supply to the heater block. The probe temperature is displayed on an LCD panel. The controller box is also equipped with a data acquisition system to acquire data from the soil conductivity device, a depth encoder, and GC detectors.

Non-sampling sensors integrated with the cone penetrometer

Cone penetration test

CPT is essentially an *in situ* technique for geotechnical and hydrogeological site characterization. The test consists of pushing an instrumented cylindrical probe, known as the cone penetrometer, into the soil at a rate of 2 cm/s. The penetration depth is measured by an electronic depth encoder. The cylindrical probe has a conical tip with apex angle equal to 60°. The device is equipped with a load cell at the tip to measure the cone or tip resistance (q_t), which is the force offered by the soil to the tip during intrusion divided by the projected cone area. The projected cone area of the standard cone penetrometer is 10 cm². The cone penetrometer is also equipped with a friction sleeve (150 cm² surface area) and a load cell to measure the sleeve friction (f_s), which is the local friction between the surrounding soil and the shaft of the probe. The term friction ratio (R_f), often used in CPT data interpretation, is the ratio of the sleeve friction to the cone resistance, expressed as a percentage. A piezocone penetrometer is also instrumented with a pressure transducer for monitoring pore water pressure (u) in soils. The data acquired during a CPT may be plotted on a computer screen in real time (as q_t , f_s and u vs depth), and used for hydrogeological profiling and for determining various engineering soil properties¹.

Resistivity cone penetrometer

The resistivity cone penetrometer is equipped with two or more electrodes to measure the bulk electrical resistivity (ρ) of soils. Soil resistivity is influenced by the conductivity of the pore fluid, soil minerals and the porosity. Fine-grained soils usually have lower resistivities compared to coarse-grained soils. Sudden changes in electrical resistivity may also indicate potentially contaminated zones¹⁹. By measuring changes in resistivity, the resistivity cone penetrometer has been successfully used in delineating dense non-aqueous phase liquids (DNAPLs), acidic spills and salt water intrusions.

Vision cone penetrometer

A vision cone penetrometer (VisCPT), instrumented with a miniature CCD video camera, light source, and optical systems was developed for visual subsurface inspection as the probe is advanced into the soil¹⁰. The soil is imaged in real time through a sapphire window on the side of the probe and shows soil texture, colour, grain size and DNAPLs, eliminating the need for expensive and time-consuming soil sampling (Figure 2). A continuous stream of high resolution images are acquired in real time. The images are recorded and digitized, and computer vision techniques and image-texture analysis are used for determining soil grain size¹⁰. VisCPT allows the operator to visually detect or confirm the presence of DNAPLs such as creosote, coal tar and gasoline.

Laser-induced fluorescence-cone penetrometer

The laser-induced fluorescence (LIF) cone penetrometer was developed by the U.S. Triservices and deployed in the site characterization and analysis penetrometer system (SCAPS) for *in situ* detection of petroleum hydrocarbons¹¹. In the LIF-CPT, a nitrogen laser source (>260 nm wavelength) emits pulsed ultraviolet light that is transmitted along a fibre optic cable and dispersed into the soil through a sapphire window mounted on the cylindrical surface at the end of a cone penetrometer (Figure 3). Certain contaminants such as polycyclic aromatic, aromatic and substituted hydrocarbons, along with a few heterocyclic hydrocarbons if present, will fluoresce under the laser light. This induced fluorescence is transmitted to a

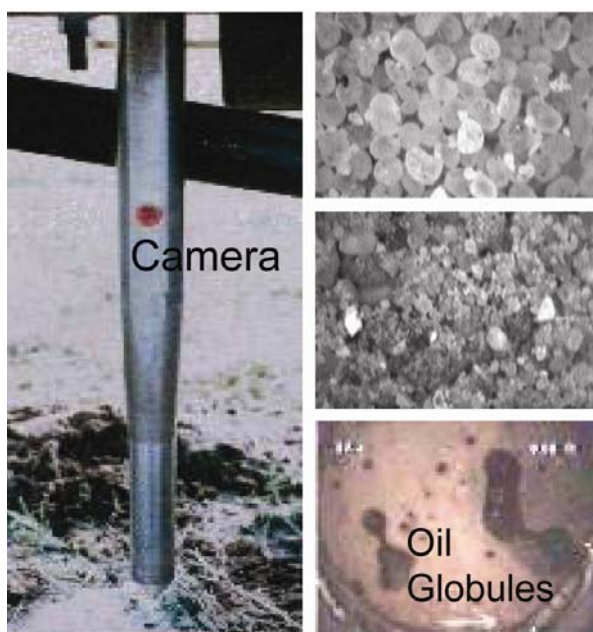


Figure 2. The VisCPT.

fluorescence detector along a second fibre optic cable. A detector (i.e., photodiode array or charged coupled device) and signal processor (optical multichannel analyser) are used as the fluorescence detector and the data is processed by a computer system. The fluorescence spectra indicates the type of contaminant present. The intensity of fluorescence can be correlated with the concentration of the contaminants. The LIF system is not suitable for contaminants that do not fluoresce, such as chlorinated hydrocarbons. A standard method of practice exists for delineating subsurface petroleum hydrocarbons and other hydrocarbons using a fibre optic based nitrogen LIF sensor system¹².

Fibre optic infrared cone penetrometer

The LIF technology described earlier cannot detect chlorinated hydrocarbons such as trichloroethylene (TCE) since they do not fluoresce. A fibre optic infrared reflectance sensor was developed by the US Navy and deployed in a cone penetrometer to detect marine diesel fuel and contaminants such as chlorinated hydrocarbons¹³. The measurement system consists of a Fourier transform infrared (FTIR) spectrometer, a reflectance probe and two optical fibres. The fibres are connected to the spectrometer, FTIR detector and probe through couplers containing gold-plated, off-axis parabolic mirrors, one for bringing collimated light to a focus at the sample surface and the other, for collimating light reflected from the exposed sample area. The system is integrated with a cone penetrometer using field-ruggedized, sulphide-based chalcogenide optical fibres to transmit IR reflectance data to a remotely located FTIR spectrometer.

Raman spectroscopy probe

Raman spectroscopy identifies inorganic and organic chemicals by means of their vibrational spectra. A chemically and mechanically robust optical Raman spectroscopy

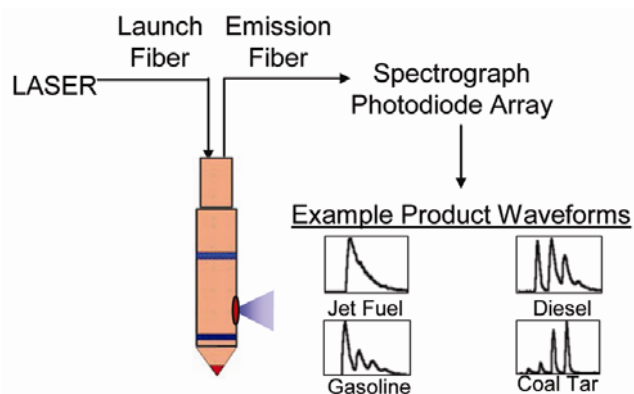


Figure 3. Laser induced fluorescence - CPT.

probe assembly for deployment in a cone penetrometer was developed at the Lawrence Livermore National Laboratory¹⁴. The assembly consisted of an optical Raman probe and a penetrometer compatible optical probe housing. The probe is optically linked via fibre optics to the light source and the detection system at the surface. A sapphire window hermetically sealed directly into the side-wall of the probe housing transmits the interrogation light beam and the resultant signal. Raman spectroscopy probe has been successfully used to identify subsurface DNAPL constituents by their unique spectral fingerprints at the Savannah River site in Aiken, South Carolina¹⁵.

Other sensors for integration with the CPT

For detecting radioactive contaminants several passive gamma-ray detectors incorporating sodium iodide or cesium iodide gamma-ray scintillators along with a photomultiplier tube or photodiode for scintillator light detection have been developed¹⁶. Other sensors that have been developed for integration with DPT include an optoelectronic sensor to quantitatively measure benzene, toluene, ethylbenzene and xylene (BTEX)¹⁷, an electro-odo-cell for the detection of organic vapours¹⁸. Sensors that are being researched for integration with the CPT include X-ray fluorescence (XRF) spectroscopy¹⁹ and laser induced breakdown spectroscopy for inorganic elements, particularly heavy metals²⁰.

An electronic nose-membrane interface probe for sniffing subsurface contaminants

The deployment of sensors with DPTs for geoenvironmental site characterization has been steadily growing in the past decade. A novel electronic nose (EN) technology was recently developed for rapidly sniffing subsurface contaminants²¹.

Mimicking the mammalian olfactory system

The EN is an automated odour recognition device that detects and identifies chemical vapours based on the principles of human olfaction^{22,23}. A typical human nose has 10 million olfactory receptors and more than 1000 specialized sensory nerve cells, or neurons, each with hair like fibres called cilia at one end. The odour molecules diffuse through the cilia into the olfactory receptors/neurons. Each neuron sends a signal along a nerve fibre called an axon to the olfactory bulb, a brain structure located just above the nose. Signals from neurons with the same receptors converge on structures called glomeruli in the olfactory bulb. The pattern of activity in these glomeruli creates signatures that the brain learns to recognize as different odours.

A portable EN for sniffing soil vapour

Analogous to the human olfactory system, the EN consists of an array of chemical sensors mounted inside an environmentally controlled chamber. The portable EN consists of an array of seven metal oxide sensors, a temperature sensor, and a humidity sensor. The metal oxide sensors are composed of tin dioxide and are doped with different impurities (palladium, platinum, etc.) in order to give them slightly different electrical characteristics. Hence, each sensor in the array gives a different electrical response (change in electrical resistance and DC output voltage) for a particular target vapour introduced into the sensing chamber. The combined output from the sensor array forms a fingerprint, or signature, that is unique for a particular odour. Different chemicals may be presented to the sensor array to build a database of signatures. This database of labelled signatures may then be used to train an artificial neural network (ANN) based pattern recognition system. ANNs are computational paradigms that work similarly to the brain in processing information. Once the network is trained for chemical recognition, the detection mode simply consists of propagating the sensor data through the trained network to yield automated real-time identification of contaminants and their concentration levels. In this study a general regression neural network (GRNN) developed by Specht (1991) was used for training and pattern recognition²¹.

Field demonstration of the EN-MIP system

The EN is integrated with an *in situ* vapour sampling membrane interface probe (MIP) for deployment in the field. The EN-MIP system was successfully tested near three monitoring wells at a gasoline contaminated highway garage site belonging to the Rhode Island Department of Transportation located in Riverside, Rhode Island, USA²⁵⁻²⁷. The field equipment and onsite analytical support included the EPA mobile laboratory for providing onsite confirmatory analysis, and the Geoprobe Direct Push Machine for advancing the MIP to the desired sampling depths (Figure 4). The probe was halted for 15 min at each depth to collect the vapour samples. The carrier gas led the vapour samples into the EN that was housed inside the EPA mobile laboratory. The collective peak response from the seven metal oxide sensors was recorded as the chemical signature (fingerprint) of the vapour sampled from a particular depth. The MIP was then advanced to the next sampling depth. *In situ* data acquired by the EN-MIP system near the MW5 test location are shown in Figure 4. The graph shows the response of the seven sensors with time for the EN-MIP tests conducted at the different depths.

Onsite analysis of vapour samples performed by the EPA in their mobile laboratory provided confirmation in

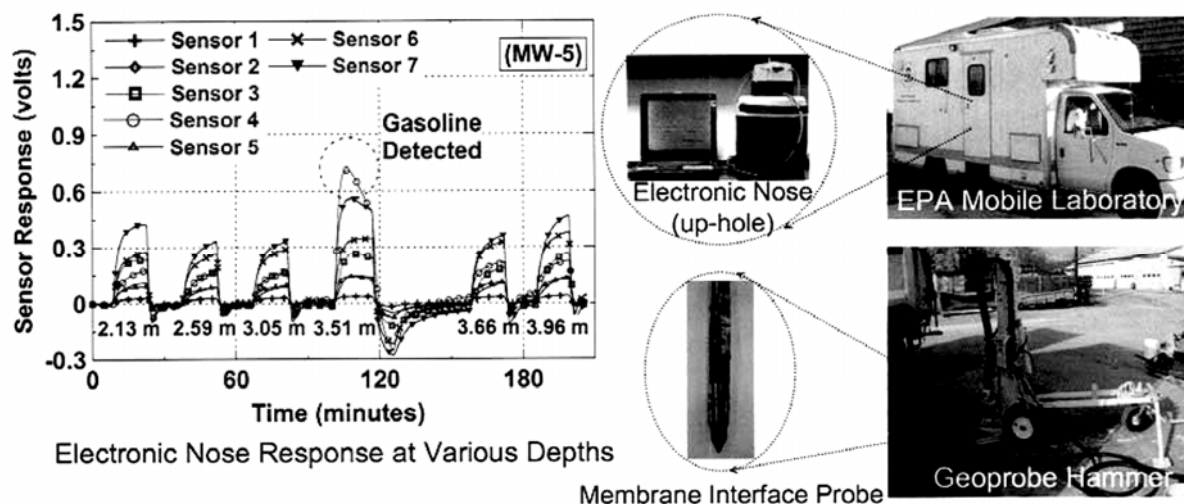


Figure 4. EN-MIP tests at a gasoline contaminated site.

Table 1. EN-MIP field demonstration results

MW No.	Depth (m)	GC/MS analysis		EN-MIP		GC/MS analysis
		BTEX in aqueous soil (mg/kg)	BTEX in aqueous soil (mg/kg)	Expected conc. level	GRNN predicted conc. level	BTEX in water (mg/l)
MW5	2.13	*	4.78	Nil	Nil	
	2.59	*	1.89	Nil	Nil	
	3.05	*	5.94	Nil	Nil	8.22
	3.51	5.98	106.45	Medium	Medium	
MW6	3.96	*	29.83	Low	Low	
	1.22	25.30	227.08	High**	N/A	
	1.52	0.88	41.79	Medium	Medium	
	1.83	0.12	32.33	Medium	Medium	20.9
	2.44	1.46	3.03	Nil	Nil	
	3.05	1.81	3.59	Nil	Nil	
MW7	3.66	*	5.12	Nil	Nil	
	2.44	0.22	3.26	Nil	Nil	
	3.05	1.48	1.00	Nil	Nil	
	3.66	*	1.01	Nil	Nil	4.01
	4.27	*	0.66	Nil	Nil	

*Indicates below reporting levels (<0.025 mg/kg) for BTEX.

**Erroneous EN-MIP test data due to operator switching delay.

the field. A Scentograph Plus II portable GC instrumented with a Micro Argon Ionization Detector (MAID) and a Photo Ionization Detector (PID) was integrated with the MIP to obtain preliminary data for training the EN-MIP system. Predictions by the EN-MIP system were correlated to the GC-MIP test results and qualitatively confirmed by conventional GC/MS laboratory analysis performed on soil samples collected using a 38 mm diameter open-tube sampler and groundwater samples collected from the monitoring wells using a bailer. A GRNN model was trained to predict the gasoline concentration levels from the signatures obtained during the EN-MIP field tests. The trained GRNN model correctly predicted the concentration levels for all five signatures used for

testing and all nine signatures used for training (Table 1). The results from the laboratory and field testing have shown that the EN-MIP system has the potential to be a reliable tool for rapid screening of gasoline contaminated sites. Additional field studies are needed to verify the validity of the technology in a variety of geological regimes.

A multisensor electronic nose cone penetrometer

The sensing chamber of the electronic nose used in the EN-MIP system has a large internal volume (175 cm³) and is located above ground (up-hole). The vapour sample flows through 30.5 m of Teflon tube before reaching

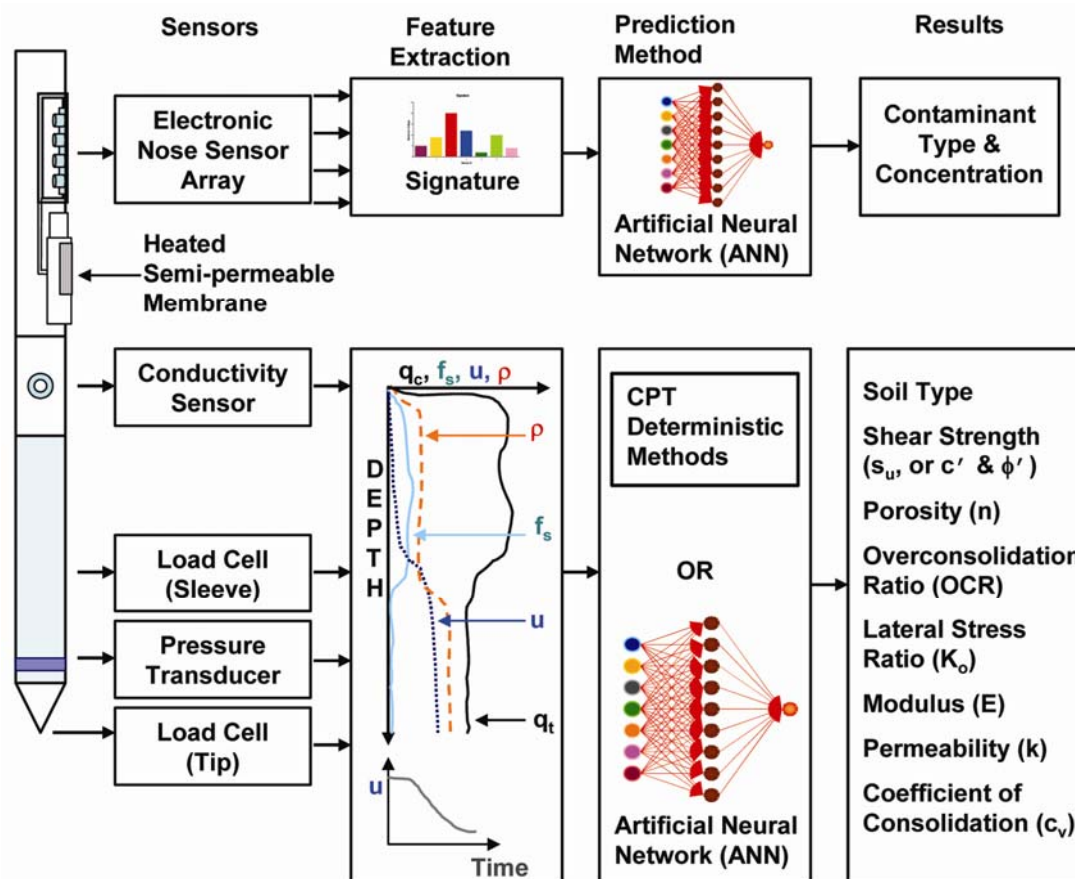


Figure 5. Multisensor electronic nose cone penetrometer.

the sensing chamber. The vapour sample gets diluted and partially absorbed by the Teflon tube and even further diluted as it is delivered to the large sensing chamber.

As part of on-going research, a new miniature EN has been developed for 'downhole' implementation inside a cone penetrometer. The small sensing chamber (internal volume less than 10 cm^3) is located immediately behind the heated membrane assembly (Figure 5). The miniaturization and placement of the EN immediately behind the vapour sampling module resulted in improvement of speed and sensitivity. The multisensor EN cone penetrometer is equipped with a miniature EN and other CPT sensors: load cells to measure tip resistance and sleeve friction, pressure transducer to measure pore water pressure, and a conductivity sensor to measure soil conductivity (Figure 5). The electronic nose cone penetration test (ENCPT) will provide *in situ* data for comprehensive geotechnical and geo-environmental site characterization.

Summary and conclusions

For *in situ* characterization of contaminated sites, several sampling and sensing techniques have been integrated with direct push technology such as the CPT. Some of the devices that have been integrated with the cone penetrom-

eter include a miniature camera for visual detection and identification and various sensors for measuring stresses, pore water pressure, electrical conductivity, temperature, pH, oxidation-reduction potential, radioactivity and hydrocarbon contamination in soils. Compared to the invasive process of borehole drilling, sampling and testing, DPTs are less invasive causing minimum site disturbance and also minimizes the risk of potential cross contamination resulting from drilling operations. The method does not generate soil cuttings thereby significantly reducing exposure of workers to potentially hazardous material and also results in significant hazardous waste disposal cost savings. Substantial indirect cost savings are also realized due to the rapid turnaround time.

Recently an *in situ* EN-MIP was developed for rapidly sniffing and identifying subsurface contaminants. This innovative EN-MIP system was successfully tested at a gasoline-contaminated site. In addition to all the advantages mentioned above for DPT, the EN-MIP technology allows rapid delineation of subsurface contaminants and acquisition of continuous, real-time data in the field. Data is processed in real-time and the results are available for making on-site decisions. All these advantages make the EN-MIP a viable economic alternative or an addition to conventional methods of site investigation. One of the

limitations of the existing EN-MIP technology is that it cannot separate compounds from a complex mixture (as a GC column). Statistical techniques and regression models (using partial least squares), however, may be used in the future with the EN-MIP for predicting the concentrations of individual compounds in simple gas mixtures. It is also possible that background odour may impact test results. Hence, the technology requires a certain amount of site specific training, depending on the complexity and variability of the background odour, as well as a more comprehensive laboratory calibration in a variety of soils having different grain size distributions. Once trained to recognize and account for the influence of soil type and background odour, the technology can be used for rapid characterization of a site. As part of ongoing research, the EN has been miniaturized and integrated with CPT sensors. It is envisioned that this multisensor EN cone penetrometer will result in more efficient and comprehensive site investigations.

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