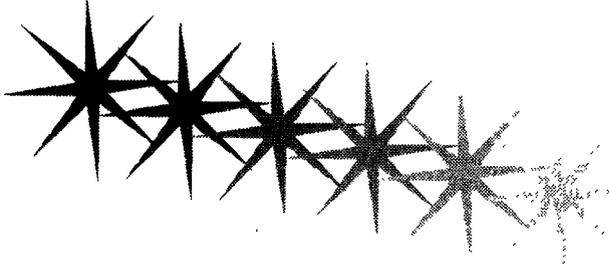


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AIR & WASTE MANAGEMENT
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Fourth International Symposium on Field Screening Methods for Hazardous Wastes and Toxic Chemicals

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FIBER OPTIC INFRARED REFLECTANCE PROBE FOR DETECTION OF AROMATIC CONSTITUENTS OF FUEL IN SOIL

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Contamination of soil with petroleum fuels is a significant environmental problem in both the public and private sector. Over the course of many years, fuel spillage during everyday refueling operations have caused significant contamination of the subsurface soils with aliphatic and aromatic hydrocarbons. Additionally, fire spills due to leaks in storage tanks or transfer lines have also contributed significantly to soil contamination. Fuels such as JP-5 are mixtures of alkanes, saturated ring compounds, olefins, and aromatic compounds (single and polycyclic). Of particular interest are the aromatic constituents of the fuel such as benzenes, naphthalenes, tetralins, etc. Some fuels, such as JP-5, can be composed of as much as 25 wt% of these aromatic additives. In order to remediate sites contaminated with fuels cost effectively, monitoring techniques which can identify and quantify the amount of contaminant in the soil are needed. The goal of the current work is to develop a fiber optic IR reflectance sensor for use in the cone penetrometer to detect both the hydrocarbon fuel and the aromatic additives in the soil. By using chalcogenide or fluoride optical fibers, the spectral window from 3 μm to 6 μm (the sapphire window cutoff) is available for detection and quantitation of fuel contamination in the soil. Results for aromatics and hydrocarbon fuels in various soil matrices will be discussed with respect to detection limit, sample preparation, and best spectral range to use for achieving the lowest detection limit.

Acknowledgment: The authors wish to thank the United States Army Environmental Center for supporting this program.

IN SITU MEASUREMENT OF VOLATILE ORGANIC COMPOUNDS IN GROUNDWATER BY METHODS COUPLED TO THE CONE PENETROMETER

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The sampling of subsurface materials via cone penetrometry provides a minimally intrusive, efficient, and cost-effective method for obtaining samples for the analysis of contaminants in groundwater, soil, and soil gas. On-site analysis of samples collected by this methodology has been demonstrated to facilitate the characterization of sites by making real-time, interactive sampling decisions possible. Devices that isolate the contaminants from groundwater and soil within the cone penetrometer (CPT) and are directly linked to analytical instrumentation at the surface would further increase the speed and accuracy of on-site analysis. The overall goal of our investigation is to develop methods and technology that will couple a CPT with field-deployable instrumentation, including gas chromatography/mass spectrometry, to directly transfer volatile organic compounds (VOCs) and semi-volatile organic compounds from subsurface material at depth to the analytical instrument in the field to provide near-real time analysis. Our presentation will focus on methods and technology to isolate VOCs from groundwater with analysis by multidetector gas chromatography (GC).

The approach involves the development of a methodology that incorporates a sparging vessel to purge VOCs from a confined volume of groundwater in the CPT, transports the analytes to a preconcentration device at the surface through a heated transfer line, and provides quantitative analysis by multidetector GC. The critical aspect of this approach is the quantitative transfer of VOCs from the groundwater to the preconcentration device at the surface. Tubings composed of stainless steel, nickel, and two types of Teflon[®] were evaluated as transfer line materials. The materials were tested by using a dynamic dilution apparatus in which a certified gas standard containing chloroform, methylchloroform, carbon tetrachloride, tetrachloroethylene, benzene, and toluene is mixed with air containing various concentrations of water. The concentration of the target analytes generated by the apparatus is determined via the analysis of samples collected on graphitized carbon black directly from the dilution manifold. The transfer line is then attached to the manifold, and several discrete volumes of gas are drawn through the tubing and collected on graphitized carbon black. Clean air is then drawn through the tubing to determine the extent of sorption. The sensitivity of the transfer of VOCs through the tubing to (1) the water content of the gas stream and (2) the temperature of the transfer line was determined. Sparging vessels of Teflon[®] and stainless steel were fabricated to fit within a standard purge-and-trap concentrator. Recoveries of analytes from spiked water samples were determined and compared to those obtained with a standard glass sparging vessel. Results from laboratory tests of a prototype of the ground water sampler that fits within a CPT will also be presented. This work was supported by the U.S. Department of Energy under Contract No. W-31-109-Eng-38 as part of the Characterization, Monitoring, and Sensor Technology Integrated Program of the Office of Technology Development.

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A CASE STUDY COMPARING THE USE OF LASER INDUCED FLUORESCENCE WITH CONE PENETROMETER TESTING TO MORE CONVENTIONAL SCREENING METHODS

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Site assessments utilizing in-situ techniques to characterize subsurface stratigraphy and contaminant distribution are becoming more accepted and commonly used. Laser Induced Fluorescence (LIF) spectroscopy with Cone Penetrometer Testing (CPT) is a new technology that provides data on stratigraphy and petroleum hydrocarbons (PHCs) in the subsurface environment. This paper presents a comparison of LIF combined with CPT to more conventional characterization methods at a manufacturing site in Nebraska.

Over the last two years, LIF technological advances have led to LIF equipment with improved durability, reduced bulk and weight, and integrated systems with CPT equipment. The technology has recently been piloted in a variety of field and soil conditions. These advances, along with the need for rapid in-situ information on the horizontal and vertical distribution of PHCs, have resulted in equipment that is now available for commercial applications.

The LIF equipment consists of a tunable laser located in a CPT truck which is connected by a fiber optic cable to a small sapphire window located on the cone penetrometer probe. Light generated from the laser is directed into the side of the borehole and onto the soil and groundwater. The light produced by the laser is at a wavelength which allows the PHC molecules to absorb photons, causing PHC materials to fluoresce. The fluorescence is returned through the sapphire window and conveyed by fiber optic cable to a detection system within the truck. The incoming data is processed and data depicting fluorescence versus depth is continuously measured and recorded as the CPT push proceeds.

The Nebraska technology demonstration site was formerly used to manufacture farm equipment. Various PHCs were stored in underground storage tanks across the site. Two PHC spill areas have been identified: one primarily consisting of xylenes, and the other a mixture of diesel fuel oil and kerosene. Free phase and dissolved product exist at both locations. The near surface geology consists of up to 35 feet of unconsolidated silt/sand and gravel overlying sandstone. The water table is found between 5 and 10 feet below the ground surface.

In each spill area, a grid area with about 20 equally spaced probe locations 50 feet apart was established. Within a 3-foot radius of each grid point, the following test program was designed: soil gas samples for on-site PHC analyses; LIF/CPT data to 20 feet; and hollow stem auger drilling with continuous split spoon sampling, organic vapor screening, and soil sample collection for off-site PHC analyses every 5 feet to a completion depth of 20 feet.

These data are qualitatively and quantitatively compared. Data collected suggest that each method provides qualitatively useful site screening information. However, significant variations in time, data interpretation, and methodology are of interest. This paper presents these findings, and also compares test time and cost, along with the advantages and disadvantages of each method.

DEVELOPMENT OF AN IN SITU VOLATILE ORGANIC CHEMICAL ANALYSIS SYSTEM

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Conventional subsurface contaminant mapping technology involving borings and laboratory analysis of soil samples is time consuming and expensive and often results in inadequate descriptions of contaminant plumes. Application of advanced chemical techniques to cone penetrometer technology provides a means for real time detection and mapping of contamination in the subsurface. Development of an in-situ volatile organic sampler for the cone penetrometer and an analytical system to detect ppb levels of BTEX compounds and TCE has been developed at the USACE Waterways Experiment Station. The sampler is pushed to a desired depth in the subsurface, an interior rod is retracted, and a measured volume of soil is sampled and purged in place. Volatilized compounds are transferred to the surface where they are trapped on tenax and desorbed on a field portable gas chromatograph. The soil sample is then expelled, and the cone penetrometer pushed to a new depth where the process is repeated. Experimental results for different soils and contaminants are discussed.

DEVELOPMENT OF MULTIANALYTE SENSOR ARRAYS FOR CONTINUOUS MONITORING OF POLLUTANTS

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Industrial development has led to the release of numerous hazardous materials into the environment, posing a potential threat to surrounding waters. Environmental analysis of sites contaminated by several chemicals calls for continuous monitoring of multiple analytes. Monitoring can be achieved by using imaging bundles (300-400µm in diameter), containing several thousand individual optical fibers for the fabrication of sensors. Multiple sensor sites are created at the distal end of the fiber by immobilizing different analyte specific fluorescent dyes. By coupling these imaging fibers to a charge coupled device (CCD), one has the ability to spatially and spectrally discriminate the multiple sensing sites simultaneously and hence monitor analyte concentrations.

Prior to immobilization of the dye the distal end of the fiber is functionalized to permit covalent attachment of the polymer matrix. Discrete regions of the fiber bundle are successively illuminated through the proximal end so as to photo-polymerize the polymer matrix containing the fluorescent dyes.

Current studies focus on the development of a multi-analyte sensor for monitoring Al^{+3} , pH, hydrocarbons and uranyl ion. For the monitoring of Al^{+3} , a variety of indicators^{1,2} are being evaluated for their applicability to sensor design. Lumogallion immobilized in poly HEMA shows considerable sensitivity and dynamic range. The fluorescent indicator eosin has been identified as the indicator for monitoring pH in the range 2.0 - 4.5. The indicator can be immobilized in an analogous fashion to fluorescein³ (pH 4.5 - 8.0). Hydrocarbon sensors have been fabricated from different photo-polymers that show response to several hydrocarbons using Nile Red as the indicator.

The current instrumentation used for the measurement of fluorescence intensities is built around an Olympus microscope, using a Hg-Xe arc lamp for excitation and a CCD camera for spatial detection of the fluorescent intensities. Computer controlled filter wheels provide the necessary wavelength selection. However, a field portable instrument capable of radiometric measurements is being developed. The development of the multianalyte sensors and field trials will be presented.

The concept of multianalyte sensors is certainly not limited to the analytes mentioned above. As additional analyte specific reagents and other transduction mechanisms are identified, these sensors will find greater scope of applicability. Due to its miniature design and ease of fabrication such sensors should be particularly valuable for continuous in-situ monitoring of pollutants.

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DETERMINATION OF SOLID/VAPOR PHASE INTERACTIONS ON QUARTZ CRYSTAL MICROBALANCES USING MOLECULAR MODELING

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A major technology barrier in the development of many chemical sensors is the lack of recognition coatings to achieve selectivity and sensitivity. The major objective of the present work is to investigate the utility of molecular modeling in predicting and interpreting recognition coating/analyte interactions. Quartz Crystal Microbalances (QCMs) coated with polyethylenimine (PEI) were studied with carboxylic acid vapors. Linear Solvation Energy Relationships (LSER) derived from gas/liquid chromatography (GLC) bonded-phase solute-solvent interactions have proven useful in studying the effects of coating/analyte interactions on QCM devices. These relationships incorporate dispersion, polarity-polarizability, and hydrogen bonding forces to model the interactions; they have been used successfully to predict coating/analyte binding affinities. In this study a homologous series of four carboxylic acids vapors (acetic, propanoic, butanoic, pentanoic) were selected to interact with PEI coatings. LSER values for each acid indicate that each additional methylene constituent increases the dispersion force interactions between the coating and analyte with a resultant increase in binding affinity. QCM devices (10 MHz) were dip-coated in a solutions of PEI and methanol and allowed to dry at room temperature to provide five thin film coatings measuring 0.69 kHz, 0.72 kHz, 1.7 kHz, 4.1 kHz, and 4.4 kHz. The acid vapors were introduced separately over the QCM devices, and the piezoelectric wave frequency changes due to absorption of the acid analytes on the PEI coating were recorded. After correcting for vapor pressure and molecular weight, the acids were ranked according to their interaction (corrected frequency change) with the coating. Linear relationships between coating thickness and acid interaction were observed for each acid ($r^2=0.990-0.996$). Since the LSER values of polarity, polarizability, and hydrogen bonding are essentially the same for each acid, it is believed that the slope of each line is related to the amount of dispersion interaction occurring which, as mentioned above, relates directly to binding affinity.

Comparison of experimental results with LSER values show similar agreement with regard to analyte/coating binding affinities. Semi-empirical (AMI) calculated dipole moments coincide trendwise with LSER values for polarity. Slopes of the plots relating frequency change versus coating thickness, indicative of dispersive interactions, coincide trendwise with semi-empirically calculated Van der Waal force values. Data analysis suggests for dispersive interactions, that a linear relationship ($r^2=.960$) exists between experimental slope values and calculated molecular modeling values. These results are promising and suggest that computer modeling may prove useful in predicting and interpreting sensor coating/analyte interactions.

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CHEMILUMINESCENT CHEMICAL SENSORS FOR INORGANIC AND ORGANIC VAPORS

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Chemiluminescence techniques have been successfully applied to the detection of numerous chemical analytes, including metals, polycyclic aromatic hydrocarbons, and various oxidizing and reducing agents. The fundamental attractions of chemiluminescence are its high sensitivity, wide dynamic range, and simplicity of instrumentation. Typically, chemiluminescent reagents are incorporated into flow injection analysis schemes which enable the detection of photons arising from chemical reactions with oxidants or reductants. Because of the continuous generation of waste that arises from these systems, and because of the increasing demand for simple, remote chemical sensors, we have begun investigating the possibilities inherent with incorporating chemiluminescent reagents within hydrogels and polymeric thin films for the detection of inorganic and organic toxic chemicals. By careful consideration of the chemiluminescence reagent, metal catalyst, polymeric support film and pH of these thin films, some selectivity can be tailored into the chemical sensors. Instrumentally, the detection cell consists simply of a small cavity supporting the polymeric thin film, behind a glass window. A photomultiplier tube is positioned directly in front of the window, while a small, hand-held vacuum pump is used to sample the air directly across the surface of the film. Current efforts are being made to extend this technology to fiber optics for remote sensing.

2,4,6-Trichlorophenylloxalate (TCPO), in the presence of a strong fluorescer such as perylene, is a highly sensitive and selective chemiluminescent monitor for H_2O_2 levels in solution. Unlike other chemiluminescent reagent schemes, the nature of the mechanism lends itself to this high degree of selectivity. We have found that the incorporation of TCPO and perylene within a polymeric thin film, such as poly(ethylacrylate), also permits the detection of low ppm levels of NO_2 in air. The use of 3-aminophthalhydrazide (luminol), while being much less selective, is significantly more sensitive to certain toxic chemicals. The incorporation of luminol and Fe(III) within a poly(vinylalcohol) hydrogel gives a film with superior sensitivity toward NO_2 (detection limit of 0.46 ppb and a response time on the order of seconds). The hydrogel serves as an effective means for eliminating humidity problems associated with other polymeric films. By substantially increasing the concentration of KOH contained within the film, the sensor becomes responsive to trace levels of O_2 . A detection limit of < 2 ppm oxygen in nitrogen (signal/noise = 3:1) was obtained with response times of less than a second. Other toxic gases which we have formulated chemiluminescent thin films for include SO_2 and hydrazine, N_2H_4 . Recently, we have investigated the use of a heated Pt filament as a pre-oxidative step prior to passage of the gas stream across the film surface. This has permitted the sensitive detection of CCl_4 , $CHCl_3$ and CH_2Cl_2 in the low ppm regime. The advantage is that the filament can be easily turned on or off, depending upon the sensing needs of the user. Discussions will center about a complete analytical characterization of the capabilities and shortfalls of these chemiluminescent chemical sensors.

FIBER OPTIC SPECTROELECTROCHEMICAL SENSING OF TRICHLOROETHYLENE

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The necessity of techniques for in situ/real-time sensing and monitoring of chlorinated hydrocarbon solvents is becoming increasingly apparent because of the ubiquitous presence of these contaminants in groundwater. Traditional laboratory sampling methods have several disadvantages such as long turnaround time, expense, manpower, and doubts about sample integrity, particularly for volatile compounds. In addition, traditional techniques are also not capable of providing early warning of the presence of these contaminants. The combination of optical fiber technology, originally developed in the telecommunication industry, with spectroscopy, however, has led to the continuing development of new sensing techniques which address the drawbacks of traditional methods.

Our approach to monitoring and sensing of chlorinated hydrocarbon contaminants, particularly trichloroethylene (TCE), incorporates spectroscopy and electrochemistry with fiber-optic sensing techniques. Spectroelectrochemical fiber optic sensing or SEFOS is, in principle, a controllable chemical sensing technique which can be adapted to fiber-optic sampling applications such as groundwater monitoring in wells or site analysis/assessment via cone penetrometers. With SEFOS, an electrochemical method is used to reduce TCE into a reactive intermediate. The TCE reactive intermediate then reacts with the relevant functional groups of a "trapping" reagent resulting in changes in the spectroscopic behavior of the reagent. The spectral changes can be used to sense TCE at levels approaching regulatory limits.

This paper will describe recent developments of the SEFOS technique for the detection of TCE and will discuss the advantages of SEFOS over present methods of monitoring chlorinated hydrocarbons. The optimum conditions needed for SEFOS as well as relevant analytical data will be presented.

UNATTENDED SCREENING FOR VINYL CHLORIDE AND CARBON TETRACHLORIDE IN AIR FROM PART PER TRILLION TO UPPER PART PER MILLION LEVEL USING A COMBINATION OF ARGON IONIZATION AND ELECTRON CAPTURE DETECTORS

Dr. A. Linenberg, President, Neil J. Lander, Product Specialist, Sontex Systems Inc., 553 Broad Avenue, Ridgefield, NJ 07657

The need to screen the atmosphere for background levels of Vinyl Chloride and Carbon Tetrachloride led to the development of a self-contained portable gas chromatograph for just such a purpose. Furthermore, the instrument was designed to operate in an automated, unattended fashion for a considerable duration.

The Argon Ionization Detector (AID) will detect compounds whose ionization potential is 11.7 eV or less. The Electron Capture Detector (ECD) is primarily used to detect extremely low levels of compounds which have a high affinity for electrons, such as halogenated compounds. Thus Vinyl Chloride (ionization Potential = 10.00 eV) and Carbon Tetrachloride (ionization Potential = 11.28 eV) can be detected with the AID. The combination of these detectors provides for a full range of detection from the sub part per trillion level to the high part per million level. Sub PPB to low PPB levels of Vinyl Chloride (0.1 to 1.0 PPB, for example) were detected using the AID in combination with a trap built specifically for this purpose. Sub PPT to low PPT levels of Carbon Tetrachloride were detected using the ECD in combination with the trap. Low PPT to high PPT levels were detected using the ECD in combination with an automated sampling loop PPB levels and up were detected using the AID in combination with the trap. Finally, PPM levels and up were detected using the AID in combination with a sampling loop.

The gas chromatograph was periodically recalibrated with certified standards of Vinyl Chloride and Carbon Tetrachloride. The entire operation of the gas chromatograph could be monitored via modem. The portability and flexibility of such a system promises extremely low level detectability of such compounds on site and on an unattended, automated basis.

A NEW PORTABLE MICRO GAS CHROMATOGRAPH FOR ENVIRONMENTAL ANALYSIS

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A truly portable gas chromatograph for field-screening of both volatile and semivolatile organic environmental compounds has been developed and tested at LSU - Institute for Environmental Studies. Based on a novel capillary column heating technique and a heated injection port, the new Micro-GC is capable of analyzing samples with retention indices of up to at least 2000 in a period of about three minutes. In addition to achieving isothermal capillary column temperature control, the new Micro-GC is also capable of performing ultra fast temperature-programming rates (up to 10° C/s) using less than 50 watts of maximum electrical power. Thus, it is possible to operate this instrument in the field using portable battery power. Its use of narrow bore capillary columns (100 µm i.d.) implies that it uses minimum amounts of carrier gas. Thus, the combination of isothermal and temperature-programming control in our portable Micro-GC at less than 50 watts maximum power implies the ability to perform field separations of organic compounds with widely varying boiling temperatures.

DESIGN CONSIDERATIONS FOR A NOVEL MINIATURIZED TANDEM GC METHOD FOR FIELD SCREENING APPLICATIONS

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A rapid, automated gas and vapor sample injection technique for capillary columns reported by Arnold et al. [1] enables operation of two GC columns in tandem (GC/GC) in such a way that a two-dimensional gas chromatogram can be obtained. This requires rapid repetitive injections of the eluent from the first column onto the second column. Typically the first column is operating in an isothermal or temperature programmed mode with a total run time of 100-1000 seconds and average peak widths in the 10-25 second range whereas the second column operates at the same temperature as the first but with repetitive injections at 2-5 second intervals and average peak widths in the 50-250 millisecond range (also depending on the type of detector used).

The power of this type of two-dimensional GC analysis has been convincingly demonstrated by Liu and Phillips [2] using a rapid thermal desorption device in combination with cryotrapping of eluent from the first column. A disadvantage of their method, however, is the high power usage level associated with the cooling and desorption cycles. Also, the associated contraction and expansion of the carrier gas flow may complicate the chromatographic conditions. Finally, only a limited range of compounds can be analyzed due to the specific vapor pressure requirements for successful operation of trap-and-desorb schemes. Highly volatile species run the risk of being incompletely trapped whereas low volatile, and particularly polar, species may become irreversibly adsorbed.

None of these problems is inherent in the approach chosen by us. However, compared to the Phillips method, our technique is potentially less sensitive due to the lack of a "cryoconcentration" step. Between the sampling intervals of the second column the effluent of the first column is vented. On the other hand, proper design of the GC separation conditions, permits the analyte concentrations within the column effluent to remain relatively close to those in the original sample. As long as a concentration sensitive detector is used with sufficiently low dead volume and signal response time to match the operating requirements of the second column, the loss of effluent due to the low duty cycle of the latter does not have a major effect on overall sensitivity. Design considerations as well as preliminary performance data will be presented. The choice of the detector, a particularly critical area in ultrahigh speed GC, will be discussed in detail.

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DETERMINATION OF BTX AND BUTADIENE IN AIR BY HAND-HELD GAS CHROMATOGRAPHY

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The need for rapid, cost-effective and actionable field analytical data has fueled the development of portable instrumentation. In the last few years, we have successfully utilized "transportable or lug-gable" field gas chromatographs for remediation and worker health and safety applications. Recently, a hand-held portable gas chromatograph has become commercially-available. Our general approach for new field analytical technology is to validate instrument performance under laboratory conditions using simulated field samples prior to field testing the instrument under "real-world" conditions.

This presentation will discuss laboratory validation and field implementation of the Photovac Snapshot hand-held gas chromatograph for determination of benzene, toluene and xylenes (BTX) and butadiene in air. Interaction between the vendor, research and development and field operators was essential in successful implementation of this technology. Reliable portable GC data has been obtained from field applications such as soil vapor, fugative emissions and general area monitoring. In the initial field tests, laboratory GC analyses were utilized to validate field GC results.

The quality of the field data is highly dependent on proper training of field operators. The knowledge of instrument operation is important but generation of reliable field data requires the implementation of field QC protocols. Minimum field QC protocol consists of calibration with a certified gas blend, calibration checks and blank analyses. This strategy has been used by instrument operators with a variety of skill levels to obtain reliable field GC data.

HANDHELD GC INSTRUMENTATION FOR CHEMICAL WEAPONS CONVENTION TREATY VERIFICATION INSPECTIONS

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Advanced field screening technology for toxic chemicals will play a crucial role in treaty verification inspections for the new Chemical Weapons Convention (CWC). Because CWC treaty inspections are projected to take place on short notice and in many different locales worldwide, highly portable and self-contained chemical instrumentation packages can be especially valuable. Furthermore, the schedule of chemicals covered by the CWC encompasses a wide range of volatilities and chemical properties making the requirements for chemical instrumentation especially challenging. In response to these needs, we have developed an advanced handheld instrument based on an innovative, ultra-low thermal mass miniaturized gas chromatograph (GC) for rapid detection of treaty prohibited chemical weapons materials and precursor chemicals. This GC consists of an integrated capillary column, heater and temperature sensor which allows computer controlled temperature ramp rates as high as 10 °C/sec. Besides the GC, the instrument incorporates a miniature trap preconcentrator, miniature dual flame photometric detectors (FPD) for phosphorus and sulfur compounds, and advanced signal processing. The instrument is self contained, can detect a wide range of both volatile and semi-volatile compounds, and is designed for handheld operation during treaty inspections.

OPEN-PATH ATMOSPHERIC MONITORING WITH A LOW-RESOLUTION FT-IR SPECTROMETER

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Contemporary approaches to open-path atmospheric monitoring by Fourier transform infrared (FT-IR) spectrometry involve measurements that are usually made at relatively high resolution with expensive instrumentation. These instruments can be heavy and difficult to move. Their performance is also limited by excessive absorption of water vapor across much of the mid-infrared spectrum. In this paper, we will demonstrate the feasibility of constructing a less expensive system that is designed to operate at significantly lower resolution than contemporary instrumentation, thereby reducing the cost and weight. Measurement at low resolution also enables measurements of some components absorbing at wavelengths that are usually masked by absorption due to water vapor to be observed at short pathlengths.

In the system to be described, radiation from a conventional infrared source is passed through a small cube-corner interferometer to a nonstandard retroreflector. The return beam is collected at unusually high efficiency and focused onto the detector for measurement of the interferogram. The efficiency of this optical arrangement is sufficiently high that liquid nitrogen cooled mercury cadmium telluride (MCT) detectors are no longer needed and either an ambient temperature deuterated triglycine sulfate (DTGS) pyroelectric bolometer or a Peltier-cooled Zn-doped MCT detector can be used.

The data processing requirements for interferograms measured using this system include the use of optimized apodization functions to maintain Beer's Law linearity for major atmospheric components (especially water vapor). Multivariate algorithms for multicomponent quantitative analysis have been compared, with partial least squares regression proving to have the best performance for vapor-phase species.

After a description of the instrument that has been constructed in our laboratory, the relative performances of the DTGS and Zn-doped MCT detectors will be compared. In addition, the performance of the prototype version of this instrument and a conventional open-path FT-IR spectrometer will be described.

FTIR OPEN-PATH ABSORPTION MEASUREMENTS OF HAZARDOUS COMPOUNDS ON AN INDUSTRIAL SITE AND A WASTE SITE IN GERMANY

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FTIR open-path absorption measurements found different applications as a screening method for hazardous compounds. Important advantages are:

- Several compounds can be measured.
- On-line measurements are possible in principle.
- Remote measurements can often be made where the access is otherwise not possible.
- No poisoning of the sensor occurs.

This method has been applied by our laboratory to measurements at an industrial site and a waste site. Recent results will be presented. Some quality assurance issues will be addressed.

AIR MONITORING WITH A COHERENT INFRARED DIFFERENTIAL ABSORPTION LIDAR

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A coherent infrared differential absorption lidar was developed that is capable of remote detection of several atmospheric molecular pollutants. Typical detectable components are ammonia, phosgene acetone, toluene, hydrogen cyanide, benzene etc.

Enhanced operation of the lidar system was demonstrated by using an "autodyne" detection scheme with appropriate selection of the laser sources. This allowed keeping mass (250 kg) and power consumption (1.5 kW) of the system low while sensitivities in the column content range of $10^{-1} \cdot 10^{-3}$ (mg/m^2)/km for ranges up to 3 km could be achieved. Results of field tests and system performance will be given.

DETECTION OF EMISSION SOURCES USING PASSIVE-REMOTE FOURIER TRANSFORM INFRARED SPECTROSCOPY

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The detection of environmentally hazardous chemicals is possible with open-path and fixed-point methods, such as open-path Fourier transform infrared (FTIR) spectroscopy and canister collection followed by gas chromatography/mass spectroscopy (GC/MS). While these methods detect chemical plumes, they cannot trace the plumes to their source. Tracing a plume to its source is important to determine responsibility for the release of fugitive emissions and to repair leaks. Source detection is also important for determining environmental compliance. Light, mobile instrumentation with real-time data analysis is essential for tracking a plume to its source.

Passive-remote FTIR systems are light and transportable. Data analysis can be performed in near real time. The basic system consists of an interferometer, focusing optics, and a detector. Data are transmitted directly to a laptop computer. All the systems discussed can be hand carried to facilitate detection of the plume source. The instrumentation uses ambient energy as its infrared source.

This instrumentation was used to detect a low pathlength release of parts per million-meter (ppm-m) concentration. A 1 ppm-m release corresponds to a 1 ppm chemical release that has a width of one meter. This release would be identical to a 10 ppm release with a width of 0.1 m. These releases were performed using a specially designed vaporizer with an orifice of 10 cm on a side. The FTIR spectrometers detected plumes of this size from distances of roughly 50 m. After a plume was detected, hand-held FTIR spectrometers were used to track the plume to its source. Plumes can be detected from open windows and other low leak-rate sources. Larger passive-remote units mounted on a tripod or a moving vehicle can also be used to initially detect a plume originating from a far-away source. Then, smaller units can be employed to detect the actual source.

TECHNIQUES FOR PREPARATION OF FIELD STANDARDS FOR VOA ANALYSIS OF WATER, SOILS AND AIR

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With the widespread use of field methods to assess contamination of the environment arises a need to prepare accurate standards for field analysis that are suited to the study at hand. The use of typical laboratory standards (1000µg/cc MeOH solutions) is very unsatisfactory because of the complex mixtures typically used in these standards. Where a site investigation has shown the need to measure accurately one or two chlorinated solvents or a few aromatic solvents (e.g. BTEX) it is counterproductive to send into the field a complex mixture containing ten to thirty solvents. This paper will discuss three methods for preparing standards for VOA analysis of water, soil and air samples in the field.

The first method is simply to prepare methanol concentrates from neat standards using a very simple technique that requires no analytical balance or other complicated laboratory equipment. Accurate standards can be prepared in minutes using a graduated cylinder or pipet and a ten-microliter syringe. Standards can be prepared singly for later mixing or as simple mixtures in methanol which match the expected complexity of the field contamination.

The second method is to prepare liquid-liquid equilibrium solutions of individual solvents of interest which are then held for future use. These standards are constantly at solubility equilibrium and it is only necessary to know the solubility of each solvent at room temperature. Dilutions can be made rapidly from the saturated aqueous phase of these solutions for subsequent headspace analysis of water or soil-water mixtures. It is also possible to prepare volatile-in-air standards from these saturated aqueous solutions with a high degree of accuracy in just minutes in the field.

A third procedure is to simply make up a dilute solution of any solvent in water, taking care to keep the mixture under the solubility limit. While these standards require special care in preparation, and have a much shorter useful life than the first two methods, they are readily prepared and can often serve a special need (e.g. an unusual solvent not often encountered in the field, and for which no readily available standards exist).

Preparation techniques will be discussed and some of the pitfalls which could prevent accurate results will be examined.

SOIL MOISTURE VARIABILITY AS AN INDICATOR OF SOIL VOC VARIABILITY

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The main objective of this study was to determine the effect of the soil sample size on the precision of volatile organic compound (VOC) measurements. EPA's high-level screening procedure (included in SW-846 Method 5030/5035) was used to measure VOCs in 2, 20, and 100-g soil samples. The high-level procedure extracts soil VOCs into methanol and analyzes an aliquot of methanol by purge-and-trap/gas chromatography. To increase the sensitivity of the procedure, the soil:methanol ratio was modified from 1:10 to 1:1 (g:mL) and the extraction time was lengthened from 2 min to several days. Extraction into a liquid permits subsampling variable amounts from a more homogeneous media. Use of an optimum/larger sample size to minimize short-range variability, can limit the number of samples needed to characterize a site and provide better, more representative data.

Short-range variability in soil VOC concentrations are effected by (but not limited to) soil texture and organic carbon content. Likewise, short-range variability in the soil moisture content is effected by soil texture and organic carbon content. Replicates of soil VOC concentrations and soil moisture contents were measured as functions of sample size. The study was performed at VOC-contaminated field sites in New York and Nevada to provide data in widely varying soils and climates. Correlations of the variability in VOC concentrations and soil moisture with sample size were generated. Variability in soil moisture content with sample size can be used to estimate an appropriate sample size for measuring soil VOCs.

The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), prepared this abstract for an oral presentation. It does not necessarily reflect the views of the EPA or ORD.

NEW MODULAR TWO STAGE PRECONCENTRATOR FOR VOCs IN AIR

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Attempts to lower detection limits of VOCs for field deployable instruments has led to the construction of a new two stage solid sorbent concentrator. Previous success of sample preconcentration with an enhanced version of a microchip gas chromatograph has been built upon by the incorporation of a concentrating and focusing trap. With the use of computer actuated valves, heaters, and flow controllers this modular device is capable of interfacing with several presently available microchip GCs as well as benchtop GCs. The combination of specific adsorbents allow a large volume of ambient air to be concentrated on a large sorbent trap and subsequently focused into a narrow desorption band from a micro trap. This process enables present micro gas chromatographs to analyze a wide range of VOCs at sub ppb levels using less than 50 watts (peak) of electrical power. The presentation will evaluate desorption curves as well as concentration factors for a variety of compounds under varying sorption conditions and demonstrate the applicability of this concentrating device for both field and laboratory analysis.

DIRECT TRACE ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN AIR USING A FIELD-DEPLOYABLE ION TRAP MASS SPECTROMETER WITH FILTERED NOISE FIELDS

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There is increasing interest in the development of field portable mass spectrometers to monitor toxic air pollutants in real-time. A direct air sampling ion trap mass spectrometer has been evaluated in the laboratory under controlled conditions. An environmental test chamber was used as the source of the target compound mixtures at known concentrations. Measurements were made of nonpolar and polar volatile organic compounds (VOCs) at trace levels, using the ion trap technology with two direct air sampling interfaces (semipermeable membrane; glow discharge ionization source).

The system is based on Teledyne's research-grade 3DQ Discovery™ three-dimensional quadrupole ion trap. Application of filtered noise field (FNF) technology minimizes the effects due to space charging normally associated with ion traps. It also provides a unique means of simultaneously isolating individual target compounds in complex mixtures. It is, therefore, capable of true selective ion monitoring and, when operated in the MS/MS mode, permits unambiguous characterization of individual compounds with high sensitivity and specificity. The instrument is small and lightweight, and can be easily deployed in the field. We have evaluated the operation of the combination system with a semipermeable tubular membrane and an atmospheric sampling glow discharge ionization source for the measurement of selected VOCs in air.

Preliminary results obtained with the combination direct air sampling/FNF/ion trap system have helped to delineate the advantages and disadvantages of the two direct air sampling interfaces. Experiments were performed to evaluate the specificity, sensitivity, response time, and effects of relative humidity on the real-time detection of nonpolar and polar VOCs. This presentation will describe measures that are being investigated to improve the specificity and sensitivity of the devices.

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MINIATURE ION MOBILITY SPECTROMETER DETECTOR

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Ion mobility spectrometry (IMS) has been a laboratory phenomenon for the last 25 years, while a hand portable unit with proven field capabilities, the Chemical Agent Monitor (CAM), has been in the military inventory for approximately a decade. The inherent simplicity of IMS and its operation at atmospheric pressure and ambient temperature conditions made it an attractive technology to transition from the laboratory to the field. Analyte ionization requires no power since this is effected by a nickel-63 source. In NATO venues, the units are used for real-time (seconds) detection of very small amounts of nerve and blister agents. Nerve agents are phosphoryl-containing compounds, and a perfect marriage was formed between that class of analytes and IMS. This is because IMS is extremely sensitive and relatively specific to phosphoryl functional groups.

Recently, IMS technology has been reduced in size to that approximating a small VCR-sized remote control hand unit. A number of revolutionary changes have occurred with respect to conventional IMS resulting in significantly lower power usage. The unit operates on demand and automatically powers down after a sample interrogation. The system has the capability of taking a background IMS scan of itself prior to sample introduction in both polarity modes, and then a scan of a sample in both polarity modes. Positive and negative ion difference spectra are the resultant information, and this entire sequence occurs in 2-3 seconds. The unit automatically turns off or can be programmed for repetitive sampling at defined intervals. The conventional pump motors which introduce, circulate and remove the sample airstream have been replaced with a molecular sieve passive system that uses no power and ensures a dry IMS system. The sample introduction system motors have been replaced with a small audio speaker that is usually found in commercial wire-trimmed radio headsets. The displacement of the speaker cone, typically a few 100 microliters, dictates the volume of sample ingested. The ionization source operates on either a nickel-63 radiation ring or a nonradioactive carbon fiber corona discharge source. With respect to the highly regulated issue of radiation, the nonradioactive ion source is the more attractive version.

Single and mixtures of illicit drug synthesis and purification solvents and drug precursor molecules were tested with the device for detection and characterization purposes and which comprise a test set of compounds that are of interest to drug and law enforcement agencies. Compounds include methanol, cyclohexanone, diethylether, phenyl-2-propanone, acetic acid, acetone, isopropanol and methylcyclohexane. Mixtures containing equal and different concentrations of the drug solvent and precursor compounds were tested for their detection of presence by headspace vapor analysis with respect to single compound ion mobility spectra.

It appears that the palm-size IMS unit has the potential of being a practical analytical tool for indoor use and it is projected to have the potential for outdoor use.

REMOTE ELECTRODE FOR MONITORING METAL CONTAMINANTS

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The development, characterization and testing of a remote electrode, for downhole monitoring of trace metals will be described. Based on the highly sensitive electrochemical stripping analysis, the novel sensor (connected to a long communication cable) addresses various challenges of remote monitoring. Convenient quantitation of ppb concentrations of copper, mercury or lead, will be reported. The new indwelling probe should be very attractive for the immediate detection of sudden metal contaminations or for providing feedback during cleanup activities, and may thus revolutionize the way by which heavy metals are monitored.

Financial support from the WERC program and Battelle PNL is acknowledged.

DEVELOPMENT AND EVALUATION OF AN OPTIMIZED MULTIPLEX ARRAY DETECTOR RAMAN SYSTEM FOR FIELD ENVIRONMENTAL ANALYSIS

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An optimized, compact Raman system for field environmental analysis is being developed. This system is based on diode laser excitation fiber optic probe technology, a triple grating imaging spectrometer, and a start-of-the-art scientific CCD camera and software. Emphasis has centered on optimizing the Raman apparatus, determining the best fiber optic probe technology that will allow remote sensing (i.e. down a well or simultaneous multipoint monitoring), establishing quality control procedures, and developing camera control and data analysis software. Sensitivity for a variety of compounds will be presented demonstrating major reductions in detection limits. New technologies which may improve detection limit considerably further will be discussed. Problems associated with fiber optic probe technology including the need to eliminate the Raman signal from the silica probe fibers, which can become an interference when the fiber length is more than a few meters, will be considered. Software for camera control and data analysis using LabVIEW for Windows (National Instruments), a graphical Windows-based programming language, will be presented which demonstrates the flexibility and ease of use needed for a field instrument that would be difficult to achieve with current commercial camera controller software in a single program.

A HAND-PORTABLE INSTRUMENT SYSTEM FOR THE REAL-TIME ANALYSIS OF CHLORINATED ORGANIC COMPOUND CONTAMINATION

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Chlorinated solvents (e.g., carbon tetrachloride, trichloroethylene) were used extensively as degreasing agents at government and industrial facilities and represent a major environmental contamination at many of these sites. Because of the toxicity of chlorinated organic compounds, reliable methods for the rapid and sensitive detection are necessary. In 1991, Transducer Research, Inc. (TRI) developed a new chemical sensor which responds selectively to vapors of chlorinated solvents. No sensor response is observed with common hydrocarbon organic compounds such as BTXs (benzene, toluene, xylene) or POLs (petroleum, oil, lubricants), and in fact, no non-halogen containing chemical has been identified which induces a measurable response. While the sensor does exhibit a small response to brominated compounds, and an even smaller response to iodine and fluorine compounds, it responds quickly, reversibly, and with a high sensitivity to vapors of chlorine compounds. Using the organic chemist's shorthand notation "R-Cl" for a chlorine-containing organic molecule, the sensor has been called it the "RCL sensor".

To exploit the analytical capabilities of the RCL sensor, an advanced chlorinated organic vapor monitor was designed and built. The RCL MONITOR was designed to analyze individual samples or monitor an area with automated repetitive analyses. Samples between 0 and 500 ppm can be determined in 90 s with a lower detection limit of 0.2 ppm using the hand-portable instrument. In addition to the development of the RCL MONITOR, advanced sampler systems have been developed to: (1) extend the dynamic range of the instrument through autodilution of the vapor and (2) allow chemical analyses to be performed for condensed phase contamination with a unique closed-loop recirculation sampler. When interfaced to these specialized samplers, the RCL analyzer is capable of measuring chlorinated solvent contamination in the vapor phase up to 5000 ppm and in water and other condensed media from 10 to over 10,000 ppb_w.

The performance of RCL MONITOR was demonstrated in field tests at several DOE facilities. Several important applications have been identified in which measurement and monitoring of chlorinated hydrocarbons is essential. These include:

- Environmental Compliance
- Health and Safety
- Clean-up Process Monitor
- Environmental Modeling
- Site Characterization

Case studies for each identified application are currently underway. Initial results have demonstrated that the performance of the RCL MONITOR compares favorably to CLP methods now in use.

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"FIELD SCREENING" SAMPLING METHODS USED IN PLACE OF "STANDARD" SAMPLING METHODS TO PROVIDE DEFINITIVE DATA FOR THE INVESTIGATION OF A NATIONAL PRIORITY LIST SITE

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The objective of this presentation is to report the effective use of "field screening" sample collection methods in place of "standard" sample collection methods to provide definitive data at a National Priority List (NPL) Site.

The "standard" method of collecting samples in unconsolidated material uses an auger drilling rig; soil samples are collected using split-spoon samplers; and groundwater samples are collected from monitoring wells. This methodology is time consuming, costly, and creates added wastes (e.g., drill cuttings and large volumes of purge waters). Geoprobe™ sampling devices can be used to collect soil and groundwater samples significantly faster, and with a minimum of generated wastes.

Although considered a "field screening" sample collection method, the Geoprobe™ sampling system can be used to collect samples with comparable quality to "standard" sampling methods and can provide defensible data to support site status decisions.

The U.S. Environmental Protection Agency (EPA) placed Dover Air Force Base (AFB), Dover, Delaware, on the NPL because of the presence of contaminated soil and groundwater. Nine major areas of associated constituents and 59 individual sites were identified. A basinwide Remedial Investigation (RI) is under way to define the extent of contamination. To accelerate the investigation, a field screening program was initiated before the RI. This program utilized soil gas and geophysical surveys, Geoprobe™ sampling, and on-site and fixed-base laboratory sample analyses. The program provided a clearer picture of conditions at the Base and allowed a more efficient use of resources during the RI.

Normally "screening" samples are submitted to a field laboratory, which provides analytical results at EPA Level II data quality. Level II data are, by EPA definition, not acceptable for use in Risk Assessments and, therefore, are not acceptable in supporting final decisions on sites. However, during the field screening program at Dover AFB, an innovation on the normal procedure was implemented. Fifty-nine of the 203 groundwater samples and all 16 soil samples collected were submitted to an EPA Contract Laboratory Program (CLP) facility for analyses. The CLP laboratory provided analytical results at EPA Level IV data quality. Level IV data are usable for Human Health Risk Assessments and can support final decisions on sites (e.g., no further action). The decision to use this approach was reached by the Dover project team, which included staff from EPA Region III, to expedite the decision-making process and move forward on addressing the environmental challenges at the Base.

Using "screening" sampling methods instead of "standard" methods provided other benefits to the project, including:

- saving more than \$200,000 in sample collection alone,
- eliminating drilling waste,
- saving a minimum of 250 project/work-hours, and
- reducing the duration of field activities by approximately 20 days.

In summary, "screening" sampling methods are acceptable for collecting samples that can be used to support final decisions on the status of sites and also provide cost and time savings over "standard" sampling methods.

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A COMBINED TECHNOLOGY APPROACH TO MULTILEVEL GROUND-WATER SCREENING

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Several commercially available technologies were combined to rapidly characterize a historical fuel spill in a sand and gravel outwash aquifer. Historical data indicate that possibly 1 to 6 million gallons of aviation gasoline had been discharged at the source area from 1955 to 1970. The Remedial Investigation (RI) at this site indicates fuel-related contamination in the capillary fringe beneath the source area. Rapid horizontal groundwater velocities (~1.7 ft/day) and the 39-year time span between the first discharge and the present could possibly place any resulting groundwater contamination far from the source. Downgradient groundwater wells installed during the RI show no significant contamination, which suggests that the groundwater wells installed during the RI were not screened in the plume, the quantities of fuel released at the site were much lower than estimated, or degradation of the fuels is taking place.

To determine the presence and extent or absence of fuel-related contamination and the possibility of past or ongoing biodegradation associated with this site, several technologies were integrated into the field program. Drive point technology (i.e., Geoprobe) was selected as the intrusive sampling method because it (1) provides rapid sampling rates, (2) reduces investigation-derived waste (it minimizes purge volumes because it uses 1-in.-diam casings and generates no drill cuttings), and (3) minimizes formation disturbance because it uses small-diameter drive rods and rotary motion is lacking. This technology advanced a 1-in.-OD steel drive rod into the aquifer to depths ranging from 72 to 120 ft below ground surface. After advancement to the desired depth, a screen was extended from the 1-in. rods, and an inertial pump was used to sample groundwater. Groundwater sampling began at the bottom of the boring and continued at discrete 10-ft intervals to the water table. Eh, pH, specific conductivity, turbidity, temperature, and dissolved oxygen were measured in a closed cell apparatus and recorded at each interval. A mobile on-site field laboratory analyzed samples from each interval for fuel-related compounds, total petroleum hydrocarbons, and dissolved carbon dioxide and bicarbonate. Accurate locations for each sample point were determined using global positioning system technology. Although existing groundwater maps were used in the initial placement of the Geoprobe borings, locations were adjusted in the field based on the data generated in the field laboratory. After the screening phase, permanent 1-in. stainless steel monitoring wells with prepacked stainless steel screens were installed using drive point technology.

Advantages of combining drive point technology, a mobile laboratory, a global positioning system, and other technologies include (1) minimization of investigation-derived waste, (2) minimization of formation disturbance, (3) rapid generation of multilevel groundwater screening data to facilitate decision making while in the field (27 days in the field at 30 sampling locations analyzing 190 samples plus quality control samples and installing two permanent monitoring wells), and (4) generation of a complete data set by one field team in one mobilization effort. This combined approach to site investigations resulted in a significant savings of time and money for the client.

*Work performed by Analysis Corp. under general order contract 30B-9778C with Martin Marietta Energy Systems, Inc.

**Work performed by Automated Sciences Group, Inc., under general order contract 30B-9778C with Martin Marietta Energy Systems, Inc.

***Work performed under general order contract 77B-99167C with Advanced Sciences, Inc. with Martin Marietta Energy Systems, Inc.

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FIELD ANALYTICAL SUPPORT DURING SUPERFUND SITE REMEDIATION

William L. Goldschmidt, David R. Catherman, and David E. Gallis, Ph.D., ERM, Inc.

The ERM-FAST[®] service provided field analytical support for two projects involving soils remediation at superfund sites in northeastern Pennsylvania and southern New Jersey. These case studies offer illustrations of the effectiveness of field analysis for vastly differing site contaminants and exemplify the power of "real time" field analytical support in reducing time and expense during a project's remedial phase.

The first CERCLA site involved an active manufacturing facility in northeastern Pennsylvania where remediation of the site soils centered around 12 volatile organic compounds (VOCs) which included aromatic and chlorinated alkene hydrocarbons. The EPA required "clean soil" levels to be less than 100 parts per billion (ppb) of "total leachable" volatiles. It was determined that the entire list of compounds could be resolved and detected using a portable Photovac[®] 10S70 gas chromatograph/photomization detector (GC/PID). QA/QC protocols and SOPs were developed at an analytical data quality level consistent with CLP protocols. Customized data processing software was developed to meet the field data deliverables requirements and to facilitate the validation process. The on-site facility results versus CLP laboratory results for TCE and PCE in 100 split sample analyses were consistent with respect to "clean" soils and "dirty" soils.

The second CERCLA remediation site was a former lagoon area in the southern New Jersey containing sludge bands and soils with elevated levels of chromium, copper, and nickel. The selected remedy utilized "Soil Washing" which required quick analytical feedback to determine optimal plant operating efficiency and to determine the extent of excavation required in each area. The real-time analytical results required by the plant had to be representative of CLP results in order to ensure comparability with EPA required analyses. X-Ray Fluorescence (XRF) Spectroscopy was selected as the appropriate analytical instrumentation and methodology. A data comparability study was performed to ascertain the dynamic accuracy range of the instrument compared to CLP laboratory results. A correlation between the field XRF and CLP laboratory results for 20 authentic split samples was statistically very good for all three analytes even though the samples contained contamination well below the action levels.

As illustrated by these case studies, field analytical data has proven to be a powerful tool toward proper, complete, and cost-effective execution of remedial efforts. When "real-time" data is available for many interrelated aspects of a project, otherwise tedious remedial efforts can become relatively inexpensive and quickly paced. Both of the cases presented resulted in substantial savings on analytical cost as well as the savings realized through efficient and effective process and schedule management. However, these benefits can only be realized through proper analytical strategies developed around well-defined project and data quality objectives.

INTEGRATED TECHNOLOGIES TO EVALUATE THE IMPACT OF EMIS- SIONS AT A PETROLEUM RECOVERY SUPERFUND SITE

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The integration of several technologies proved successful in assessing the air pathway impact of site emissions on a neighboring community. This presentation will illustrate the results of integrating several technologies to assess the impact of volatile organic compounds (VOCs) from a petroleum recovery superfund site on the ambient air quality of an adjacent neighborhood. The technologies integrated include:

- TAGA analysis
- GC FID analysis
- microchip GC analysis
- GC AID analysis
- GC/MS analysis
- Flux chamber sampling
- Summa canister sampling
- air dispersion modeling

The Trace Atmospheric Gas Analyzer (TAGA) mobile mass spectrometer/mass spectrometer (MS/MS) was used to monitor the air concentrations while traveling the roads of the neighboring community and to analyze samples collected by other methods (i.e., tedlar bag grab samples). Seven flame ionization detector (FID) gas chromatographs (GCs) were placed on site and transmitted readings to the command post where they were logged at 15-minute intervals. A field portable capillary column microchip GC was used for detecting and quantifying compounds in soil gas samples. A GC/MS analyzer and a lap-top PC-controlled field portable GC argon ionization detector (AID) were used to confirm the TAGA results conducted on grab samples. Flux chambers were used to collect air samples on top of buried lagoons in order to determine the emission rate of each lagoon independently. Summa canisters were used to collect ambient air, flux, and soil gas samples. These samples were sent to labs for analysis to confirm all analyses. Air dispersion modeling allowed for the back-calculation of emission rates from the site operations and the determination of short- and long-term impacts from the site's air emissions.

The integration of these technologies facilitated:

- the estimation of emissions from buried waste lagoons
- the estimation of emissions from site operations
- the identification of additional or incidental sources
- the confirmation of modeling procedures
- the estimation of background concentrations
- the estimation of impact from site emissions

ENVIRONMENTAL AND ATMOSPHERIC MEASUREMENTS AND DATA COLLECTION FROM A HOT AIR BALLOON

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Tethered and free-flying manned hot air balloons have been demonstrated as platforms for various atmospheric measurements and remote sensing tasks. We have been successfully accomplishing these tasks since the winter of 1993. These platforms are inexpensive to operate, do not cause atmospheric disturbances due to high speed flight, are extremely stable and free of vibrations inherent in aircraft structures. The equipment operated and tested on the balloons includes, but not limited to, various spectrometers, high resolution CCD cameras, mid- and far-infrared cameras, a radiometer, a FLIR system, video recording equipment, ozone and nitrogen oxide analyzers, collection canisters, aerodynamic particle sizer with associated computer equipment, tether-sonde, and a 2.9kW portable generator carried beneath the balloon providing power to the equipment. Vertical profiling of pollutants have been accomplished in wintertime inversions and summertime conditions in urban areas of Utah. At the Dugway Proving Grounds, we have flown the balloon into diesel and fog oil smokes generated by turbine generators to obtain particle size distributions. The Defence Research Agency, part of the United Kingdom's Ministry of Defence, teamed with the 545 Test Group, Utah Test and Training Range, Hill AFB, Utah, to acquire data on atmospheric transmission through slant paths from 0 to 30 degrees, with the horizontal, at various ranges. Measurements of the image profile of a focussed argon ion laser beam were made with a high resolution CCD camera in the balloon. Fast radiometric scans of the balloon were attempted from a radiometer located on the ground. Fourier transform infrared (FTIR) spectrometers operating in the 8 to 12 micron region were used in conjunction with a coaligned Inframetrics infrared camera, with various narrowband filters, to characterize chemical plumes being emitted from a simulated industrial stack. The instruments were mounted in the hot air balloon operated at several different altitudes in order to measure the near field (0-800 meter) plume characteristics at different effective horizontal resolutions and to assess the sensitivity of these instruments for measurements in the far field regime. We will be performing an experiment where there will be controlled releases and collections of gases in order to provide truth data for measurements that will be taken by a CO₂ lidar system. Ultraviolet scattering and absorption experiments have been carried out at the Dugway Proving Grounds, UT with the Cosmic Ray Research Team of the University of Utah using their High Resolution ultraviolet sensor arrays. This paper will present and discuss the results of these tests. In addition, results from other tests performed with the balloon, to include remote sensing through various obscurants and a test of a low cost optical tracking system, will be presented.

NEW ADVANCES IN SOIL GAS SURVEYS: CONTINUOUS MONITORING USING A MASS SPECTROMETER (SVS-MS)

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Onsite Environmental Laboratories, Inc. (ONSITE) of Fremont, Ca. has integrated a Viking SpectraTrak 620 GC/MS system with conventional direct push soil gas survey hardware to produce an innovative approach to soil vapor surveys. Hundreds of data points may be obtained each day with a complete Mass Spectrometer (MS) scan of each soil gas sample at an analysis cost of \$7 to \$20 per point.

SVS-MS significantly improves the quality and increases the quantity of analytical data available through soil vapor surveys. Current methodologies limit the number of samples per day by the run time of the GC and the required detectors for the specific analytes. ONSITE's new methodology enables clients to receive their data in 3D at the end of the day so that they may evaluate activities and make decisions in real time.

Using argon as an internal standard, ONSITE is able to quantitate contaminants more accurately, by eliminating the inherent error in determining sample volume using conventional methods.

Utilizing the MS scan, ONSITE is able to measure not only the target compounds but also such tracers of aerobic and anaerobic bacterial activity in soil as CO₂, CH₄ and H₂S. These key indicators may provide critical information to the geoscientist evaluating the subsurface conditions.

Data acquisition, or "analysis time" as is referred to in conventional soil gas surveys, is no longer an issue with ONSITE's new process. The soil gas is introduced directly to the MS via a silicon rubber membrane resulting in real time, instantaneous results. Project managers in the field are now able to evaluate the subsurface conditions in real time as the probe is being advanced.

MICELLAR ELECTROKINETIC CHROMATOGRAPHY: A NEW TOOL FOR FIELD-SCREENING OF SEMIVOLATILES

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Micellar electrokinetic chromatography (MEKC), one of many operational modes of capillary electrophoresis (CE), is a new technology with great potential for field screening. MEKC is a powerful and simple separation/determinative technique when coupled with ultraviolet/visible or laser-induced fluorescence detection. MEKC can be combined with recent developments in supercritical fluid extraction (SFE) and solid phase extraction (SPE) to achieve very rapid sample analysis. The U.S. EPA Environmental Monitoring Systems Laboratory, Las Vegas, has an active interest in extraction technology as well as separation/determinative techniques. One of the goals of our research using MEKC has been to reduce the time of SFE/SPE/MEKC to an average of about 6 to 9 min. per sample. The power of MEKC will be shown for typical semivolatiles, such as phenols, amines, and polynuclear aromatic hydrocarbons. Advantages of MEKC over other techniques (e.g., gas chromatography and liquid chromatography) include complete regeneration of buffer/micellar agent and complete capillary rinsing between samples; use of inexpensive fused silica columns; rapid separations of analytes on short capillaries; inherently high on-column sensitivity; high theoretical plate numbers; and ruggedness. Ultraminuturization of the CE technique and sub-second separation times have also been demonstrated by other researchers.

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), prepared this abstract for a proposed oral presentation. It does not necessarily reflect the views of the EPA or ORD.

ENVIRONMENTAL APPLICATIONS OF A SIMPLE BIOLUMINESCENCE DEVICE

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The measurement of Adenosine Tri-Phosphate (ATP) has classically been used as a means to monitor the presence of bacteria in clinical materials, particularly urine; however, the instrumentation, reagents, and variability of the technique have excluded its use in environmental applications. A system is described here which has simplified the performance of this measurement and may result in the use of bioluminescence as a means to monitor fluxes in the biomass of the environment.

The classical system utilized liquid reagents with a rather large, cumbersome detector system. Advances in drying techniques and miniaturized electronics have resulted in the development of a potentially field-deployable system. The reagents are stabilized by spray-drying them on to a ticket and the electronics fit into a device about the size of a large calculator. The sample is introduced into a tube and filtered to remove interferences. Other pretreatment steps may include lysis of somatic cells. A bacteria/pollen specific lytic agent is then added, and the sample is then transferred to the test ticket. It is then placed into the instrument and read.

Results in both the laboratory and the field show that the system has a sensitivity of about 10^5 organisms/ml for both sporulated and vegetative bacteria. Several pollens have also been evaluated and shown to be reactive. Work is continuing in the evaluation of other biological materials, such as mold spores.

The data suggests that this device can be interfaced with simple sampler devices and used as means to monitor the biomass content of air and water. Environmental applications could include such areas as sludge analysis and indoor-air quality. Several examples of the applications of this technology to these areas will be discussed.

**EXPEDITED SITE CHARACTERIZATION (ESC) USING THE M³ APPROACH
M³ = MASSIVE, MODERATE, MINIMUM**

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The main objective of this approach is to quickly and cost effectively identify and classify potential Areas of Concern (AOCs) as "clean" or contaminated, thus allowing potentially responsible parties (PRPs) to save limited resources by ceasing costly investigations and undertaking removal type actions expeditiously. The ESC M³ approach also overcomes the high degree of uncertainty typically associated with traditional site investigations resulting from a lack of comprehensive scoping. Thus, EPA Region IX has agreed to accept and use, for risk assessment purposes, the data generated from the ESC M³ approach, providing the data quality is known and confirmation analyses are performed. The extraordinary benefit will be to eliminate any further action on those AOCs found to be clean using this approach. Finally this approach reduces the large number of non-detected samples that are customarily submitted for CLP-type (i.e., Contract Laboratory Program) analyses.

The ESC M³ approach consists of the following three steps: Step 1) a "massive" sampling effort is first conducted at an AOC (e.g., 200 samples are collected using a grid approach); the samples are analyzed on a daily basis using real time onsite methods and field screening (FS)-type data are generated; Step 2) a "moderate" sampling effort is then conducted (e.g., 20 splits of the Step 1 samples from "clean" locations in a clean AOC or a lesser amount of splits from "hot" locations in a contaminated AOC, along with boundary location [exterior] samples) to provide onsite verification of the FS-type data; the samples are analyzed using onsite CLP-type methods and field quantitation (FQ)-type data are generated with an agreed upon level of QC; and finally, Step 3) a "minimum" sampling effort is conducted (e.g., 4 splits of the Step 2 samples from "clean" locations for a clean AOC and from "hot" and boundary locations for a contaminated AOC) to provide verification of the FQ-type data; these confirmation samples are sent to an offsite laboratory for analysis, and CLP-type data are generated.

Note that both the FS and FQ steps will generate quantitative results (i.e., concentration values for every analyte). The main difference is in the level of precision. Studies have shown the FS data precision to be comparable to CLP-type precision. While the level of QC for the CLP-type data is known, the appropriate levels and amounts of QC for the FS-type and FQ-type data still need to be defined.

All FS-type and FQ-type data will be loaded into an onsite data management system with three-dimensional (3-D) visualization capabilities. These data will be viewed by site managers onsite and by Remedial Project Managers (including regulatory agencies) via modem transmissions at the end of each day. Thus, remedial decisions will be made on a daily basis, rather than, as is typical of current practices, months or years after each phase of the site characterization field work has been completed. It is possible that only one field investigative effort will be required using this approach since data gaps can be seen on a daily basis and then accounted for on succeeding days while the investigation is ongoing. Remedialization time and costs can be eliminated greatly accelerating the remedial process while drastically reducing the costs.

Recent cost estimates at a Federal Facility in California have shown this approach can realize a substantial cost savings when compared to the traditional RI/FS site characterization approach. This approach is being planned for implementation at two Federal Facilities during the summer/fall of 1994. Case studies will be presented at the February 22-24, 1994 FS Symposium in Las Vegas, NV.

ACCELERATED SITE INVESTIGATION USING FIELD SCREENING METHODS: A CASE STUDY OF A TEAM APPROACH

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Field screening data is becoming a necessary tool for environmental site investigations. The use of mobile and close-support laboratories has allowed for rapid, informed decision-making and is evolving a new approach to site investigations. Analytical Technologies, Inc. and Site Works, Inc. have teamed to provide quick turnaround data to support a large scale site investigation under the Navy Clean program during the summer of 1994. The teaming arrangement on this project has combined each company's expertise into an integrated analytical laboratory providing high volume, quick turnaround methods for TRPH, PCB's, PNA's, and metals. The methods used include fast GC/MS (SIM) for volatile and semi-volatile organics, field transportable ICAP for metals, and portable IR for TRPH. This paper will discuss the use of field screening data and a team approach to providing on-site analytical services. Issues such as mobilization, quality assurance, data quality objectives, operating considerations, and field support of a long-term, remote project will be discussed.

THE USE OF RAPID TURN-AROUND HEATED HEADSPACE/GAS CHROMATOGRAPHY TO SUPPORT REGULATORY SOIL CLEAN-UP STANDARDS

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Previous studies have indicated that gas chromatography can be effective as a screening tool to delineate areas of contamination on-site; however, there are few data supporting the use of a field laboratory to fully support a large scale site remediations. In recent years analytical instrumentation has evolved to withstand the rigors of field work, and field analysis has coincidentally evolved from a "screening" tool to a true support tool able to provide useable data in defense of state soil clean-up criteria. A water extraction/heated headspace method was selected for this project because of its precision, accuracy and ability to turnaround as many as 30 environmental samples per day per instrument. In addition to evaluating soil clean-up criteria analytical data enabled the remediation contractor to optimize the treatment systems and focus their effort.

During this project approximately 2500 samples were analyzed in the field laboratory over a period of seven months using the heated headspace method. The state and project requirements, in addition to the duration and volume of samples involved, required that a well defined QA/QC program be implemented and enforced. Quality control criteria were established to emulate standard USEPA GC protocols (Methods 8000/8010/8020) for calibrations, blanks, surrogate spikes, laboratory control samples, field duplicates, matrix spikes, and independent check standards. Site specific control limits for these measures were often more restrictive than the recommendations of the standard method. In addition to these internal QA/QC measures, approximately 10% of the samples were split with a certified laboratory for USEPA method 8010/8020 analysis. Using these data an extensive database was established quantitatively evaluating the precision, accuracy, representativeness, comparability and completeness of the field heated headspace method.

This paper discusses the heated headspace method, sample handling and analytical protocols as well as the project and Data Quality Objectives. Extensive statistical analysis of both the internal laboratory QC data and the independent split data validate the heated headspace method as a viable "stand-alone" alternative to off-site analysis. The paper discusses the strengths and limitations of the on-site method, and how it can best be incorporated into today's budget and time sensitive remediation projects.

TANDEM LEVEL II AND LEVEL III FIELD ANALYSES FOR IN-FIELD DECISION MAKING

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In order to provide cost effective and timely analytical support for a site investigation in central Pennsylvania, Environmental Resources Management-Field Analytical Services Technologies (ERM-FAST[®]) performed field soil analyses targeting both Level II and Level III data quality objectives. Of specific concern were 17 volatile organic compound, including the less volatile dichlorobenzenes. The soil levels of these contaminants of interest were required on a rapid turnaround basis in order to make decisions regarding subsequent boring locations and other analytical logistics.

For 50 anticipated boring locations, rapid turnaround Level II analyses were needed for each of 5 intervals per boring. Rapid turnaround head space analyses (ca. two hours) were performed on-site using a Perkin-Elmer Autosystem GC with a PID and an ELCD in series. These Level II GC results were used to determine the best locations for subsequent borings, the most illustrative three of five intervals to be analyzed by on-site GC/MS for Level III data quality, and to infer the proper dilution factor for each sample analyzed by on-site GC/MS. On-site GC/MS was performed using a Viking 640 GC/MS system with cryo focussing, and in accordance with SW-846 Method 8260.

Presented is a statistical comparison of on-site GC/MS, GC, and commercial laboratory confirmation analyses. Project cost, time and strategic benefits are also discussed, as well as instrument performance and instrument selection strategy.

THE DIFFUSION OF INNOVATIONS IN THE ENVIRONMENTAL MARKET-PLACE

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Recently, there has been much talk by public officials, industry and others about the need to facilitate the adoption of new technologies within the environmental marketplace. Currently there are a number of barriers that exist which hinder the adoption process. These barriers include: permit and compliance programs and requirements, the trend to legislate technological solutions, balkanized regulatory programs, a dearth of education and training programs in the use of new technologies, and the lack of neutral third party evaluation and validation of new technologies.

While all these barriers play some role in the adoption process, often the degree to which a specific barrier hinders the use of an innovative technology is not well understood. Therefore, individuals both in the public and private sector, may develop solutions to perceived barriers that ultimately may have very little impact in increasing the rate of adoption for a new technology.

To address the issue of barriers to technology adoption, one must first obtain a fundamental understanding of how a new technology is diffused within a given sector of the environmental marketplace. Without this understanding, the barriers that one may try to alleviate may not be the critical barriers that are hindering the use of a new technology.

This presentation will identify some of the most critical barriers that currently hinder technology adoption. Secondly, the various methods by which technologies are diffused into the environmental marketplace will be described. Examples will be given where changes in policy to encourage technology adoption have not had the intended impact due to the lack of consideration for the diffusion process. The presentation will conclude with recommendations of how to incorporate the knowledge of diffusion processes into technology adoption policies, leading to greater success for new technology developers in the environmental marketplace.

IMPEDIMENTS TO NEW TECHNOLOGIES FOR HAZARDOUS AIR POLLUTANT MONITORING

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This presentation describes policy and regulatory impediments encountered in efforts to foster and adopt new measurement technologies for hazardous air pollutant monitoring required by the Clean Air Act of 1990. Through the Center for Process Analytical Chemistry, a National Science Foundation public-private research partnership program, our work has revealed that introduction of new monitoring methods is not limited by technology nor market considerations but by regulatory impediments. Cooperation between petroleum industry members of CPAC and the EPA to identify and introduce new technologies indicated that a variety of instrumentation could be applied to hazardous air pollutant monitoring required by these new regulations. Yet, Agency programs are ill equipped to enlist them. We have found that formal rule making procedures do not always reach interested scientists and lack flexibility to incorporate new technologies. Communication of Agency research interests and programs with the private sector could be improved; participation in scientific meetings and workshops apparently is not enough to stimulate collaboration. Agency surveys of new technologies are incomplete. Surveys do not sample the technical community adequately.

Frequently, industry responses lack detail. Technical support, essential to define performance acceptance criteria, protocol review, and quality assurance guidance is not widely available. Yet these ingredients are essential for industry to invest in research of promising technologies. We have taken steps to improve opportunities. Through formation of a workgroup, efforts are underway to produce a directory of organizations and individuals to improve the coverage of Agency technology surveys and to improve industry participation in these surveys. Through collaborative field studies, efforts are underway to identify regulatory barriers, enlist the support of Agency officials to reduce or eliminate them, and to develop performance criteria, protocol reviews, and data evaluations that will foster investment by industry in new monitoring technology research. From this experience, performance criteria, quality assurance requirements, and comparability demonstrations will be defined in more detail for subsequent studies. The take home message from our work indicates that greater effort to identify and reduce regulatory barriers is warranted to foster the introduction and application of new technologies for environmental monitoring.

A SUMMARY OF METHODS FOR FIELD ANALYSIS OF HYDROCARBON CONTAMINATED SOIL AND GROUNDWATER

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A variety of improved field-based technologies, methods, and procedures are being used to perform rapid and cost-effective analysis of organic compounds in a more streamlined process than is possible with conventional assessments. In many of these investigations, the appropriate use of these methods can result in an effective characterization with increased spatial and temporal information in less time and with fewer phases of assessment than is typically done using conventional sampling technologies and off-site or fixed-base laboratories. Although the use of field analytical methods for conducting on-site analysis of hydrocarbon-contaminated soil and groundwater is increasing, such methods are not currently in wide use primarily for the following reasons: (1) the lack of regulatory acceptance, (2) the perception that field analytical methods do not provide data of adequate quality for making decisions, and (3) the perception that field analytical methods do not necessarily result in cost savings.

The American Petroleum Institute (API) is currently developing a series of technical documents to address key issues that technical, project management, and regulatory personnel may need to know in order to select and use these field analytical methods to their full potential. The overall objective of this project is to develop technical guidance in a series of documents to be used in establishing an independent set of field analytical methods that do not require constant validation by comparison laboratories and can be used in on-site decision making.

This paper will discuss these documents being developed by presenting an overview of the following: (1) a compendium of recognized and emerging field analytical methods, (2) a framework for the appropriate selection and use of recognized field analytical methods in the site characterization/corrective action process, and (3) a protocol for producing and evaluating field performance data. The compendium focuses on the most widely used field analytical technologies including total organic vapor analyzers, portable gas chromatographs, immunoassay and colorimetric kits, infrared detectors, and dissolved oxygen/reduction-oxidation potential electrodes. The compendium also lists other field analytical techniques that are not as widely used but show promise for future application. The framework provides a characterization process based on site-specific considerations and goals and identifies the advantages and limitations of different field analytical methods in achieving site-specific goals. The field performance protocol provides a means to establish method performance and to meet data quality objectives using field-generated analytical data. These documents can assist in generating higher quality, reproducible, and standardized field analytical data in order to minimize the use of costly and time consuming off-site sample analysis, and increase the credibility and use of field data for risk evaluation and remedial decision making.

FIELD IMPLEMENTATION COMPLEXITIES OF EPA DEVELOPMENTAL AND REAL-TIME METHODS DURING REMEDIATION AT REMOTE HAZARDOUS WASTE SITES: CASE STUDY

Elizabeth L. Green, Eagle Environmental Health, Inc., Elizabeth A. Cunningham, Tenneco, Inc., Carl Grabinski, Joslyn Corporation

The objective of this presentation is to provide insight into the complexities of field implementation of EPA developmental methods and real-time monitoring methods for polynuclear aromatic hydrocarbons, pentachlorophenol, particulates and metals at remote hazardous waste site remediations.

Ambient air monitoring was conducted at an abandoned creosoting plant in Louisiana in order to characterize the potential off-site migration of selected constituents through the air pathway during remediation of the site. Off-site migration of constituents was a concern due to the close proximity of commercial and residential areas to the site. In addition, real-time monitoring was conducted so that site activities could be adjusted based on action levels in a timely fashion in order to reduce any unacceptable levels of constituents identified by the real-time monitoring.

Ambient air monitoring was conducted for polynuclear aromatic hydrocarbons and pentachlorophenol using EPA TO-13 method; and for total suspended particulates and metals using the method from 40 CFR, part 50, Appendix B. Real-time monitoring was conducted for particulates using a Mini-Ram and for organics using a MSA Gascarder photo-ionization detector. Monitoring was conducted over the course of a year (1992) in three phases. Approximately 8,000 data points were generated during the course of this project.

Field implementation of multiple developmental and real-time methods presents many challenges. In particular, relative to developmental methods, laboratories are generally unfamiliar with the developmental and analytical methods and often do not maintain the required media and equipment because of the limited demand for the method. Additionally, because the method is not routinely performed by many laboratories, the potential idiosyncracies of the method may not be well understood and therefore quality control procedures may not be well established. Poor turnaround times due to unanticipated analytical complications can also have a significant impact on the usefulness of the data on an ongoing basis.

From a logistic standpoint, many factors must be considered in order to implement a successful air monitoring program which will provide defensible data. Seemingly minor factors such as the source of effective power at remote locations (because generators can produce contaminants which could bias organic results) can have a significant negative impact if not appropriately considered in the initial planning of the project. In addition, daily start-up and shut down procedures of multiple pieces of equipment involving many different methods can put strains on manpower and quality control procedures.

Finally, a management system for large amounts of data must be pre-designed with the end points in mind in order to place the data in a usable format that facilitates statistical analysis and reporting at the completion of the project.

LASER-INDUCED FLUORESCENCE (LIF) SPECTROSCOPY OF POLYNUCLEAR AROMATIC COMPOUNDS (PAC) AND MINERAL OILS IN HUMIC ACID- AND SOIL-CONTAINING COMPARTMENTS

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Laser spectroscopy has been established as an important tool in environmental research. Among the various spectroscopic techniques employed, laser-induced fluorescence (LIF) is distinguished by outstanding sensitivity and high selectivity, and has therefore found considerable fundamental and practical interest. However, due to the heterogeneity and opacity of soil matrices, the qualitative and quantitative investigation of fluorescence signals obtained from soil-containing environmental compartments imposes usually considerable experimental difficulties.

In order to investigate the benefits and limitations of luminescence analysis in humic acid- and soil-containing samples we use selected PAC as model fluorophores and mineral oils as realistic contaminants. Single- and multi-channel stationary and time-resolved fluorescence measurements were performed to obtain basic photophysical information and experimental data relevant for the in-situ and on-line analysis of soil-containing samples. Results of the following investigations will be presented:

- Investigation of the quenching of PAC fluorescence by humic acids. The combination of stationary and time-resolved measurements provides insight in the mechanisms of fluorescence quenching and in fluorophore/humic acid-interactions.
- LIF-measurements directly from the surface of soils doped with model and mineral oils. Quantitative relationships between fluorescence intensities and concentrations were obtained in ranges relevant for realistic PAC and mineral oil contamination of soils.
- In-situ and on-line LIF-measurements on soil columns and in bioreactors. The determination of probability distributions of fluorescence signal intensities yield information about the distribution of the contaminants on the soil surface and the degree of heterogeneity in these systems.

The results obtained in these studies will be evaluated with regard to the development and application of in-situ and on-line luminescence analytical methods under conditions realistic e.g. for soil decontamination processing.

THE INFLUENCE OF PAH CONCENTRATION AND DISTRIBUTION ON REAL-TIME IN SITU MEASUREMENTS OF PETROLEUM PRODUCTS IN SOILS USING LASER INDUCED FLUORESCENCE

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This paper describes the results of a real-time, in situ survey using laser induced fluorescence techniques to evaluate the extent and type of hydrocarbon product contamination in soil. Real-time soil petroleum product measurements were made through the sapphire window of a probe that was pushed into the soil from a truck-mounted cone penetrometer test (CPT) system. A 337-nm excitation source induced fluorescence in polynuclear aromatic hydrocarbons (PAH) present in petroleum products, and the signal measured with a fluorometer. After removal of the soil probe, a split spoon coring system was used to collect discrete soil samples down the push hole. Additional discrete laser induced fluorescence measurements were made using soil removed from composites of six inch split spoon cores.

Discrete soil samples were also collected from the homogenized six inch split spoon core, extracted and analyzed for over forty parent and alkyl substituted PAH compounds by gas chromatography/mass spectrometry using a modified EPA Method 8270. The modifications were: 1) redefinition of the surrogate and internal standard, and target analyte list to include primarily diagnostic hydrocarbon compounds, 2) the use of selected ion monitoring to increase analytical sensitivity, and 3) the quantification of alkylated PAH homologues (e.g. C₁ to C₄-phenanthrenes, etc.). This method produced data that was used to identify product types present in the soil, determine linkages between samples and source products, and provided a basis for evaluating the relationship between PAH concentrations and laser induced fluorescence measurements made in the soil borings. Total PAH and individual PAH concentrations in the soil were evaluated relative to product type and laser induced fluorescence response using principal component analysis.

The laser induced fluorescence system used in this study was found to provide continuous and reliable real-time hydrocarbon profiles - a dramatic advantage over time consuming and costly discrete sampling/analysis methods traditionally used for site monitoring well placement, monitoring of remediation countermeasures, or tracking hydrocarbon contamination to sources.

A FIELD SCREENING METHOD FOR TRACE ELEMENTS IN SOIL

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A field screening method for heavy metals in soils, sand and sewage sludge samples has been studied by means of time resolved optical emission spectrometry from laser induced plasma. The instrumental setup has been developed as a fast screening detector for a future application as an on-line and in-situ method. A solid state laser pulse is focused onto the sample to produce plasma. An optical fiber system is used to collect the light emitted from the plasma. Spectral and temporal resolutions are obtained by means of a small spectrograph and an intensified photodiode array. Several experimental parameters have been optimized. This method enables fast analysis, without any chemical preparation of the samples. Various factors affecting the detection limits and the quality of the analysis have been investigated. These include aerosol production, crater formation, size effects, timing effects, laser intensities and humidity. In order to improve reproducibility of elemental analysis, a special data analysis program has been developed. It consists of a data analysis program and of the Principal Component Regression (PCR) calibration technique, which utilizes many spectral lines for each element. A special renormalization algorithm has been tested for internal calibration. The computer program provides good calibration plots and detection limits in the $10 \mu\text{g g}^{-1}$ range, which are usually below the ecological requirements.

LASER INDUCED BREAKDOWN SPECTROSCOPY FOR RAPID DELINEATION OF METALS IN SOILS

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Laser Induced Breakdown Spectroscopy (LIBS) has been used effectively in recent years as a rapid method for classification of metal surfaces. The established technique involves a laser beam which is tightly focused onto a metal surface to initiate the formation of a plasma. The time resolved emission of the plasma is collected and analyzed to reveal the characteristic spectral signature of the metal.

The present work addresses the potential application of LIBS over fiber optic cables as a real time in-situ metal contamination field screening tool for use in soils in conjunction with a cone penetrometer. In this application the intense beam of a Nd:YAG laser is launched into a fiber optic cable and subsequently focused onto a soil sample spiked with a metal contaminant. The system limiting factors of required surface power density, fiber optic damage threshold, and signal collection efficiency will be addressed.

PORTABLE AIR MONITORING LABORATORIES

D. Einholz, K. Belius, J. McCullough, and J. Valentine, Arthur D. Little, Inc., 15 Acorn Park, Cambridge, MA 02140

The U.S. Army contracted with Arthur D. Little, Inc. to design, fabricate, test and deliver a suite of specialized testing laboratory facilities for manned and unmanned use to support Project Retrograde, the removal of chemical munitions from West Germany. This project, which was carried out for the Chemical Research, Development and Engineering Center (CRDEC) of the U.S. Army, Munitions, and Chemical Command (AMCCOM), was specifically focussed on designing and building facilities which would be used to perform the vital functions of monitoring the air at many points on the transport vessels for any traces of the chemical agents which might inadvertently escape from the munitions cargo.

The design and fabrication planning had to incorporate the following key needs, among others:

- The nature of the mission was such that all apparatus, materials, spare parts for both apparatus and equipment, and utility needs had to be carried with the suite of labs. There would be no supply depot or repair/spares accessible during the mission;
- The facilities had to be fully self-contained within standard International Shipping Organization (ISO) van/containers, and mounted/supported to withstand stresses of shipping and intended use; all spare parts were to be carried in the ISO containers;
- The facilities and utilities had to accommodate and support the intended chemical measurement techniques, principally specialized sample collection/transfer operations and gas chromatography, including the handling of small amounts of toxic chemical agent materials to prepare instrument calibration standards over the entire mission duration;
- The portable air monitoring laboratories were to be manned full-time by three persons while the test shelters would have automated test equipment and be manned only on a part-time basis;
- There was a requirement for equipment redundancy to ensure no interruption in laboratory function, and the equipment mounting/location and spare part storage had to be appropriate;
- The laboratory function could not be interrupted by loss of external electrical power or by extremes in vessel motion, including + or - 30° roll;
- HVAC and special air filtration would be required to provide a comfortable work environment and an over-pressure of "clean" air to prevent incursion of agent-contaminated air in the event of any cargo leakage.

Lessons learned during the successful design and operation will be discussed.

FIELD SCREENING FOR MILITARY UNIQUE COMPOUNDS UTILIZING THE REAL TIME ANALYTICAL PLATFORM (RTAP)

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The U.S. Army Operations Directorate, Chemical Support Division, Monitoring Branch monitors airborne levels of a variety of military compounds to ensure worker safety and the protection of the environment at sites where chemical agents are used or stored and during disposal operations. The monitoring system fielded in the Real Time Analytical Platform is based on an air sampling technique using solid sorbent collection and thermal desorption technology combined with capillary gas chromatography with multiple gas chromatography detectors. The mobile unit is equipped to analyze a variety of surety compounds, delivering documentation to meet regulatory requirements with a high level of sensitivity, selectivity, extensive dynamic range with excellent linearity, in real time or near real time. The system can sample continuously through a heated transfer hose for direct, on-line requirements such as site monitoring projects, or easily switch to off-site sampling capability using solid sorbent cartridges for sample collection that are returned to the mobile unit or laboratory for analysis.

The RTAP analytical system replaces multiple methods using diverse instrumentation with a single instrument configuration. Training time is reduced, since technicians are able to analyze a wide variety of compounds with the same instruments using very similar methods. Wet chemistry procedures that generated hazardous waste have been eliminated, resulting in savings on decontamination and disposal expenses.

This paper will describe the instrumentation, its use for both screening and confirmation of various chemical surety materials, and describe how the units have been fielded as part of emergency response efforts at several sites.

GEOPHYSICAL SURVEYING USING A REMOTELY CONTROLLED VEHICLE

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A remote characterization system (RCS) will be used at the Aberdeen Proving Grounds (APG), Edgewood Area in August 1994 to execute geophysical surveys of ~300 acres at Carroll Island and 20 acres at Graces Quarters. The intent of the program is to verify the boundaries of potential burial or disposal sites identified during previous investigations demarcated by pits, mounds, or depressions and to check for new areas identified during site walkovers.

The RCS development was a coordinated effort between Pacific Northwest Laboratories, Oak Ridge National Laboratory, Idaho National Engineering Laboratory, and Sandia National Laboratories. The remotely piloted vehicle can be equipped with various types of equipment including magnetometers, electromagnetic induction sensors, ground-penetrating radar (GPR), gamma radiation detectors, and phototonization detectors. The vehicle is designed with a minimum of metal to present a low electromagnetic signature to each of the sensors, thereby limiting interference with sensor operation. A base station houses the controls and monitors. Control commands sent from the base station and data from two video cameras mounted on the front and back of the RCS are used to "drive" the vehicle. The commands are transmitted via an ethernet link on a microwave frequency. Sensor readings flow continuously from the RCS to the base station. A differential global positioning system (GPS) is used to track the position of the vehicle in real time to submeter accuracy. The GPS data can be processed to achieve an accuracy to within 15 cm.

Some of the intended APG Edgewood Area sites have had previous geophysical surveys performed, including an EM-31 electromagnetic survey. The proposed RCS program—consisting of GPR, magnetometer, and EM-61 electromagnetic surveys—has been designed (1) to confirm previous geophysical surveys; (2) to supplement previous geophysical survey results, specifically through the use of the more-sensitive EM-61; and (3) to collect data in site areas previously inaccessible to traditional geophysical methods. A benefit of using the RCS is that it can access marshy areas and areas with uneven terrain that were difficult to get to during previous surveys. Also, because the three tools (GPR, magnetometer, and EM-61) will be on the RCS simultaneously, the survey will be more time effective than if traditional survey methods are used.

The intent of this study is to demonstrate the versatility of the RCS and the application to sites with access problems such as unsuitable terrain and to areas in which contamination levels are dangerous to humans. Additionally, the ability of the RCS to carry more than one sensor tool during operation will eliminate the need for multiple traverses (per geophysical tool) over difficult terrain.

*Work performed by Automated Sciences Group, Inc., under general order contract number 30B-9778C with Martin Marietta Energy Systems, Inc.

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A VEHICLE MOUNTED MULTISENSOR ARRAY FOR WASTE SITE CHARACTERIZATION

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OBJECTIVE

This presentation will provide a brief overview of the IGMMDT sensors and of the IGMMDT integrated system design. Preliminary results of field tests of the IGMMDT array will be presented. The potential applicability of the IGMMDT platform, its sensors, and its data analysis algorithms to field screening and site characterization for hazardous wastes and toxic chemicals will also be discussed.

TYPE OF TESTING AND EVALUATION TO BE CONDUCTED

The Improved Ground Mobile Mine Detection Testbed (IGMMDT) is a vehicle mounted, multi-sensor array designed and fielded for the U.S. Army's Ft. Belvoir Countermine technology group. The purpose of IGMMDT is to locate, identify, and mark anti-tank and anti-personnel land mines making use of multiple sensor technology and data fusion to correlate sensory inputs. The sensor array consists of ground penetrating radar, metal detectors, dual-band infrared, and optical cameras. Data collected by these sensors are processed in real-time (100's of milliseconds) using high speed signal and image processing hardware. The data from each sensory channel is processed at two levels. The first level of processing (low level inference) uses advanced signal and image processing techniques to analyze the data acquired by the sensor to locate and identify threats (mines) in the sensor's field-of-view. The second level of processing (high level inference) then forms a consensus of the outputs from the low level inference processes of all the sensors to make a final threat/non-threat decision. A neural network is used to perform the high level inferencing and is trained to correlate the outputs from the different sensors over a range of environmental conditions and identify threats. IGMMDT is designed to be a modular sensor testbed which provides rapid site survey capability and in-situ data analysis for site characterization. As such, different and newly emerging sensor technologies may be easily integrated onto the array, including those that are used for the mapping and characterization of hazardous waste sites or for the detection and characterization of unexploded ordnance (UXO).

E-SMART: A STANDARD APPROACH TO REAL-TIME, IN SITU ENVIRONMENTAL MONITORING AND PROCESS CONTROL NETWORKS

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To meet growing environmental needs, both the government and the private sector are developing better sensors for a wide range of chemical and radiological contaminants for use in real-time, in situ environmental monitoring applications such as in situ bioremediation or post closure monitoring. However, it is desirable to ensure that the resulting sensors utilize a standard approach to network communications so that data or operating commands can be exchanged between any smart sensor, actuator, or control computer system on the network. This goal will allow sensors or other remediation process control devices such as valves or pumps to be easily installed, configured, and operated within large, heterogeneous environmental monitoring networks including sensors and actuators of different types from different manufacturers. It will also ensure that the data generated by diverse environmental sensors can be combined and analyzed in useful ways. Furthermore, this goal promotes the use of modular hardware and software components allowing for greater flexibility and lower cost in designing and implementing a monitoring network.

The Environmental Systems Management, Analysis and Reporting Network (E-SMART) was developed by General Atomics for the US Air Force Armstrong Laboratory in response to these needs. E-SMART is a fully integrated environmental monitoring system with expert system capabilities. It should significantly reduce the costs for environmental monitoring network installation, configurations and operation, help ensure environmental compliance at monitoring sites, and reduce health and safety risks.

The two key goals of E-SMART are: (1) to create an openly available standard for interconnecting environmental monitoring sensors into sensor and remediation process control networks, and (2) to develop an intelligent information processing system to collect, manage, and analyze the resulting sensor data and to provide process control capabilities. E-SMART achieves the first goal through the use of microcontroller technology to standardize environmental sensor data and command protocols over a digital communications network. Any sensor or environmental monitoring equipment manufacturer will be able to implement the E-SMART environmental network standard using commercially available and cost-effective electronic components. The second goal of providing intelligent information processing power is achieved through the use of Microsoft Windows™-based network management, data management, expert system and user-friendly graphical interfaces. E-SMART integrates these technologies into a cost-effective, user-friendly system that gives the system operator complete, real time environmental monitoring and control capabilities.

A number of E-SMART-compatible sensors are now being developed. These sensors are designed to measure hydrological parameters, nonspecific hydrocarbon vapor concentrations, and specific chemical analyte concentrations in gas and liquid phase. E-SMART compatible radiation sensors have also been proposed. The development status of these sensors, preliminary at field test results, and the performance of the E-SMART system will be discussed.

ENHANCING THE SAMPLING PROCEDURE THROUGH A GEOSTATISTICAL ANALYSIS

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Field screening technologies are increasingly integrated into environmental investigative and remediation activities. Obvious advantages include rapid, cost-effective results. However, with discrete sampling points, there remains uncertainty in the estimation from field screening data to application of further on-site activities. Geostatistics is a branch of applied statistics which is particularly suited to environmental interpolation questions because of its dependence on the spatial correlation. Thus, discrete field screening sample information is transformed into a contour plot, with associated confidence intervals, used to plan future sampling activities or remediation options.

Geostatistics as a tool in the development of the sampling and analysis plan has not yet been fully explored. Clearly, the frequency and spacing of the field screening locations will affect the overall precision of the geostatistical analysis. Using a combination of synthetic and hard data, the relationship between the sample locations and the precision of the kriging estimates has been explored. This research indicates that there are some minimum spatial requirements which must be imposed upon the field screening data in order to ensure that precision criteria are met.

The geostatistical analysis enhances the information provided by the raw data alone. It provides insight into the spatial disposition of the contaminant as well as the possible intervariable relationships. This approach has been used with raw data generated from x-ray fluorescence spectrometers, Hydroponch groundwater investigations, immunoassay technology, and seismic field screening technologies.

USE OF EDGE DETECTION AND FFT FILTERING TECHNIQUES TO REJECT FLUORESCENCE IN RAMAN SPECTRA

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Although Raman spectroscopy is a useful technique to identify and quantitate species in a given matrix, it has been limited in its applicability by fluorescence. Often the lasers used in Raman studies are at wavelengths that stimulate fluorescence. As a phenomenon, fluorescence is approximately 10^6 - 10^8 times stronger than Raman scattering. There are two approaches to overcoming this problem. One is to adjust the experimental technique such that fluorescence does not occur, or is much reduced, i.e., use an excitation wavelength which does not stimulate fluorescence. The other approach is to extract the Raman signal contained within the fluorescence signal. This approach includes such techniques as shifted Raman difference spectroscopy (SERDS)¹, polarization modulation², and picosecond pulsing and gating³ which takes advantage of the disparity between vibrational and fluorescence lifetimes. However, these approaches require instrumental modifications which can be costly and complex.

Since Raman signals are very narrow and fluorescence appears as a very broad band, we began to investigate the possibility of using first derivative spectroscopy, also referred to as edge detection, and fast Fourier transform (FFT) filtering techniques to discriminate between the two. It was found that both edge detection and FFT filtering were effective in rejecting fluorescence in spectra exhibiting reduced noise levels as a result of signal averaging. With regard to edge detection, smoothing the data prior to taking the derivative greatly improves the final results. The greatest disadvantage of edge detection is that we are not accustomed to looking at Raman spectra in a derivative representation. However, with curve-fitting, the derivative curves can be converted into conventional Raman spectra. In FFT filtering, the fluorescence and Raman signals are separated in the Fourier domain, with the fluorescence occurring at low frequencies. Multiplying the Fourier domain spectrum by a high pass filter eliminates the low frequency spectral features while allowing the high frequency features through thereby rejecting fluorescence. The main advantage of edge detection and FFT filtering is that they require no modifications of the existing instrumentation. Furthermore, there are commercially available software packages which will take first derivatives of spectra and perform FFTs on a real-time basis making it possible to manipulate the spectral data as it is received.

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EXPERT AIR MONITORING SYSTEM (EXAMS): AN EXPERT SYSTEM TO USE FTIR FOR AIR MONITORING

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The goal of this research was to investigate the feasibility of developing an MS-Windows-based expert system for monitoring workplace air by FTIR spectrometry. The expert system includes both expert, algorithmic and blackboard components, as well as a well-developed user interface. The expert system enables the non-spectroscopist to correctly utilize this powerful analytical technique. In a complementary fashion, the expert system also allows the non-environmental specialist who has skills in spectroscopy to correctly interpret the environmental significance of results obtained using FTIR.

This expert system currently operates in occupational environments with known atmospheric contaminants, but with the ability to flag the presence of unanticipated compounds. Decision tracing allows the experienced user to evaluate the expert system's decisions. Quality assurance methods address the quality of the reported data.

The expert system was evaluated against previously published results and found to perform as well or better than the expert spectroscopists. Evaluation of the system using five gas mixtures of varied complexity against eight expert spectroscopists gave results equal to or better than the experts.

ASSESSMENT OF RADIATION CONTAMINATION AT AN ABANDONED TIN SMELTER FACILITY IN GALVESTON COUNTY, TEXAS

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Since 1992 Ecology and Environment, Inc. (E & E) has been conducting a site assessment under the direction of the Region 6 United States Environmental Protection Agency (US EPA) to determine the nature and extent of radiation contamination at the Tex Tin Corporation Site located in Texas City, Galveston County, Texas. The site comprises approximately 170 acres and was originally constructed by the US government as a World War II emergency tin supply plant. The facility processed tin ore from 1941 until 1989, producing Grade A tin ingots and waste products; primarily an iron-rich acidic liquid and a solid slag. The facility smelled secondary copper from 1989-91. Several hazardous waste units occur on-site, including wastewater treatment ponds, gypsum slurry ponds, a pond of acidic liquid waste, several closed acid ponds, drums of spent catalyst material, a licensed radioactive landfill containing depleted uranium, several slag piles with elevated levels of naturally occurring radioactive materials (NORM), and other slag piles of various sizes.

Elevated gamma radiation levels were identified using rapid field screening surveys with a micro-Roentgen (μ R) meter. Detailed gamma surveys were conducted at locations with radiation levels elevated above background by recording gamma radiation levels at approximately 15 foot grid intervals using a μ R meter at a height of one meter above the soil surface, and a 1x1 inch sodium iodide (NaI) detector at the soil surface. Positional coordinates of each survey point were determined using a Global Positioning System (GPS). All data were recorded on the GPS data logger. Approximately 1500 data points were collected over 10 acres. Data were analyzed using geostatistical modeling and kriging to develop highly accurate contour maps of gamma radiation levels. Radiation levels recorded during the survey ranged from less than 10 μ R/hour (background) to more than 500 μ R/hour.

Gamma spectroscopic analysis was performed using a field-portable multichannel analyzer (MCA) system connected to a 3x3 inch NaI detector. This system was used to determine gamma-emitting radionuclide composition and activity from samples collected on site. Results from the gamma survey were used to establish 30 sample locations from which 4-ounce composite samples were collected. Slag and soil material from an additional 15 sample locations were collected, dried and graded. The 4-ounce containers were filled with material from eight different particle size fractions from each location, for a total of 120 samples. MCA spectra were obtained from each sample. Radium-226 activity levels ranged from below detection limits to more than 200 picocuries per gram. Ten percent of the samples were sent to an outside radiochemistry lab for confirmation analysis. The field screening results and the laboratory results showed a close correspondence.

Radon flux rates were also measured from slag piles on the site using large area charcoal collectors (LACCs). For each of three sample dates, forty samples were collected for 24 hours from five locations. Positional coordinates of sample locations were determined using GPS. Estimated radon flux rates were less than one picocurie per square meter per second.

FIELD TESTS OF PASSIVE ALPHA DETECTORS AT THE NEVADA TEST SITE AND THE ROCKY FLATS PLANT FOR SCREENING OF RADIOLOGICALLY CONTAMINATED SOILS

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Two types of passive alpha detectors, developed for indoor radon measurements, have been field tested for applications to screening of alpha-emitting contaminants (e.g. Pu, Am, U, Th) in soils. Electret Ionization chambers (EIC's) measure air ionization due to alpha emission from the soil surface, while Alpha Track Detectors (ATD's) register alpha particles as microscopic damage pits. The detectors are inexpensive, rugged, simple to use, and respond reproducibly and linearly over two orders of magnitude of contamination levels. It was found that alpha emission rates varied widely with soil composition, so procedures were developed for establishing site-specific detector calibrations. Both types of detectors were deployed directly on the surface of soils contaminated with Pu-239/240 and Am-241 at the Nevada Test Site and the Rocky Flats Plant. Deployment times of 5 mins to 72 hours were necessary in order to reliably detect contamination levels of 2000hCi/g down to 100pCi/g. The advantages of the EIC detectors were found to be their immediate on-site readout capability, while the primary advantages of the ATD detectors were their insensitivity to beta/gamma radiations and ability to indicate the presence of hot particles. Techniques were developed to discriminate against environmental radon and background $\alpha/\beta/\gamma$ radiations. ATD's in the form of long narrow strips, deployed in stake-driven holes, were used to obtain contamination depth profiles quickly and reproducibly. Progress on the development of a field-deployable ATD etching and analysis system will be reported.

USE OF PASSIVE ALPHA DETECTORS TO SCREEN FOR URANIUM CONTAMINATION IN A FIELD AT FERNALD, OHIO

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Among techniques that are well established for making passive, time-integrated measurements of airborne alpha activity in indoor environments, two are readily adaptable to the task of measuring alpha activity in situ for cases where soil has become contaminated. One technique (Alter and Fleischer, Health Physics 40:693-702, 1981) involves exposure of small (1cm x 2cm) plastic slabs (called alpha track detectors or ATDs) to air or soil containing alpha radioactivity which induces radiation damage sites. Following exposure, chemical etching enlarges the damaged sites. Optical scanning of the etch sites allows estimation of alpha activity concentration. The other technique (Kotrappa et al., Health Physics 41:35-46, 1981) involves exposure of small chambers, called electret ionization chambers (EICs), holding electrostatically polarized Teflon® discs. Alpha particles entering the air space within the chamber induce a cloud of secondary electrons that neutralize the charge on the disc. The rate of discharge is proportional to alpha activity concentration in air or on soil surfaces. Because several isotopes of uranium give off high energy (4 - 5 MeV) alpha particles when the atoms undergo radioactive decay, it is possible to use either ATDs or EICs to measure uranium contamination in soil.

An integrated demonstration program, hosted by the Fernald Environmental Restoration Management Company, has been established for investigating technologies applicable to the characterization and remediation of soils contaminated with uranium. During the summer of 1994, the staff of the integrated demonstration program conducted an intercomparison of several characterization technologies well-suited to the task of mapping the location and amount of uranium contamination in soil. A site was selected and prepared. Over a two-month period several technologies were demonstrated. The hosts prepared a set of homogenized soil samples for calibration purposes to be used by all technologies. A set of measurement sites were laid out and all technologies were applied to the marked sites.

This paper will present the preliminary results from a field test at Fernald in 1993 as well as results obtained at the intercomparison. Results have shown:

- (1) measurement costs are low, around \$10 per measurement plus site labor for placement and retrieval of devices,
- (2) calibration coefficients are consistent among soil samples from a particular DOE site but vary among sites,
- (3) moisture condensation occurs on detectors left overnight and this interferes with the measurement process, and
- (4) there is very good agreement between the two techniques ($r^2 > 0.85$).

In addition, measurement accuracies and uncertainties will be discussed.

NON-CONVENTIONAL PASSIVE SENSORS FOR MONITORING TRITIUM ON SURFACES*

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This work targets development of two types of small passive, solid-state detectors for in-situ measurement of tritium, or other weak beta-emitting radionuclides, on surfaces. One type of detector, the exoelectron dosimeter, is "aspirin-tablet" in size and shape; the other type is an electret ionization chamber (EIC) whose dimensions resemble a "sand dollar." Preliminary testing shows that quantitative measurements are realized with exposure times of only a few tens of minutes and about one hour with the exoelectron dosimeters and EIC, respectively, (at the DOE release guideline of 5000 dpm/100cm² of fixed beta contamination). Hence it is possible to make quick screening measurements for characterizing the extent and concentration of surface tritium problems or verifying that post cleanup is satisfactory. Further development is desirable to produce a secure source of thin-layer exoelectron dosimeter that have improved physical strength. The small size of the detectors allows deployment in locations difficult to access with gas-flow proportional counters. Both types of integrating device cost only a few dollars and are reusable. Measurements can, therefore, be made in the field that are faster, cheaper, safer and in several respects, better than those possible with the baseline monitoring technology.

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FIELD SCREENING ANALYSIS VS LABORATORY ANALYSIS FOR SOIL AND GROUNDWATER, A COMPARISON

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The Hazardous Waste Remedial Actions Program is a federal facilities environmental services center operated by Martin Marietta Energy Systems, Inc., for the Department of Energy. Since its inception in 1985, HAZWRAP has conducted Remedial Investigations and environmental studies at more than 500 federal facilities, encompassing thousands of individual sites. As part of its mission, HAZWRAP also identifies, evaluates, and demonstrates new and innovative technologies and technical approaches to address environmental problems.

The goal of optimizing laboratory analysis with field screening of soil and groundwater to delineate the extent of contamination has been a standard approach of HAZWRAP-led investigations for the past six years. Field screening with analysis has provided a more thorough characterization of contamination, reduced the number of laboratory samples, reduced field iterations, and allowed for more accurate assessments of site conditions. During that time, differing sampling and analytical techniques have been used to perform field screening on projects throughout the United States.

A comprehensive comparison of field screening vs laboratory data for soil and groundwater from many of these projects is presented to determine the effectiveness of field screening. Factors such as sample handling, analytical method, soil type, geographic region, and sampling approach will be presented to identify trends and lessons learned to help verify the utility of field screening and hopefully to guide future efforts.

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COMPARISON OF FIELD SCREENING TECHNIQUES WITH FUEL-CONTAMINATED SOILS

Chris Klopp, Wisconsin DNR, and David Turfiff, EN CHEM Inc.

Field Screening is an important part of conducting cost effective and comprehensive site investigations. The use of field screening has been limited compared with its potential role at contaminated sites. Regulators limit the use of field screening (in lieu of laboratory analyses), in part, because there is little information on the accuracy and precision of field screening techniques. In addition, soil collection techniques, which are thought to have a significant effect on results, have received little attention by regulators.

The heterogeneity of the soil matrix makes it difficult to study field screening and soil collection techniques by comparing field and lab analysis of collocated samples taken in the field. Our study used a technique to homogenize soil samples developed by Paul King of P&D Environmental. This technique consists of using a mixing barrel that is rotated to equilibrate the soil. To simulate "real" soil samples, we used soils taken from actual leaking underground storage tank (LUST) sites. After the soils were equilibrated with respect to concentration, samples were taken for several field screening techniques, and for laboratory analysis. Separate studies used the same equilibration system to prepare soils for comparison of soil collection techniques.

The field screening techniques compared were:

Field GC
Jar Headspace
Lab in a Bag (LIAB)
Immunoassay

The soil collection methods compared were:

EN CORE sampler
Brass Tube
Graef Anholt Schoelmer (GAS) sampler
Soil Syringe
Spatula

All field screening analyses were performed in triplicate. Gasoline contaminated soils were laboratory analyzed using the Wisconsin Modified Gasoline Range Organics (GRO) method in series with EPA method 8020. Diesel contaminated soils were laboratory analyzed using the Wisconsin Modified Diesel Range Organics (DRRO) method.

Results that will be discussed include:

1. Accuracy and precision of the field screening techniques.
2. Strengths, weaknesses and special considerations of each screening method.
3. Recommendations for appropriate use of each field screening techniques.
4. What methods of sample collection and storage are appropriate when samples won't be analyzed immediately.

INTERCOMPARISON OF IN SITU MEASUREMENTS OF PETROLEUM HYDROCARBONS USING A CONE PENETROMETER DEPLOYED LASER-INDUCED FLUORESCENCE (LIF) SENSOR WITH CONVENTIONAL LABORATORY-BASED MEASUREMENTS

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The Tri-Service (Army, Navy, Air Force) Site Characterization and Analysis Penetrometer System (SCAPS) program conducted the first demonstration of the use of real-time chemical sensors deployed with a cone penetrometer for delineation of subsurface contaminant plumes. The spectroscopic based sensor uses an optical fiber (up to 100 meters long) to transmit 337 nanometer excitation energy from a pulsed nitrogen laser located at the surface through a sapphire window in the penetrometer probe. Fluorescence induced in aromatic hydrocarbons in the soil that contacts the window is returned to the surface over a second optical fiber where it is spectrally quantified using a photodiode array detector. Over the last several years this first generation nitrogen laser-based LIF sensor and more recently the "tunable" wavelength based LIF system have been demonstrated at many different sites. However, until recently, there has not been a well orchestrated effort to compare sensor results with conventional laboratory-based chemical measurements.

In this paper we report the results of a comprehensive field validation effort in which measurements from the cone penetrometer deployed LIF sensor are compared directly with results from conventional laboratory-based chemical analyses of samples collected using traditional methods. To minimize spatial and temporal variability associated with sampling, discrete samples were collected immediately after push measurements by overdrilling the push hole with an auger and collecting soil samples with a split spoon sampler. Secondary fluorescence measurements were made on splits of the laboratory samples to provide direct comparison with laboratory results. All discrete samples were analyzed by EPA methods 418.1, Total Recoverable Petroleum Hydrocarbons (TRPH) and EPA method 8015, Total Petroleum Hydrocarbons (TPH). At some sites samples were also analyzed for Semi-Volatile Organic Compounds (SVOC), EPA Method 8270, and/or an enhanced 8270 that quantifies 40 individual polynuclear aromatic hydrocarbons (PAHs).

Results from three sites investigated to date (an active fuel tank farm, an abandoned fuel farm and a fire fighting training facility) indicate that the in situ sensor data agrees with TPH measurements on a detect/non-detect basis 91% of the time. Fluorescence data agrees with TRPH results 78% of the time which compares favorably with the agreement (83%) between the two lab methods (TRPH and TPH). Correlation of quantitative data will also be discussed.

COMPARISON OF SOIL PRESERVATION AND ANALYSIS METHODS FOR VOC ANALYTES

David Turriff, EN CHEM Inc., and Chris Klopp, Wisconsin DNR

A number of studies have identified a problem with loss of volatiles using traditional VOC methods. Many of these studies have compared traditional techniques with methods using methanol to preserve soil samples. A question still to be answered is "How long can VOC samples be held using traditional techniques as compared with methanol preservation methods?" Decisions are made everyday based on VOC data of questionable validity. It is important that the methods and holding times for volatiles be founded on scientific data that ensures quality and representative data.

The heterogeneity of the soil matrix makes it difficult to study holding times and compare analytical techniques using analysis of collocated samples taken in the field. Our study used a technique to homogenize soil samples developed by Paul King of P&D Environmental. This technique consists of using a mixing barrel that is rotated to equilibrate the soil. To simulate "real" soil samples, we used soils taken from actual leaking underground storage tank (LUST) sites. After the soils were equilibrated with respect to concentration, samples were taken for 3 analytical techniques and several holding times.

The holding times and analytical techniques used were:

EN CORE Method

Store in EN CORE sampler, methanol preserve, store at 4 C, analyze using Wisconsin Modified Gasoline Range Organics (GRO) method in series with EPA method 8020 or Wisconsin Modified Diesel Range Organics (DRO) method. Holding times investigated were immediate preservation, 2 hours, 6 hours, 12 hours, 24 hours, 48 hours, 6 days, 10 days, and 14 days.

WDNR Method

Sample using EN CORE sampler and store in 60 ml VOC vial with headspace in the vial, methanol preserve, store at 4 C, analyze using Wisconsin Modified GRO method in series with EPA method 8020 or Wisconsin Modified DRO method. Holding times investigated were immediate preservation, 2 hours, 6 hours, 12 hours, 24 hours, 48 hours, 6 days, 10 days, and 14 days.

Traditional "EPA" Method

Sample using a spatula and store in 2 oz jar with no headspace, store at 4 C, subsample and analyze using Wisconsin Modified GRO method in series with EPA method 8020 or Wisconsin Modified DRO method. Holding times investigated were 48 hours, 6 days, 14 days.

Results that will be discussed include:

1. Performance of each analytical method.
2. Reasonable holding times for the techniques.
3. The relationship of holding time and sample storage.

PROPER USE OF STATISTICAL ANALYSIS FOR EVALUATING AND MAXIMIZING THE UTILITY OF FIELD ANALYTICAL DATA

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In the past several years field chemistry has been recognized as an integral component of site and remedial investigations. The appeal of these methods has been low cost, rapid turnaround and the ability to generate many data points. It is common for an investigation using field chemistry to produce two or three orders of magnitude more data than the traditional off-site laboratory approach for the same cost. This latter point has created an unforeseen and often unaddressed problem of examining, interpreting and manipulating a large volume of data. Statistical analysis is well-equipped to address these issues, but only if properly applied. The availability of powerful microcomputers and software packages offering statistical tools has also increased dramatically over the past few years, but unfortunately these tools do not address statistical theory concerning when or how to apply the various tests. Consequently, if statistical theory is applied at all, conclusions are often drawn based on improperly applied statistical analysis.

This paper focuses on the basic statistical tools available to investigators, as well as when and how these tools should be applied. Few sets of environmental data are normally distributed (i.e. follow a Gaussian distribution), yet statistical procedures which assume normally distributed data are arbitrarily and most often applied. This paper examines how to determine whether or not a set of data is normally distributed, if and how the data can be transformed to meet the assumptions of normality, and the consequences of using Gaussian-based statistical techniques on non-Gaussian data sets. USEPA often requires that field analytical results and be compared to off-site results. Based on the observed distribution, the appropriate statistical technique (analysis of variance [ANOVA], non-parametric statistics, etc.) can be selected. Interpretation of the various statistical tests is also discussed.

Statistical analysis should be applied during the planning stage to ensure sufficient samples will be collected to achieve a desired level of confidence, during the field work to optimize sampling locations, and after the field work to determine the reliability and comparability of the data, trends within the data, or predictive model relationships. This paper discusses several case studies of both correctly and incorrectly applied statistical analysis, and it will demonstrate how, if correctly applied, statistics can generate a tremendous cost savings to a project. For example, using statistical analysis, an empirical, site-specific model was generated illustrating the relationship between total elemental analysis in the field and TCLP laboratory analysis, thereby reducing laboratory costs by 60%.

Statistical analysis is the "missing link" to an integrated approach to site/remedial investigations using field analytical data. It is imperative that quantitative statistical methods are used to draw conclusions from field chemical data that has been generated with extensive QA/QC protocols to be as quantitative as possible. Statistical analysis can be used to extract more information from a given data set than is readily apparent. This type of analysis and interpretation can also reduce costs and inaccurate data interpretation by not having to repeat work as a result of lack, or inappropriate application of, statistical analysis.

ASSESSING THE USABILITY OF X-RAY FLUORESCENCE DATA

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Energy dispersive X-ray fluorescence (EDXRF) is an important and open essential analytical method for characterizing inorganic contaminants in soils at hazardous waste sites. Its ability to generate real-time results at low cost has made EDXRF the typical choice for obtaining field screening data. These same attributes, along with the availability of more sophisticated instrumentation, have led to an increased interest in using EDXRF to obtain high quality data for remedial investigation, remedial design and remedial action. Often, however, the evaluation of EDXRF data usability compares it with wet chemical methods and neglects critical data quality issues in risk assessment. Since EDXRF is a non-contract laboratory program (CLP) method, specific guidance for evaluating data usability is lacking. This paper examines statistical methods for evaluating EDXRF data usability based on uncertainty requirements of the baseline risk assessment and remediation process, using data from several recent soils investigations. Results indicate that evaluation of EDXRF data should emphasize detection limits, false-positives and false-negatives, along with application of the common CLP data quality criteria, and that strict reliance on the correlation between EDXRF and CLP (confirmation) data is often inappropriate. In addition, matrix effects, which are often of concern but seldom properly addressed, should be evaluated with regard to data quality objectives and inherent spatial variability of contaminants. Consideration of these and other issues is important in assessing EDXRF data quality and establishing a consistent approach for assessing data usability.

THREE-DIMENSIONAL COMPUTER VISUALIZATION OF FIELD SCREENING INFORMATION: EXAMPLES AND APPLICATIONS

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The first step in conducting environmental investigations is the development of a conceptual model of the area to be investigated, including the probable distribution of contaminants. Sampling locations are based on this conceptual model. Field screening techniques allow the investigator to confirm or revise the conceptual model as the investigation is being conducted and provide the investigator with real-time information about groundwater contamination at discrete depth intervals. This information enhances accurate well screen placement and optimization of subsequent well locations. The obvious benefits of field screening are reducing the time and cost associated with field investigations and defining the nature and extent of contamination in one field effort.

Groundwater field screening techniques also provide a profusion of information that is essential in interpreting contaminant fate and transport, selecting remedial alternatives, and designing remediation systems. To derive maximum value from the field screening information, it must be compiled in a usable tool to perform these evaluations. Because field screening techniques produce a much more complete data set than conventional techniques, the three-dimensional representations of groundwater plumes are accurate. Plume configuration relative to geologic controls can be visualized easily; plume volumes at different contaminant concentrations can be calculated; and probable source release scenarios can be evaluated.

This paper will give an overview of the screened hollow-stem auger field screening technique and its application in conducting groundwater investigations at a major National Priorities List (NPL) site. The three-dimensional visualization models of field screening information will be presented to illustrate the previously mentioned points. Future applications of the three-dimensional modeling will be examined. Continued usefulness of field screening information throughout the entire environmental cleanup process underscores the cost-effectiveness of designing investigations to incorporate the field screening approach.

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** Work performed under subcontract DE-AC05-86OR21644 with Analysis Corporation under Martin Marietta Energy Systems, Inc., contract with the U.S. Department of Energy.

LINKING 3-D GRAPHICS WITH SCREENING OF VOC CONTAMINATION IN REAL TIME

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A field investigation for characterizing groundwater contamination related to three potential source areas is planned at a Landfill at Robins Air Force Base (AFB), Georgia. The approach employs a Field Assessment Screening Technology (FAST) concept. The basics of the FAST concept applied to this investigation included:

- "push" drive technology (Geoprobe) for sampling and a conductivity logging tool for stratigraphic definition,
- field laboratory analysis of groundwater and vapors by gas chromatography, and
- electronic data transfer and computerized graphical imaging to and from the field for real-time data visualization and decision making.

Of these elements, the real-time electronic data transfer and computerized graphical imaging is the most innovative and promising. This investigation will illustrate the future potential and difficulties inherent with real-time 3-D visualization of contaminants and site stratigraphy.

The case study involves application of the Dynamic Graphics Earthvision™ software to visualize contaminants and stratigraphy as data are collected. A 3-D subsurface image was constructed at the beginning of the investigation based upon existing monitoring well and chemical data at the site. The image will then be supplemented periodically during the investigation to visualize the real-time screening results. Profiles of groundwater contamination will be developed through the on-site GC analysis of nine target volatile organic compounds (VOCs). A conductivity logging tool will be used to provide stratigraphic definition, and each location will be surveyed in real-time. The collective field data—VOC screening data, surveying coordinates, and lithologic data—will be compiled in the field, transmitted by modem to an off-site location, assembled using the Dynamic Graphics system, and retransmitted to the site for review and evaluation. The real-time visualization of results is used to guide the ongoing investigation, optimize future sampling locations and depths, and ensure that the total extent of VOC contamination is assessed before demobilization.

The entire study is scheduled to cover a 3- to 4-month period including work plan development, review, and approval, field implementation, and a summary report of findings. The link up of real-time field screening results with real-time visualization and presentation systems will save time and money during the remedial investigation process.

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ASSESSMENT OF LEAD IN SOIL, DUST AND PAINT USING A PORTABLE HIGH RESOLUTION XRF ANALYSER

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One of the most significant changes in the field of Lead Poisoning Prevention is a growing realization that testing young children for blood lead levels does not prevent children from being exposed but only hopes to minimize further damage. With nearly 300,000 young children at risk of lead poisoning, it is imperative that a new approach be taken. Pretesting of the child's environment prior to it's 6th through 36th months of life, (this is the most critical time because of the characteristic hand-to-mouth activity such children exhibit) allows the opportunity for appropriate steps to be taken to prevent poisoning. Studies increasingly show that one of the most significant lead exposure pathways is via soil ingestion. Therefore, testing for lead should encompass the total home environment, including both inside and outside testing for lead in paint, house dust, and soil.

The only viable instrument for monitoring lead in paint, soil, and house dust is the portable XRF analyser. We have tested a new XRF instrument, manufactured by HNU Systems, that has the advantages of easy portability, simple operation, complete data storage, no substrate interferences and good response to moderate levels of lead in paint, soil and house dust (300-500 ppm range).

With easy to follow procedures for calibration, sample collection and sample analysis, this instrument allows any user, with proper training, to rapidly perform an assessment of lead in a home environment. This assessment would be performed both prior to and after any remediation. Post-remediation assessment is critical since many children are inadvertently poisoned each year by well-meaning efforts to remove lead paint.

While this study focuses on lead contamination and the role of portable XRF in monitoring and protection of children, this instrument also has the capability to rapidly assess other heavy metals in environmental samples because of the high-resolution capabilities of the detector.

MULTIPHASIC USE OF AN X-MET 880 XRF TO DETECT LEAD IN SOIL AT THE IDAHO NATIONAL ENGINEERING LABORATORY

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An X-MET 880 energy dispersive x-ray fluorescence spectrometer (XRF) with dual-source probe was successfully utilized to characterize two sites of lead contaminated soil at the Idaho National Engineering Laboratory (INEL). Tasks in the sampling and analysis plan accomplished using the XRF included:

- Semi-quantitative screening to determine a) samples sent for SW-846 analysis and b) locating hot-spots for vertical coring. Semi-quantitative analysis of selected samples using SW-846 results to develop calibration curve
- Quantitative analysis of samples for risk assessment and characterization
- In-situ measurement of asphalt and cement fissures
- Analysis of metal debris removed from the sites for recycling or disposal

A 15-foot systematic grid layout was used to assess the spatial distribution of contamination. Transects laid along prominent wind directions were used to evaluate the airborne transport of contaminants. Subplots were used to estimate the variation in contaminant concentrations between sample points.

Semi-quantitative soil analyses were performed by calibrating the XRF with similar soil standards, collecting, sieving, and drying samples, and performing a direct measurement "in the bag". Semi-quantitative results were used to select samples for SW-846 analysis, which would provide the comparative data for site-specific calibration standards (SSCS). The SSCS were used to re-calibrate the XRF and provide quantitative data for use in risk assessment and characterization.

The locations of asphalt and concrete fissures were documented to characterize the distribution of contamination in hard to sample areas. Analyzing scrap metal collected from the sites allowed a determination to be made if the scrap was lead, and, if so, the purity of the lead.

The X-MET 880 was used for a wide spectrum of needs, providing data in a timely and cost effective manner. The alternative would have been to collect fewer samples and send the samples for more costly CLP analysis. Limiting the sample locations may have missed significant hot-spots due to the heterogeneous nature of each site.

PORTABLE X-RAY FLUORESCENCE SPECTROMETRY IN SUPPORT OF A SUPERFUND ACCELERATED CLEAN-UP MODEL (SACM) PROJECT - A CASE STUDY

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The U.S. Environmental Protection Agency (U.S. EPA), in an effort to accelerate the assessment and remediation of Superfund sites, has developed a fast track program called the Superfund Accelerated Clean-Up Model (SACM). The purpose of SACM is to pull together various existing programs under a single umbrella to streamline the administrative and discuss making process, thereby reducing site clean-up time and costs.

Innovative screening methods, such as field portable x-ray fluorescence spectrometry (FPXRF), were used at a SACM site called Salt Lake Valley Smelters (SLVS) to delineate the extent of lead contamination in surface soils. Over 1700 surface soil samples were collected, analyzed and depicted, using geo-statistical modelling software, during a two week period in July 1992, at the SLVS site, Salt Lake County, Utah.

An inter-agency SACM task force was assembled to assess the extent of lead contamination due to historic lead smelting activities. The SLVS SACM task force was spearheaded by Region VIII of the U.S. Environmental Protection Agency (U.S. EPA) and the Utah Department of Environmental Quality (UDEQ). Technical support was provided by the U.S. EPA Environmental Response Team (U.S. EPA/ERT). This support included sample preparation and the use of four FPXRF by field chemists and technicians from WESTON's Response Engineering and Analytical Contract (REAC) and Technical Assistance Team (TAT) projects. In addition, the U.S. EPA/ERT and REAC provided daily on-site posting and modelling of FPXRF data, which aided in directing sampling efforts for the following day.

Sampling of surface soils from 15 separate abandoned or inactive smelter sites, grouped into 8 different areas, over an area of several square miles, was performed by the U.S. EPA Region VIII TATs, the UDEQ, the U.S. EPA/ERT, and the U.S. Coast Guard Pacific Coast Strike Team.

Sample management, tracking and quality assurance and control (QA/QC) were of paramount concern. Numerous agencies were collecting and submitting samples from difference areas, at the same time. In addition, the progress of the sampling efforts, and the distribution of lead contamination needed to be assessed daily in order to properly schedule man-power and materials, as well as address public right-to-know concerns.

A unique sample code was developed which would identify a specific area location and its northing (y) and easting (x) coordinates on a systematic grid which was laid out over each of the 8 areas. Once collected and labelled, the sample could be tracked during the preparation, FPXRF analysis, data reduction, posting and modelling phases, on a daily basis. A software program was written to download the data from the FPXRFs to directly generate files that could be printed FPXRF results for immediate use, and files that could be imported into the geo-statistical modeling programs. The program not only eliminated transcription errors, but also reduced the time necessary for the daily posting and modelling of FPXRF data.

FPXRFs lend themselves to the rapid generation of on-site data. This data can be used to determine spatial distribution of metals, and to direct future site activities on a daily basis. QA/QC protocols were developed to continually ensure data integrity, and the proper use of the FPXRFs instrument.

SCREENING FOR METALS IN VARIOUS MATRICES BY PORTABLE X-RAY FLUORESCENCE SPECTROMETRY/SINGLE REFERENCE STANDARD CALIBRATION

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X-ray fluorescence (XRF) spectrometry is a non-destructive method of total metal analysis that may require very little sample preparation. For this reason, XRF has been used to identify and quantify metals in solid matrices during remedial investigations and feasibility studies (RI/FS) of suspected hazardous waste sites. Most past studies with field-portable XRF analysis have relied on matrix matched standards for either the fundamental parameter or empirical coefficients methods of instrumental calibration. The paucity of certified reference materials currently available for fundamental parameter applications and the need to prepare multiple sets of standards for empirical coefficient analyses can delay the initiation of a site investigation by several weeks when several sample matrices are encountered.

To avoid delays at the start of a site investigation, an alternative instrumental calibration method has been proposed that requires only a single reference material of with a particle size (5600 mm) and moisture content similar to those of the samples to be analyzed. This reference material is used to establish element-specific response factors and the corresponding Compton K_{α} peak intensity. The latter parameter is used for correcting matrix differences between the reference standard and the samples.

This study was conducted to evaluate the single standard/incoherent (Compton K_{α}) backscattering normalization approach for quantifying metals in certified, field- and laboratory-prepared environmental samples. The results obtained using this method of calibration satisfied the data quality objectives for field screening of soils, dust, paint chips, and incinerated sludge sample matrices. These findings, along with a description of this alternative XRF calibration method, will be presented.

HIGH-SPEED GAS CHROMATOGRAPHY AIR MONITORING INSTRUMENT FOR SITE REMEDIATION AND HAZARDOUS WASTE DISPOSAL APPLICATIONS

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Contaminated site remediation and hazardous waste disposal activities pose considerable health risks to workers as well as to communities down wind from these activities. Special clothing and other safety precautions often are required to minimize potential worker exposure. Often these safety precautions reduce the efficiency of the remediation and cleanup activities. Improved, real-time monitoring of ambient air is necessary in order to reduce these health risks and improve the efficiency of these activities. Since many of the volatile organic compounds (VOCs) pose significant risks at even very low concentrations, high sensitivity monitoring techniques are required. In addition, because of the wide range of possible contaminants, high specificity is important for the reliable assessment of potential worker and adjacent community exposure.

Gas chromatography (GC) is used frequently for the analysis of VOCs. GC is characterized by high sensitivity, excellent selectivity, good accuracy and precision and excessively long analysis times. Recently, high-speed GC instruments have been developed which use a gas cooled and electrically heated cryotrapping device for sample gas collection and introduction. The metal cold trap tube is cooled by a continuous flow of cold gas. A vacuum pump is used to pull ambient air into the trap tube. Following sample collection, the flow direction through the trap tube is reversed, and the tube is rapidly heated using a capacitive discharge power supply. This results in the generation of a very narrow sample vapor plug which is introduced into the separation column.

Design features and preliminary results will be presented for a high-speed GC instrument for ambient air monitoring. The instrument is designed for high-speed analysis with a cycle time of about ten seconds. A relatively short capillary separation column is used with unusually high carrier gas flow rates. While complete separation of all components may not be obtained on this timeframe, decision quality data is generated regarding contamination levels. Since sample vapor is cryointegrated (pre-concentrated) in the cold trap tube, detection limits (volume/volume basis) in the low ppb range are obtained for most VOCs. Lower detection limits with enhanced selectivity for aromatic compounds and for chlorinated hydrocarbon compounds can be achieved with high-speed GC by the use of photoionization detectors and electron capture detectors, respectively. However, because of the very short timeframe of these measurements, chromatographic performance may be sacrificed. These trade-offs will be discussed. The cryofocusing sample collection and inlet system described here has good tolerance for water vapor, and water saturated samples up to about 1.0 mL can be collected and introduced into the high-speed GC instrument with minimal loss in sample collection and chromatographic performance. Data comparing dry and water saturated samples will be presented.

An important advantage of the high-speed GC air monitoring instrument is higher sensitivity than can be achieved with either laser based spectroscopies or solid state sensors. By the use of selective detectors, greater specificity also can be achieved than with these other methods. These features should significantly reduce the risk of worker and adjacent community exposure to VOCs during site remediation and hazardous waste cleanup activities.

A MOBILE CO₂-LIDAR-SYSTEM FOR THE MONITORING OF HAZARDOUS TRACE GASES IN INDUSTRIAL AREAS

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A compact, tunable CO₂ lidar system for field use has been developed. This instrument will be used to monitor e.g. halogenated ethane, halogenated methane, ammonia and ozone in the vicinity of industrial areas. The measurement principle is based on the DAS-technique (Differential Absorption Spectroscopy) in the spectral range from 9-11 µm for a maximum distance of 5 km.

In a first step topographic targets are used as diffuse reflectors, that means the results of the measurements are path integrated concentrations of the detected gas. The topographic targets deliver high, reliable and stable signals which leads to a high SNR and therefore to low detection limits for the trace gases. In a next step a range resolved system is planned, which should be combined with a FTIR-interferometer as a powerful station for environmental monitoring.

The heart of the lidar is a compact dual chamber CO₂ laser, delivering two parallel laser beams. Both lasers share a common gas volume and can be triggered independently with a pulse repetition rate for each laser of up to 30 Hz (burst mode for 10 seconds: 100 Hz). Since the laser is air cooled, auxiliary devices such as circulator pumps, cooling hoses etc. are not needed. The resonator consists of the discharge gaps, two reflective gratings (135 grooves/mm) for the spectral tuning and two highly reflective output couplers (R=80%, f=10m). The length of the resonator is 0.7 m. The two laser beams are s-polarized and are combined by a special beam combining optics.

This system can be operated with a LCD-touchscreen (64 sensible fields), which is integrated in an industrial PC (VME bus). Therefore it is not necessary to use an external keyboard or a monitor to run the lidar. The results of the measurements can also be shown on the LCD-screen.

In comparison to other lidar systems this device is remarkably smaller and lighter thus resulting in significant flexibility. The compact size of the system makes the use and handling easier. It increases the mobility and leads to a shorter time of access to measurement results.

The concept of combining the CO₂ lidar system and a FTIR interferometer would unit in a useful way the analytical capability of a FTIR (complete spectral coverage from 2 µm up to about 15 µm) and the possibilities of operating a range resolving CO₂ lidar system (active sounding).

PASSIVE FT-IR FOR ATMOSPHERIC ASSESSMENT OF VOLATILE ORGANIC COMPOUNDS (VOCs)

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With the success and acceptance of active open-path FT-IR atmospheric monitoring techniques in recent years, interest has been building in the potential of passive FT-IR techniques for site characterization and regulatory applications. Although much of the instrumentation and theory involved in active and passive FT-IR atmospheric monitoring are similar, passive FT-IR analyses offer additional challenges which must be addressed if passive techniques are to be developed for routine use.

In this talk, the similarities and differences between active and passive FT-IR atmospheric monitoring will be discussed. The results of recent field investigations will be used to illustrate the additional problems associated with passive analysis as well as to provide a realistic estimate of the potential performance of passive FT-IR techniques in many applications with current instrumentation.

MOBILE MONITORING TO IDENTIFY SOURCES AND EVALUATE IMPACT: A RECEPTOR MODELING STUDY USING THE TAGA 6000E

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A receptor modeling procedure has been developed in conjunction with mobile monitoring to identify and quantify emissions from sites with several potential sources. The characterization of these sources allows for the evaluation of the impact of their emissions. The combination of mobile monitoring and modeling provides an accurate account of each source's contribution to the ambient air quality using data received during initial monitoring runs and confirms the results provided by subsequent monitoring runs.

The modeling employs the Fugitive Dust Model (FDM) because of its iterative area source algorithm and its versatility to evaluate impacts of particulate or vaporous emissions. Emissions from the potential sources were utilized in order to estimate their relative impact on the downwind receptors.

Mobile monitoring was conducted using the Sciencetm Trace Atmospheric Gas Analyzer (TAGA) 6000E, a mass spectrometer/mass spectrometer (MS/MS). The TAGA collected and determined the concentrations of selected VOCs at 2.25 second intervals during mobile monitoring at the receptors of interest. This report will focus on the detection and dispersion of vinyl chloride/1,2-dichloroethane.

The TAGA data revealed concentration distributions skewed in manners which implied that the cross sections of the plumes were taken from several angles. Each plume's cross section was then separated and compared against concentrations calculated by the model. The potential sources whose predicted concentrations did not correlate to the observed concentrations were disregarded. The emission rates of those that showed good correlation were scaled to produce receptor concentrations similar to those observed by the TAGA.

After all of the sources were identified and their emissions quantified, the impact on receptors within the neighborhood were estimated. The concentrations estimated by this procedure were then compared to those collected by the TAGA during later sampling runs. The predicted average concentration across the plume was within ± 5 percent of the plume-averaged TAGA values and the predicted peak concentrations were underestimated by 25 percent. These values fall well within acceptable values for modeling.

TAGA mobile monitoring provides an exceptional database for locating and evaluating sources because each datum is supported by an adjacent datum (2.25 seconds apart); this not only provides a quantity at a specific geographic location but allows for the determination, in a Lagrangian sense, of where that geographic position is located within the plume. The tandem use of modeling and TAGA mobile monitoring provides an excellent method for evaluating impacts from sites with numerous potential sources because it:

- removes the need for sampling at each source individually,
- provides a method for self verification, and
- does not produce unrealizable extrapolation of results beyond the fence-line.

FIELD VALIDATION OF GORE-SORBERSM SCREENING SURVEYS AT SITES CONTAINING HIGH MOLECULAR WEIGHT PETROLEUM PRODUCTS

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Soil vapor surveys which employ GORE-SORBERTM Modules were introduced at the Third Field Screening Methods Symposium. These are passive soil vapor collectors, constructed from expanded polytetrafluoroethylene, (ePTFE, similar to Teflon[®]) containers filled with materials selected to detect a broad range of volatile and semivolatile organic compounds. Modules contain replicate collectors, which are normally inserted to a three foot depth using a hole bored with a 1" diameter auger. A crew of two can install up to eighty samplers in one day. After field exposure and retrieval, soil vapor analytes are thermally desorbed and cryofocused, separated via gas chromatography and identified with mass spectroscopy. Each sample is routinely examined for two dozen target soil vapor analytes, as well as prevalent and/or specially requested non-target soil vapor analytes. Compounds detected by this method range from vinyl chloride and trichloroethylene, to diesel range alkanes and polycyclic aromatic hydrocarbons. Results are measured in micrograms per sorber, calculated from reference standards run with each survey, and are presented in report and tabular forms, accompanied by color contour maps overlaid onto site plans. Unresolved hydrocarbon envelopes detected in modules are tentatively identified via a fluids library.

Screening surveys have been demonstrated to reduce the overall cost of site investigation, by subsequently reducing the number of monitoring wells required at the site. This is achieved by using the results of the soil vapor survey to transform the role of wells from one of exploration to one of confirmation. Fewer iterations of investigation and a more rapid remediation start-up may result if a comprehensive passive soil vapor survey is conducted early in a project. Screening surveys have been successfully utilized in dry, moist, saturated, and frozen soils in the United States, Europe, and the Far East. Most screening surveys have been conducted under site conditions that severely limit conventional soil vapor detection methods, such as saturated soils, low permeability clays, and estuarine marshlands. Surveys are described which successfully detected coal tars at manufactured gas plants, petroleum products at storage terminals, and creosotes at an asphalt plant. Confirmatory soil and ground water data are provided for each case.

GORE-SORBER Screening Surveys are a Service mark of W.L. Gore & Associates, Inc. GORE-SORBER Modules are a registered trademark of W. L. Gore & Associates, Inc. Teflon is a registered trademark of E.I. du Pont de Nemours & Co., Inc.

THE USE OF FIELD SCREENING IN PCB REMEDIATION: A CASE STUDY

Akhil Desai, Sanjay Saraf, Brad Anderer and Raja Venkateswar, TRC Environmental Corporation

Field screening methods serve as a vital tool in delineating the extent of onsite contamination. Screening samples in the field provide onsite personnel sampling activities in areas requiring better site remediation, and can be applied not only for evaluating emerging remedial technologies, but also for performing remediation using established technologies.

This paper describes the use of a field gas chromatograph (GC) equipped with an electron capture detector (ECD) for remediating a PCB contaminated site. The primary remedial technology used was excavation and land disposal. The field GC/ECD was particularly appropriate for this application because the PCB contamination was non-uniform and widespread, with pockets of high concentration. The GC/ECD was instrumental in identifying these "hot spots". A wide variety of matrices had to be sampled and analyzed for field screening. These included metallic surfaces of miscellaneous equipment, structural steel, concrete, soil, sediments, general construction debris, oil, and water. Wipe samples were collected for metallic and concrete surfaces, while grab/composite samples were collected for other matrices. The detection limit for wipe samples was 0.5 ug/100 cm², for soil/sediment samples was 0.1 mg/Kg (ppm), and for water samples was 5.0 ug/L (ppb). Special sample handling and extraction procedures were employed to assure sample integrity, rapid turn around time, analytical precision and accuracy. A total of approximately 2800 samples were collected and analyzed in the field. These included an appropriate number of Quality Assurance/Quality Control (QA/QC) samples. Approximately 10% of the samples collected were submitted to an approved commercial laboratory for verification and confirmation. The agreement between the samples analyzed onsite and in the commercial laboratory is excellent. The paper presents comparative data for different matrices and evaluates the performance of the reagent used for decontaminating the concrete and metallic surfaces. Data validation methods used for field screening are also discussed.

The field GC/ECD proved to be a valuable asset in successfully completing the remediation project and should be considered as a viable tool for similar remediation projects.

COMPARISON OF MEASUREMENTS WITH PETROSENSE® PORTABLE HYDROCARBON ANALYZER (PHA-100) AND LABORATORY ANALYSIS OF BAILED SAMPLES (EPA METHOD 8015M) WHEN USED IN MONITORING SPARGING PROCESS

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The PetroSense® PHA-100 portable hydrocarbon analyzer has been used in a project to measure the concentration of hydrocarbons at an inactive UST site, and in another project to give real time information regarding sparging. The measurements made by the PHA-100 were compared to a gas chromatographic (GC) analysis performed by an independent commercial laboratory using EPA Method 8015M and EPA Method 624.

There are two popular methods for removing hydrocarbons and other contaminants from water being evaluated. One is use of pump and treatment with carbon or air stripping, and the other is use of a sparging process consisting of injecting air through the water to strip them. The sparging process consists of drilling wells into the contaminated water, injecting air into one well, and monitoring the other wells placed around the injection well for both the amounts of air flow, and measuring the contamination levels. The current process to determine the contaminant level is taking samples according to EPA Method 8015M and EPA Method 624 to obtain concentration levels, which can take from one to ten days.

The use of the analyzer in a sparging application was to provide a relative reading of the concentration levels in real time to assist in process evaluation. Measurements with the PHA-100 analyzer can be either absolute or relative. Analytical operation will give (based on a p-xylene equivalent) readings within a ± 3 ppm accuracy in water and ± 10 ppm in vapor. The typical relative screening readings are for determining if hydrocarbons are present, and whether the concentration is increasing or decreasing.

The readings confirmed that the sparging process was working. The levels of hydrocarbons measured 15 ppm and were decreasing, indicating that the hydrocarbon envelop was moving. Using the real time information, the technician was able to quickly confirm process operation.

The ability to obtain quick relative reading in direct PPM readings is extremely useful in the field. The analyzer readings were able to provide immediate values to help determine if the process was working, and help trouble shoot problems with the installation. The analyzer values will not provide the absolute values required for determining if a site has been cleaned, but by correlation with the laboratory results, will provide indications of progress. The final determination will still have to be accomplished through normal laboratory controlled analyses, but the value of being able to determine if the process is working quickly without waiting for lab analysis definitely provides real value in having such an analyzer as a standard field tool.

Based on the study of the two projects, the real time measurements should reduce the costs of laboratory analysis on larger projects.

FIBER OPTIC CHEMICAL SENSOR (FOCS™) TECHNOLOGY FOR THE DETECTION OF HYDROCARBONS IN AIR AND DISSOLVED IN WATER: PETROSENSE® PORTABLE HYDROCARBON ANALYZER (PHA 100) AND THE PETROSENSE® CMS 5000

Devinder P. Saini, Roger Hinke, and Stan Klainer, FCI Environmental, 1181 Grier Drive, Building B, Las Vegas, Nevada 89119

Due to the ever increasing pollution created by hydrocarbon storage and transport, there is a need for rapid sensing results, and low cost field instrumentation. The information acquired by the instrumentation needs to be real time, accurate and reliable. This information should be obtained in-situ for the best possible results.

The PetroSense® Portable Hydrocarbon Analyzer (PHA 100) and the PetroSense® CMS 5000 for continuous monitoring, are instruments which can detect the presence of hydrocarbons dissolved in water as well as detect hydrocarbons in the air. They allow the user to obtain real time in situ data on hydrocarbon levels present. They can also measure the progress of any remediation that is occurring.

Both the PetroSense® PHA 100 and the PetroSense® CMS 5000 use fiber optic chemical sensor (FOCS™) technology. The refractive index of a proprietary coating on the side of an optical fiber is modified by the presence of hydrocarbons. This modification results in a change of light received by a photo detector. The change in the light signal is proportional to the concentration of hydrocarbons present in water or air. The fiber is housed in a probe which also contains the electronics. The probe is rugged and water proof.

In this paper we will discuss in detail the fiber optic chemical sensor (FOCS™) technology used, its advantages and disadvantages over the existing technology, and the performance of the probes and instruments in the laboratory and the field will also be discussed.

THE ROLE OF OPTICAL CHEMICAL SENSORS IN ENVIRONMENTAL APPLICATIONS

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With the ever increasing regulations on pollution control there now exists an increasing demand for sensors to detect pollution in the environment. Manufacturers are required by the EPA to quantify their emissions of over 300 toxic chemicals that are discharged, recycled or disposed of. For the emissions to be properly quantified there is a need for instrumentation that can be used in-situ in the field to provide real time information.

Most analytical methods used today are not suitable for the field. They were primarily designed for the laboratory, and are not easily transferable to the field. There is clearly a need for new technologies to step in and fill the gap that now exists between what is required and what is achievable.

Optical sensor technology is something that could fill the gap. Some of the most sensitive analytical techniques, such as Fluorescence, Absorption and Refractive index, are based on optics. FCI Environmental has recently introduced two instruments that can measure dissolved hydrocarbons in water as well as in the air. These instruments are the PetroSense® Portable Hydrocarbon Analyzer (PHA 100) and the PetroSense® CMS 5000 for continuous monitoring of hydrocarbons. They provide real time in-situ data on the hydrocarbon levels present in water and air.

In this paper we will be discussing new sensors that will meet the needs of the industry. These will include multi parameter probes with optical sensors that can sense more than one pollutant. Sensors for heavy metals, oxygen, Carbon dioxide etc. The requirements for a good field sensor and its application in the real environment will also be discussed.

USE OF FIELD SCREENING TO DELINEATE A LOW-LEVEL GROUND-WATER PLUME OF ETHYLENE DIBROMIDE

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During routine groundwater sampling of monitoring wells downgradient of a groundwater extraction system under construction at the Massachusetts Military Reservation, trace levels of ethylene dibromide (EDB) were discovered in one monitoring well. The groundwater extraction system was designed to contain a chlorinated solvent (predominantly dichloroethylene, trichloroethylene, and tetrachloroethylene) plume. The occurrence of EDB was not anticipated and had only recently been added as a contaminant of concern for ongoing Base investigations. Because of its low state regulatory limit in groundwater (0.02 µg/L) and potential impact on the location of the extraction system, a two-phased field screening approach was undertaken to confirm the presence/absence and extent of EDB. The initial field effort consisted of sampling and analysis of existing wells to confirm the detection and determine if an upgradient source could be identified.

Approximately 30 samples were sent to a local laboratory for analysis of EDB and volatile organic compounds (VOCs). The sampling confirmed the EDB previously detected; however, other EDB analytical results were negative at a detection limit of 0.01 µg/L. With regulatory concurrence, an investigation to determine the extent of the EDB contamination in the vicinity of the extraction system and potential impact on design was undertaken. The investigation relied on screened hollow-stem auger sampling of groundwater at discrete intervals with on-site analysis of EDB and VOCs (modified EPA Methods 504 and 8010/8020). The detection limit for EDB was 0.005 µg/L. Confirmation samples (10% of total screening samples) were collected and submitted to a fixed-base laboratory (EPA Methods 504 and 8010/8020) to support the field screening data. Construction of the extraction system was delayed during the investigation. Eight deep (i.e., approximately 200 ft) screened auger borings were sampled at ten ft intervals to delineate the extent of the EDB and VOC contamination and to determine its relationship to the chlorinated solvent plume targeted for extraction.

Field screening results confirmed that EDB and VOCs were downgradient of the extraction system. Based on the screening data of compounds detected, the relative concentrations of the VOCs, and the intervals of detection, it was concluded that the EDB and VOCs represented a separate contaminant plume deeper in the aquifer than the solvent plume. These conclusions were later confirmed with Contract Laboratory Program (CLP) samples collected from new and existing wells. Based on field screening data and with regulatory concurrence, it was determined that the extraction system design would not require modification. The delay in the extraction system construction totalled 66 days with an additional cost of \$10,800. However, the use of screening data versus CLP data saved approximately 60 days. This would have severely impacted the construction schedule and resulted in significant additional costs.

CASE STUDIES ON SCREENING SOIL SAMPLES FOR TNT AND CHLORDANE AND PRACTICAL ASPECTS FOR BRINGING THESE FIELD SCREENING METHODS INTO THE LABORATORY

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The objective of this paper is to share specific experiences with screening analyses for explosives (TNT) and pesticide (chlordane) residues in soil matrices. In addition, the paper will include a discussion of the practical aspects of adapting these analytical methods for screening purposes and the QA/QC aspects of performing this work under restrictive time and cost limitations.

Over the last several years, Midwest Research Institute (MRI) has analyzed hundreds of soil samples for various regulatory-based programs. Quality control data will be presented to show method precision, matrix effects, and spiked chemical recovery that have been achieved for this work. In addition, data will also be presented for representative samples which have been reanalyzed using EPA methodologies to confirm the absence or concentration of the screened analytes.

MRI has been successful in effectively controlling operational factors which impact cost and time of analyses. In the absence of a fully functional on-site laboratory, alternatives are suggested which make it possible to provide valid analysis results within 24 hours of sample collection, including transport to the laboratory. Options and alternatives will be discussed for data validation and communication of results to help stream-line the reporting process without sacrificing data quality and documentary evidence.

This paper provides further support for the use of screening methods as a tool in environmental investigations, improving a program's effectiveness through a broader-base of sample results and more selective use of expensive EPA methods for confirmational analyses.

AIR MONITORING AND SAMPLING TECHNIQUES USED IN SUPPORT OF A CLEANUP ACTION AT AN INACTIVE DRUM RECYCLING FACILITY

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The U.S. Environmental Protection Agency Environmental Response Team (ERT) is a national and international response team utilized by the U.S. EPA, as well, as other federal, state and local agencies, whenever they encounter particularly dangerous or technically complex situations. The U.S. EPA Region VIII On-Scene Coordinator (OSC) requested the assistance of the U.S. EPA/ERT and its Response, Engineering and Analytical Contract (REAC) to perform air sampling/monitoring for volatile organic compounds (VOCs) and sulfur containing compounds during soil removal and solidification activities at an abandoned drum recycling site.

The main objective of the air monitoring and sampling during three phases of work was to provide quality data immediately or within hours to effectively direct cleanup activities and ensure the safety of the nearby workers and citizens. As the work proceeded, problem compounds with low odor thresholds were discovered, slowing the cleanup and creating more demand for field analytical techniques with sensitivity and selectivity for the target compounds of concern. An overview of each phase of site cleanup and associated air monitoring/sampling is presented. Selected equipment and techniques which provided rapid data on-site will be highlighted and the results briefly discussed.

FIELD GAS CHROMATOGRAPHY ANALYSIS AS INPUT FOR VADOSE ZONE MODELING: A CASE STUDY

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Many field programs use relatively inexpensive field screening techniques to gather a large data base. Typically, confirmation samples are selected for laboratory analysis based on these screening data, and this practice significantly increases project costs. At Williams Air Force Base (AFB) in Mesa, Arizona, jet fuel contamination in the vadose zone was characterized using field gas chromatograph (GC) analysis only. Because of the thick vadose zone at Williams AFB (210 ft), it was desirable to develop a field program that gathered enough data to adequately characterize the site for multiphase flow and transport modeling in a cost-effective manner.

Because the data would be used only as input into the modeling effort, the project team decided to maximize the amount of field screening data while eliminating the fixed-base laboratory data. The field program included drilling 15 borings to the groundwater table (210 ft), collecting split-spoon soil samples at 10-ft intervals, and analyzing all samples for total petroleum hydrocarbons (Modified 8015 JP-4 standard) and benzene, toluene, ethylbenzene, and xylenes (Method 8020) using an on-site field GC. Because of the large quantity of samples, field GC costs were approximately one-half of fixed-base laboratory costs. Additional soil samples were collected for geotechnical analyses, thus providing additional model input parameters including vertical hydraulic conductivity, grain-size analysis, porosity, and bulk density. The data were input into the two-dimensional Multiphase Flow and Transport Computer Model (MOTRANS). The model was calibrated to existing site conditions by simulating a jet fuel spill near ground surface and projecting migration for 20 years. This corresponds to the estimated amount of time since the first fuel spill. The modeled jet fuel distribution in the vadose zone closely resembles the existing distribution as determined by field GC analysis. Because the model can reproduce current conditions by simulating a historical spill, there is increased confidence in model predictions of future fuel migration. MOTRANS does predict that the fuel will continue to migrate through the vadose zone and impact groundwater for the next 25 years.

This project demonstrates the successful delineation of a thick vadose zone using cost-efficient field GC methods only for chemical analysis. By reproducing a historical spill at the site, modeling accurately simulated the subsurface distribution of fuel contamination as determined by field GC analysis of subsurface soil samples. This accurate simulation of known conditions provides confidence in the model predictions of fuel migration in the vadose zone for the next 25 years.

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A PRACTICAL APPLICATION OF HEWITT'S AQUEOUS EXTRACTION AND HEADSPACE FIELD GC SCREENING TECHNIQUE FOR VOCs IN SOIL SAMPLES

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A site for a confidential client consists of approximately nine (9) acres in the Upper Peninsula of Michigan. Since the 1930's, the site was used for the disposal of wood tar wastes from the production of charcoal briquettes and chemicals derived from wood. Following evaluation of several remedial alternatives; excavation, removal, shipment by rail and landfilling of the wood tar materials was selected as the most viable solution. The recommended approach was reviewed by Michigan Department of Natural Resources (MDNR) and ENSR was the designated engineer and construction manager for this project.

The major task of the interim response required removal of all visibly contaminated material (over 70,000 tons) while minimizing potential community impacts. Toxic Characteristic Leaching Procedure (TCLP) regulatory levels for volatile organic compounds were used as guidelines for loading the tar waste.

In order to assure that material being loaded into railroad cars met the waste characteristic requirements of the landfill to which it was being sent and to assure that it was properly manifested as non-hazardous; ENSR successfully employed a Photovac Model IOSPlus field GC with a Photoionization detector (PID) to screen large quantities of soil samples. The screening method is a modification of the EPA medium level VOC soil analysis and includes soil sample preservation and extraction into methanol with subsequent analysis of an aliquot of the extract headspace equilibrium over 30 ml of water in a 40 ml VOA vial. Total analysis time per sample was under 20 minutes. Analytical protocol will be presented with associated quality assurance/control data for samples from this site. Ten percent of the samples were sent for laboratory confirmation by EPA method SW846/8240. This comparison data will also be presented.

Use of this field screening during one of the most severe winters in the Upper Peninsula saved the client over \$200,000 in laboratory costs and enabled work to progress in a timely fashion with minimal impacts on the surrounding community.

GAS CHROMATOGRAPHIC MONITORING OF TANK HEADSPACE VAPOR SAMPLE QUALITY

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Hanford site situated in the south central part of Washington State was used for nuclear material processing beginning in the early forties. The chemicals and solvents used in the nuclear material processing generated large amount of waste. The chemical waste has been stored in large tanks (0.5 to 1.0 million gallon capacity) since then. Many of these tanks are about 50 years old. The determination of tank headspace vapor composition is extremely complex and important since this information has a potential to help resolve critical safety issues such as: hydrogen generation, flammable gases, and noxious vapors. This information on vapor phase composition of inorganic and organic compounds is essential to help develop a strategy for safe storage of waste in the tanks, for long periods of time. In view of the nature of the problems, complexity, and sensitivity associated with such tanks, characterization of the tank headspace vapors pose several challenges. Such as, little information is known about the nature of the tank waste and chemical pathways which generate noxious vapors, the potential for explosion or flammability, and employee exposure to toxic vapors and radiation. This paper presents the data on the use of a heated probe to draw tank headspace vapor sample; a heated manifold mounted on a field sampling truck for automated sample collection using a series of valves, flow controllers, pressure transducers, and thermocouples; SUMMA canisters and sorbant tubes as media for sample collection; and a gas chromatograph for checking the vapor sample integrity/amount. This presentation focusses on the gas chromatographic data in particular, and attempts to describe a mechanism to monitor the vapor sample quality, by determining the total non-methane organic compounds in the sample stream.

*Principal Author

ADAPTING THE BRUEL & KJAER MULTI-GAS MONITOR-TYPE 1302 TO MEASURE SELECTED VOLATILE ORGANIC CHEMICALS (VOCs) IN SOIL

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The Bruel & Kjaer Multi-gas Monitor-Type 1302 can simultaneously measure up to 5 different gases/vapors in a single air sample by using up to 5 different optical filters combined with a photoacoustic detection method. The analytical method has previously been validated for the measurement of VOCs in water and submitted to the DOE Methods Compendium (Method OS030). In that method, VOCs are purged with air from water samples into a Tedlar bag, and the bag's contents are sampled and measured using the Multi-gas Monitor. Currently, the analytical method is being modified for use to measure VOCs in soil. The approach being taken is to dilute the test soil samples with water and to purge the VOCs from the resulting slurry, with a flow of air, into a Tedlar bag. Subsequent analysis of the bag's contents is the same as was described for VOCs in water. Effective purging of the sample is the key to this method. For example, it is essential that the sample be constantly mixed to prevent the soil from settling which dramatically decreases the effectiveness of the purge. Various purge parameters, such as the most effective method of mixing the slurry, rate of air flow, size and shape of the purge vessel, sample size and water to soil ratio, are being investigated. Results describing the effectiveness of the method for measuring chloroform, carbon tetrachloride, trichloroethylene, tetrachloroethylene and acetone in soil will be presented.

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FIBER OPTIC RAMAN VOC VAPOR SENSOR

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Contamination of the environment by volatile organic compounds such as aromatics and chlorinated hydrocarbons is a significant problem in both surface waters, ground waters, and soils. The objective of the current work at NRL is the development of a sensitive, highly selective fiber optic Raman spectrometric sensor capable of detecting ppm levels of VOC's in both the air and water. The NRL Raman probe utilizes a polymenc adsorbent to concentrate the VOC vapor in the optical path of the Raman probe; the Raman spectrum of the VOC adsorbed into the polymer is then acquired. The probe response is fully reversible for a number of different VOC's such as benzene, toluene, TCE, and carbon tetrachloride. At high levels of vapor (mg/L) the probe can be internally referenced using either polymer or silica Raman lines as a reference. Low levels of VOC's (ppm(v)) can be detected via spectral subtraction of a reference spectrum from the VOC containing spectrum.

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DEVELOPMENT OF SELECTIVE MASS SENSORS FOR FIELD MONITORING OF ORGANIC VAPORS

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The detection of potentially harmful vapors at low concentrations is a current need in many environmental monitoring applications. Chemical sensors have promising advantages for field measurement of pollutants. These advantages include lower cost, smaller size of instrumentation, and simpler operation than traditional analytical techniques. Mass sensors have the additional advantage of high sensitivity, but they lack specificity. This limitation can be overcome with the use of recognition coatings that will selectively interact with the analytes of interest. The objective of this work is to develop recognition coatings that provide both selective and sensitive interactions with vapors (e.g., acids, amines, and hydrocarbons). These coatings can be used with quartz crystal microbalance (QCM) or surface acoustic wave (SAW) mass sensors.

Various amine polymers have been examined as candidate coatings for interaction with the analytes. Commonly, the coating process involves polymer materials dissolved in a suitable solvent and then deposited on a sensor surface. In this study an emerging coating technique was explored. Materials were deposited onto the sensor surface from vapor streams of volatile substances activated through plasma discharge. This process allows preparation and deposition of recognition coatings to be done in one step. Coating materials were characterized by measuring their reactivity with different vapor analytes using linear energy solvation relationships (LSER). These relationships were used in this study to characterize commercially available and plasma deposited coatings. An LSER equation was defined and used to predict the reactivity of the coating material toward specific vapors. The equations have predicted correctly the interaction strength for other vapors. This method allows researchers to design coating materials which selectively react with specific analytes of interest.

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USE OF ULTRASOUND IN MONITORING CHEMICAL CONTAMINATION IN WATER

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The U.S. Environmental Protection Agency has been examining the potential of combining sonication with other technologies for monitoring specific classes of organic pollutants in water. The present research specifically addresses the use of ultrasonic processors to decompose contaminants such as organochlorine compounds into products which can be detected using commercially available electrodes as a field screening method. For example, changes in chloride, conductivity, and pH were measured before and after sonication in order to detect the presence of organochlorine pollutants. The results obtained are very promising. Chloride ion could be detected in aqueous solutions of low ppm concentrations of carbon tetrachloride, chloroform, and trichloroethylene after one minute sonication. The increases of chloride ion were accompanied by increases in conductivity and decreases of pH. Ion chromatography of solutions before and after sonication showed that formate ion was also formed. Aromatic and polyaromatic chloro compounds represented by chlorobenzene and polychlorobiphenyls, respectively, did not form chloride ion as readily as did carbon tetrachloride, chloroform, and trichloroethylene. The potential of combining sonication with commercially available measurement technologies for monitoring specific pollutants in water is judged to be high. The results achieved with the organochlorine compounds tested served as proof-of-principle and formed a base of information which can be used to develop field screening methods based on sonochemistry.

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), prepared this abstract for a proposed oral presentation. It does not necessarily reflect the views of the EPA or ORD.

A FIBER OPTIC ABSORPTION SENSOR FOR THE DETECTION OF VOLATILE ORGANIC COMPOUNDS IN AQUEOUS SOLUTIONS

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Extensive contamination of ground water from organic solvents has placed a large emphasis on the development of instruments for remote in-situ sensing. Fiber optic chemical sensors (FOCSs) have made a great deal of progress in this area. A core-based intrinsic fiber optic absorption sensor has been developed in our laboratory for the detection of volatile organic compounds. The distal ends of transmitting and receiving fibers are connected by a small cylindrical section of an optically clear silicone rubber. The silicone rubber acts both as a light pipe and as a selective membrane into which the analyte molecules can diffuse. The sensor has been used to detect volatile organics (trichloroethylene, 1,1-dichloroethylene, and benzene) in both aqueous solutions and in the vapor phase or headspace. Absorption spectra obtained in the near-infrared (NIR) provide qualitative and quantitative information about the analyte. Water, which has strong broad-band absorption in the NIR, is excluded from the spectra due to the hydrophobic properties of the silicone rubber. The rate limiting step is shown to be the diffusion through the Nernstian boundary layer surrounding the sensor and not the diffusion through the silicone polymer. The rate of analyte diffusion into the sensor, as measured by the t_{90} values (the time required for the sensor to reach 90% of the equilibrium value), is 5-10 minutes for measurements in aqueous solutions and on the order of 1 minute for measurements made in the headspace. Results of the initial studies and current limits of detection will be presented.

OXYGEN AND CARBON DIOXIDE SOIL GAS SURVEY INSTRUMENT

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The work of Robbins et al.¹ has demonstrated a correlation between the amount of soil contamination at leaking underground storage tank sites, and the ratio of carbon dioxide to oxygen in the soil gas.

The focus of this project was to build a single site assessment instrument capable of accurately measuring both carbon dioxide and oxygen over an extended concentration range of 0-21%. This instrument offers the multiple benefits of simplified field measurements, reduced operator error, increased precision, and lower sample costs.

Can recent advances in carbon dioxide and oxygen sensor technology be transferred for use in underground storage tank site assessments? Historically, costly field tests would be performed to answer this question. This project has evaluated sensor performance through laboratory testing designed to parallel field conditions.

Sensor performance was measured with an automated system constructed to test the sensors as they would be used in the field. Test conditions were carefully selected in order to obtain the most information per data point. The oxygen and carbon dioxide levels were chosen to reflect gas compositions of great interest for soil gas surveying. Methane, synthetic gasoline, water vapor, and hydrogen sulfide concentrations were chosen to reflect field conditions and to determine the effects of soil gas vapors on oxygen and carbon dioxide sensor performance.

The test program characterized sensor response in terms of precision, accuracy (bias), response and recovery, specificity, and stability. The sensors were also evaluated with respect to warm-up time, power requirements, sample size, and costs. Oxygen and carbon dioxide vapor sensors used in the following applications have been tested: health and safety monitoring, automobile emissions monitoring, building ventilation control and combustion control.

Electrochemical and paramagnetic oxygen sensors, and infrared carbon dioxide sensors show the most promise for this application. They are relatively unaffected by soil gas vapors, respond in less than one minute, and require gas samples of less than one liter. Sensor precision, accuracy and drift values are better than 3% of reading at high concentrations (20% oxygen or carbon dioxide). At lower concentrations (1% oxygen or carbon dioxide) these values are sensor specific. The best sensors having precision, accuracy and drift values that are better than 10% of reading.

Sensor performance versus cost will be reviewed. This will be followed by the presentation of the performance specifications and measurements of a prototype soil gas survey instrument using a set of the most appropriate oxygen and carbon dioxide sensors.

¹Robbins, G. A., Brendan, Deyo, Binkhorst, G., "Use of Portable Oxygen and Carbon Dioxide Detectors to Screen Soil Gas for Subsurface Gasoline Contamination", U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Office of Underground Storage Tanks, Las Vegas, Nevada, CR-814542, and CR-817587, January 8, 1992.

FIBER OPTIC CHEMICAL/ENVIRONMENTAL SENSORS BASED ON ACTIVE COATINGS

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The use of optical waveguides for sensing physical and chemical parameters in chemical process industries is a rapidly growing field. Simple amplitude or intensity modulated sensors are currently used for chemical, biomedical and environmental monitoring and control.

The development of porous glass and plastic fiber optic technology, combined with photochemically active coating techniques such as dye impregnated sol-gel coating as highly stable substrate have led to a new class of high sensitivity, fiber optic chemical sensors exhibiting enhanced long term stability. In these sensors, absorption or luminescence type indicators are encapsulated into highly durable porous glass matrix which is coated on the surface of optical fibers. As a pioneer, Center for Fiber Optic Research at Rutgers is currently developing sensors based on these new technologies ranging from environmental industry to food process industry. Ground water monitoring and on-line monitoring of food flavor are examples of these newly developed sensors.

A brief review on fiber optic sensors and their applications in process control along with recent progress in this area will be given. Also some of the recent progress on fiber optic sensors by Rutgers' fiber optic sensor group including pH, ammonia gas, O₂ and DO, H₂S and other gas and liquid systems will be presented.

DOWN HOLE ULTRAVIOLET FLUORESCENT DEVICE FOR MEASURING IN SITU VOLATILE ORGANIC CONCENTRATIONS IN GROUND WATER

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We have developed a miniature fluorescence device for measuring volatile organic compound concentrations in ground water in a well. The device consists of a Hg vapor lamp, excitation filter, sample chamber, emission filter and a miniature photomultiplier tube. Filters are chosen for optimal fluorescence signals from BTEX compounds. The current size of the instrument is approximately 4-inches in diameter and 6-inches long. The instrument is lowered into a bore hole - tethered by a powerline and data communication lines. It has instantaneous output. The current design has a flow through cell which allows water to continuously pass through the cell while it is being lowered into the well. We are in the process of bench and field testing, and refining the instrument. We believe that it will be possible to build a device suitable for installation within a cone penetrometer. Our current detection limit for Benzene is about 10 ppmv. We anticipate that the lower detection limit can be substantially reduced once further refinements are made. The intended use of the instrument is for rapid assessment of VOCs in wells and for permanent installation in a well for long term monitoring.

ABOVE GROUND AND IN SITU FIELD SCREENING OF VOCs USING PORTABLE ACOUSTIC WAVE SENSOR (PAWS) SYSTEMS

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Surface acoustic wave (SAW) devices are extremely sensitive to changes in the mass or other physical properties of a thin film cast onto the device surface. For example, mass changes of tens of picograms can be detected. Using thin films to absorb the chemical species of interest, this sensitivity can be used to detect and monitor a wide range of volatile organic compounds (VOCs). Detection levels with current systems range from 0.1 to 10 ppm for typical VOCs. Using elastomeric polymers such as polyisobutylene, diffusional properties result in rapid (few seconds) and reversible responses enabling real-time monitoring.

As with most sensors, some temperature and humidity sensitivity is inherent. Temperature sensitivity is overcome by active temperature control, "on-chip" temperature measurement, and software compensation. A new and effective system for the on-line determination of the sensor response when no VOCs are present has been demonstrated. This system consists of a Nafion membrane for humidity equilibration and a three-way valve to pass ambient air (scrubbed using an adsorbent if necessary) to the sensor on demand. By incorporating this system for establishing sensor baseline, these systems can be used on-line or in situ for long term monitoring.

Two basic portable acoustic wave sensor systems have been developed. Both contain two polymer-coated SAW sensors, one acts as a reference and the other as the active sensor. Oscillator electronics drive the sensors and provide two independent outputs, a mixed frequency that tracks changes in wave velocity and a power detector voltage that tracks changes in wave attenuation. The ratio of these two signals has been shown to provide limited discrimination of isolated chemical species while the more sensitive frequency response can be used to quantify the isolated species. One system consists of a shoe-box sized module interfaced to a notebook computer. This system has been demonstrated for real-time, on-line monitoring of off-gas concentrations from soil vapor extractions. In addition, monitoring of vapor samples drawn from vadose zone wells and from cone penetrometer sampling probes has been demonstrated.

A second system consists of a down-hole probe with associated packers for isolating a selected section of a well, a cable for power and data communication to a notebook computer at the surface, and a stainless steel sampling line to compare the in situ measurements with surface analyses. Results from both systems have demonstrated quantitative analysis of isolated species with simple and easy-to-use hardware and software. Agreement with baseline instruments has been within 2% during some field demonstrations. In addition, a wide dynamic range has been demonstrated: in one test, a 10 ppm detection level was demonstrated with the same system that accurately measured 25,000 ppm from another well. Current work is focused on extending the capabilities to mixture analysis using arrays of SAW sensors and to water analysis using specially designed sampling modules.

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HAND-HELD POTENTIOMETRIC STRIPPING ANALYZER FOR FIELD SCREENING OF TOXIC METALS

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The combination of potentiometric stripping analysis (PSA) and screen printed electrodes allows one to perform groundwater heavy metal analysis in the field at low ppb levels. The instrumentation required to perform multiple metal analyses with PSA is inexpensive and user-friendly. A typical analysis can be completed in 2-3 minutes, and the selectivity and lack of oxygen interference of this technique allows one to minimize sample preparation time.

Inexpensive disposable electrodes are now commercially available. When used with the proper reagents, these electrodes are capable of detecting the following metals simultaneously: Pb, Cd, and Zn. During the next year, electrodes will be introduced which will detect all 14 EPA Priority Pollutant Metals, many of them as multianalyte sets.

A typical analysis consists of adding sample, activating the reagents, placing several drops of the solution on the disposable electrode, and pushing the "start" button on the electronics. The battery operated hand-held electronics automatically analyzes the sample and reports the result. The LOD and CV for most metals is 5 ppb and 5% respectively. No calibration in the field is necessary, since the electronics automatically compensate the displayed result with an internal standard which is included in the reagent. Preparing the instrument for the next sample involves removing the used electrode and inserting a fresh electrode into the instrument.

The results of beta testing of the instrument with several types of aqueous samples in the field will be summarized.

MULTIPLEXED FIBER-OPTIC FLUORESCENCE SENSORS FOR MEASUREMENT OF FUELS IN A MODEL AQUIFER

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A multiplexed fiber-optic sensing system has been constructed for laboratory use in a model aquifer. This system uses laser-induced fluorescence to measure fuel components in groundwater. Tunable pulsed ultraviolet light is sent sequentially into an array of single-fiber optical sensors which are inserted into wells. Preliminary results indicate that quantitative concentration measurements can be made to approximately 1 ppb for strong fluorosceners such as naphthalene, and that turbidity corrections are feasible. The two objectives of this work are: 1) to examine fluorescence interferences (e.g. turbidity, background humic fluorescence) under carefully controlled laboratory conditions to develop methods for handling interferences encountered in field measurements; and 2) develop models for pollutant transport in groundwater.

CHEMSENSOR®: RESPONSE TO GASOLINE AND DIESEL CONTAMINATED GROUNDWATER

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ChemSensor is a rugged, field-portable instrument capable of in-situ measurement of organic compounds, especially volatile organic compounds (VOCs) in water and process streams. The response to organics is reversible, requiring only that the user clean and re-zero the probe between measurements. ChemSensor gives field sampling personnel an indication of the concentration of organic contamination at a site in real time. This helps in the assessment of leaking underground storage tank (UST) sites prior to receipt of analytical lab test results and also provides a tool for monitoring the cleanup at a site over time. This paper discusses how to interpret the results obtained from ChemSensor for site assessment at both gasoline and diesel contaminated UST sites.

ChemSensor consists of a probe connected to a meter via electrical cable that enables in-situ sampling to standard depths up to 100 feet (custom lengths longer than 100 feet also available). The sensing element of the probe is a quartz optical fiber coated with a hydrophobic and organophylic chemical coating sensitive to organic compounds. ChemSensor responds to virtually all organic species dissolved in water, dependent upon the aqueous solubility and refractive index of the compound.

Samples were obtained from both gasoline and diesel fuel contaminated sites and analyzed with ChemSensor and in the laboratory using a gas chromatograph. A linear relationship between ChemSensor and the gas chromatograph data was demonstrated for total BTEX (gasoline) and total petroleum hydrocarbons (diesel fuel). A discussion of the interpretation of ChemSensor data for monitoring contamination levels and remediation processes is presented.

The test results demonstrate that ChemSensor is a useful field portable analytical tool for determining concentrations of total organic compounds in water and a timely supplement to laboratory results. Interpretation of the data from ChemSensor monitoring of a remediation site over time can give the user an indication of the progress of the clean-up without requiring excessive characterization of the response via confirmatory laboratory analysis. ChemSensor can also be used to optimize the collection of samples for laboratory analysis at a new site by indicating the level of contamination within and among the wells in real time.

NONAQUEOUS PHASE LIQUIDS: SEARCHING FOR THE NEEDLE IN THE HAYSTACK

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Subsurface contamination by NonAqueous Phase Liquids (NAPLs) is a major concern at many government and private sites because of the threat to drinking water supplies. The threat is a long-term one, arising from the limited water solubility of NAPLs. In soil and below or on top of a groundwater aquifer they can be present in small, undiluted "pools" that are depleted only very slowly by dissolution into large volumes of water. The result is widespread pollution that can continue for many years if the NAPLs are not isolated and/or removed. However, NAPLs must first be located, a task which is often difficult.

A promising approach to the problem of locating NAPLs is to utilize a cone penetrometer or other minimally invasive device equipped with sensors that can locate contaminants in situ and in real time. We have been developing and evaluating optical sensors based on Raman, fluorescence, and internal reflection spectroscopies for locating common NAPL contaminants such as gasoline or jet fuel, and chlorinated hydrocarbon solvents such as trichloroethylene, tetrachloroethylene, and carbon tetrachloride.

This paper will describe the development of the three, complementary approaches to NAPL detection, discuss the merits and disadvantages of each, and present results obtained in the field at U.S. Department of Energy sites using sensors integrated into a cone penetrometer.

THE ROLE OF CONE PENETRATION TESTING IN EXPEDITED SITE CHARACTERIZATION; A CASE HISTORY

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Expedited site characterization (ESC) focuses on utilizing noninvasive and minimally intrusive investigation techniques synergistically to efficiently and effectively characterize hazardous waste sites. A key aspect of the process is the use of rapid data collection, interpretation and visualization technologies to update the conceptual site model as the investigation proceeds. The USDOE's Ames Laboratory has recently carried out a demonstration of the expedited site characterization process at a former manufactured gas plant site. This paper focuses on the role of an innovative cone penetrometer system, Site Characterization and Analysis Penetrometer System (SCAPS) in the ESC process.

The SCAPS unit utilizes tip and sleeve resistance measurements for continuous soil stratigraphic profiling while simultaneously using either resistivity or laser induced fluorescence (LIF) to obtain further subsurface information such as pore fluid characteristics and the presence of petroleum hydrocarbon products. Moreover, soil and groundwater samples may be obtained from selected depths while a grouting plant is available to grout the penetrometer holes on completion of testing.

The geology of the site consists of surficial fill overlying clayey to sandy and gravelly alluvial and glaciofluvial deposits overlying discontinuous clayey glacial tills in turn overlying limestone and dolomite bedrock. In the light of the site geology and the fact that the main contaminant of interest is a gas manufacturing residue DNAPL containing polycyclic aromatic hydrocarbons (PAH's), the main objectives of the SCAPS program were to define the site stratigraphy, including the continuity of the various units, define the surface topography of the lower clayey till unit to assist in tracking the transport and fate of the DNAPL and to detect residual contamination in the subsurface. The SCAPS results were integrated with the results of other non- or minimally intrusive techniques such as surface geophysics and Geoprobe sampling and conductivity logging to develop the site model.

Data collected from the SCAPS investigation indicate that the three objectives defined above were effectively met. SCAPS was able to clearly indicate the contacts between the various stratigraphic units encountered over the site and in particular, provided useful information identifying the topography of the lower clayey till unit overlying the bedrock. The agreement between the SCAPS stratigraphic logs and logs from auger boreholes near some of the SCAPS locations was good. The LIF probe was effective in detecting regions of contamination in the subsurface as PAH compounds tend to have a relatively strong fluorescence response. Elevated LIF readings (above background) were encountered at various levels in the stratigraphy indicating movement of the DNAPL down through the stratigraphy. Elevated LIF readings were also recorded in the soil directly overlying the lower clayey till indicating a tendency of the DNAPL to pool on this confining layer. These results compare well with trends observed from the other investigation techniques. In summary, the SCAPS was very effective in further defining the site geological model by providing continuous stratigraphic profiles at selected locations and in providing a good indication as to the fate and transport of the DNAPL. Moreover, the availability of the data on a real time basis allowed for effective integration of the SCAPS into the ESC process.

REAL-TIME MONITORING OF VOLATILE CHLORINATED COMPOUNDS IN THE SUBSURFACE ENVIRONMENT USING A HALOSNIFF/CONE PENETROMETER SYSTEM

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The U.S. Environmental Protection Agency (EPA) has regulatory jurisdiction for the cleanup of hazardous wastes sites designated by the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), and Superfund Amendments and Reauthorization Act (SARA). According to EPA's guidance specific activities are required to characterize a site listed under CERCLA or SARA. This activity includes a preliminary site investigation followed by a comprehensive remedial site investigation. During remedial investigation site activities, various environmental samples are collected including air, water, and soils and sediments. Because of the sheer volume of samples sent to the laboratory for analysis and the high analytical cost associated to produce legally defensible analytical data, remedial investigations costs have skyrocketed. Clearly, alternative characterization technologies are needed. A new innovative technology being evaluated couples a cone penetrometer system to a chlorinated compound specific detector system (Halosniff). The cone penetrometer provides access to the subsurface environment and the Halosniff system measures the concentrations of volatile chlorinated compounds in the gas phase (soil gas) in real-time.

Testing of the cone penetrometer/Halosniff system was conducted at the U.S. Department of Energy's Hanford Site located in southeastern Washington State. The test location was adjacent to several cribs that received from 500 to 1500 tons of CCl_4 . The cone penetrometer system used consisted of a 30,900-kg vehicle pushing a 44.5 mm (OD) by 19 mm (ID) rod system equipped with a gas sampling tip capable of sampling soil gas continuously. A 4.8-mm Teflon tube connected the gas sampling tip to the gas sampling manifold of the Halosniff System. Four pushes were conducted to a maximum depth of 100 feet. Halosniff monitored the concentration of volatile chlorinated compounds, consisting of greater than 95% carbon tetrachloride and up to 5% chloroform, as a function of depth. Maximum concentrations of volatile chlorinated compounds in soil gas exceed 20,000 ppm. During overnight monitoring with Halosniff, unexpected cyclic behavior in volatile chlorinated compounds concentrations was observed. After several hours at 20,000 ppm the concentrations suddenly decreased to 2000 ppm for several more hours, followed by an increase back to 20,000 ppm. This behavior was observed by several other monitoring instruments. The following evening similar behavior was also observed.

An in-depth description of the Halosniff technology, concentration profiles of volatile chlorinated compounds as a function of depth and time, and other relevant observations will be presented.

This project was funded by the U.S. Department of Energy (DOE), Office of Technology Development under Contract DE-AC05-76R101830. Pacific Northwest Laboratory is operated for the DOE by Battelle Memorial Institute.

SITE CHARACTERIZATION USING THE CONE PENETROMETER

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An electric cone penetrometer testing (CPT) program and a groundwater CPT program were conducted at an active industrial waste treatment plant near San Diego, California, to characterize subsurface stratigraphy and groundwater contamination to depths of 160 ft below ground surface (bgs). The intent of the sampling program was (1) to characterize the subsurface geology and (2) to delineate trichloroethene and other solvent contamination within a specific site. The CPT was the preferred testing method because of its ability to produce quantifiable stratigraphic data in log form. These logs could be used to identify potential confining beds where dense nonaqueous phase liquid (DNAPL) contamination (solvents) could pool. The stratigraphic information was used to determine target depths for groundwater sampling.

Subsurface lithology was interpreted from electric CPT logs based on values for tip resistance, sleeve friction, and pore pressure. Generally, clean, coarse-grained sands have higher tip strengths, lower sleeve friction, and little excess pore pressure. Finer-grained silts and clays have lower tip strengths, higher sleeve friction, and high excess pore pressure. To check the site's interpreted stratigraphy, a CPT location was selected ~10 ft from where a 270-ft continuously cored boring was completed. Overall, the electric CPT log correlated well with the physical evidence from the boring. The ability to quantify the electric CPT log by comparing it to the cores collected from the deep borehole was important because the comparison provided defensible correlation between soil cores and the electric CPT logs. The typical subsurface stratigraphy identified by the CPT log consisted of interbedded sands and silts; significant horizons of silty clay (at 40 ft bgs) and clay (at 85 ft bgs) were identified as confining intervals where DNAPLs pooled.

Twenty-two electric CPT holes were pushed to gather the data necessary to complete the subsurface geologic interpretation and plan the groundwater sampling points. Ninety-four depth-discrete CPT groundwater samples were collected from 23 locations. Samples were submitted to a fixed-base laboratory for overnight turnaround and were analyzed for volatile organic compounds. EPA Level III data validation was performed on sample results so data could be used for a human health risk assessment. Sufficient quality assurance/quality control samples were collected to accommodate this requirement. Groundwater results demonstrated that the CPT accurately characterized the extent of DNAPL contamination in the shallow groundwater above the first and second confining intervals. The CPT samples consistently displayed highest concentrations immediately above confining layers, with the zone above the 85-ft clay exhibiting the highest detects overall (i.e., 300,000 µg/L). Below the 85-ft clay, contaminant concentrations dropped significantly to less than 1,000 µg/L.

Using the CPT provided a cost-effective means to collect stratigraphic information lacking from previous investigations. Based on the CPT logs, depth-discrete samples were targeted for zones most likely to accumulate DNAPLs. This methodology fully characterized the extent and magnitude of contamination at the site. Local and state agencies overseeing this project were unanimous in supporting the use of CPT technology and basing remedial action decisions on CPT data. Furthermore, they believed the results to be more accurate than results from conventional monitoring wells.

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REAL-TIME, CONTINUOUS MEASUREMENT OF SUBSURFACE PETROLEUM CONTAMINATION WITH THE RAPID OPTICAL SCREENING TOOL (ROST)

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The development of a tunable laser system for in-situ chemical analysis via fiber optic spectroscopy has been presented at the previous symposia in this series. At the last symposium (February 1993), we reported initial results on subsurface detection of petroleum, oil, and lubricants by laser-induced fluorescence cone penetrometer testing (LIF-CPT). Now the laser system has been commercialized as the Rapid Optical Screening Tool (ROST). A consortium of Unisys Corporation; Dakota Technologies, Inc.; North Dakota State University; and the United States Air Force, Armstrong Laboratory has been awarded a Technology Reinvestment Project award from the Advanced Research Projects Agency (ARPA) for productization of ROST.

ROST is an integrated package with many different possible applications beyond cone penetrometer testing. The laser source, detector, and control analysis software are fully integrated. An important design consideration has been making ROST compact and self-contained so that it can be conveniently transported and deployed in a wide variety of environments. The repetition rate of the wavelength tunable laser source has been upgraded to 50 pulses/second, compared to previous versions that operated at 10 pulses/second. With the higher pulse repetition rate, petroleum contamination can be continuously recorded at better than 1 cm spatial resolution while the cone penetrometer is advanced at the standard rate (2 cm/second). The time response of the digital oscilloscope, which captures the temporal profile of the pulsed fluorescence signal from the photomultiplier tube detector, has been similarly upgraded. An optional array detector for simultaneous collection of the entire fluorescence spectrum has been added.

The topics covered in this talk include: (1) a technical overview of the ROST hardware; (2) details of its mobilization and installation for LIF-CPT work; (3) the data interface between ROST and the standard CPT geotechnical measurements; (4) lessons learned during day to day operation at many different sites that pose a wide range of soil conditions and contaminant types.

PERFORMANCE CHARACTERIZATION OF THE RAPID OPTICAL SCREENING TOOL FOR LASER-INDUCED FLUORESCENCE CONE PENETROMETER TESTING

Gregory D. Gillispie and Randy W. St. Germain, *Dakota Technologies, Inc., 2301 12th St. N., Suite E, Fargo, ND 58102*

The Rapid Optical Screening Tool (ROST) is the commercial version of the tunable laser chemical analysis system developed at North Dakota State University (NDSU) and reported at previous symposia in this series. Dakota Technologies, Inc., Unisys Corporation, and NDSU are collaborating on research and development to expand ROST's capabilities with support from a Technology Reinvestment Project award.

The initial ROST application is detection of subsurface petroleum contamination by laser-induced fluorescence cone penetrometer testing (LIF-CPT). Research to answer questions about limits of detection, matrix effects, ability to distinguish different fuel contaminant types, reliability, etc. is in progress. The actual field applications of ROST, which have already begun, will answer some of these questions. The field program is backed by extensive laboratory studies, which are designed to mimic as closely as possible the cone penetrometer environment. Limits of detection are being determined for many different soil and fuel type combinations under standardized conditions. The major challenge in these studies is how to reliably spike samples, especially with volatile fuels such as gasoline and jet fuel. The same samples are used to test algorithms we have developed for classification purposes. In one study, four different fuels (JP-4, diesel #2, unleaded gasoline, diesel fuel marine) were spiked at three different concentrations (1000, 3000, and 10000 mg/kg) on three different soil matrices. The 3000 ppm samples were used as a training set. Data then were acquired in random and blind fashion for the 1000 and 10000 ppm samples. The classification accuracy with respect to fuel type was 96%.

FIELD PORTABLE PETROLEUM ANALYSIS FOR VALIDATION OF THE SITE CHARACTERIZATION AND ANALYSIS PENETROMETER SYSTEM (SCAPS) PETROLEUM, OIL AND LUBRICANT SENSOR

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A petroleum, oil and lubricant (POL) sensor for the Site Characterization and Analysis Penetrometer System (SCAPS) system has been developed by the Tri-Services to characterize the distribution of POL contamination on military sites. The sensor is based on the detection of POL contaminants using a laser induced fluorescence (LIF) system. The SCAPS LIF sensor has been shown to be a valuable tool for the rapid screening of POL contamination in the subsurface. However, many factors can effect the LIF response of a particular fuel at a particular site. These include fuel type, age of fuel spill (eg weathering), and soil type. The LIF sensor also detects fluorescence response from any naturally occurring fluorophore, including humic substances and fluorescent minerals. These factors lead to the development of an independent procedure for verification of the LIF sensor response. This paper describes a field portable total recoverable petroleum hydrocarbon (TRPH) method based on EPA Method 418.1 and its application to on site validation of the SCAPS POL sensor response at a number of contaminated sites.

DEVELOPMENT AND TESTING OF CONE PENETROMETER SENSOR PROBE FOR IN SITU DETECTION OF EXPLOSIVE CONTAMINANTS

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This paper describes the development and testing of a sensor probe for the Site Characterization and Analysis Penetrometer System (SCAPS) for the in situ detection of TNT, RDX, HMX, and other nitrogen-containing soil contaminants. Initially, laboratory studies were conducted to determine the feasibility of employing electrochemical sensors in combination with a pyrolyzer element for detecting subsurface explosives contaminants. A method for the pyrolysis of explosives contaminants in spiked soils was developed, and it was demonstrated that electrochemical sensing of the pyrolysis products was sensitive, selective, reversible, and capable of broad dynamic range. A penetrometer probe that accommodates the electrochemical sensors (including power supply and signal conditioning electronics), the pyrolyzer unit, the pneumatic components, and geophysical sensors (tip resistance and sleeve friction sensors) for mapping soil stratigraphy was designed, fabricated and field tested at contaminated sites. Laboratory data presented in this paper include system calibration curves, lower detection limits, and estimates of the pyrolyzer's zone of influence in various soil matrices. Results of recent field tests are presented, and include examples of sensor responses in contaminated and clean sites, together with corresponding laboratory analyses of field verification samples. The prototype explosives sensor probe has demonstrated potential for SCAPS field screening and monitoring applications. Continued development, testing, and validation efforts are planned for 1995.

QUANTITATIVE AND QUALITATIVE ANALYSIS OF SPECTRAL DATA OBTAINED FROM AN INTEGRATED FIBER OPTIC FLUOROMETER AND CONE PENETROMETER

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The presentation describes the use of an artificial neural network based method for providing rapid on-line analysis of fluorescence emission spectra. The method serves to generate a real time interpretation of spectral data acquired in situ by the Navy's Site Characterization and Analysis Penetrometer System (SCAPS), an integrated optical fiber fluorometer / cone penetrometer site investigation tool currently being used to delineate subsurface petroleum contamination.

Neural network pattern recognition allows for simultaneous qualitative and quantitative analysis of raw spectral data. Quantitative estimation and speciation of different fresh and weathered petroleum products is achieved even in the presence of strong spectral interferences, non-linear response curves, and uncertain matrices. The method also provides for the rejection of spectral interferences arising from natural fluorophores, thereby minimizing the possibility of reporting false positive results.

Spectral data collected both in the lab and in the field are used in the network development process to train and test the algorithm. Performance of the neural algorithm is compared to that of multivariate statistical methods.

DATA MANAGEMENT IN SUPPORT OF A MULTI-MEDIA LEAD EXPOSURE STUDY

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The possible exposure to lead contamination from various matrices was evaluated at a U.S. Environmental Protection Agency (U.S. EPA) site under the Superfund Accelerated Clean-Up Model (SACM) program. The SACM site was spearheaded by the U.S. EPA Region VIII and the Utah Department of Environmental Quality (UDEQ). Technical support was provided by the U.S. EPA Environmental Response Team (U.S. EPA/ERT) and Roy F. Weston, under their Response Engineering and Analytical Contract (REAC).

Fifteen abandoned or inactive smelter sites in Salt Lake County, Utah, were grouped into eight separate areas. Over 1700 surface soil samples were analyzed for lead (Pb) by field portable x-ray fluorescence spectrometer (FPXRF), revealing several areas of significant lead contamination. One of the areas, to be called LEAD City from now on, was selected for an in-depth multi-media Pb exposure assessment. LEAD City was selected not only for the presence of significant Pb levels, but also because it is a residential area with many families having children six years old and younger. Children under the age of six are highly susceptible to the adverse effects of Pb exposure, including neurological and developmental disorders.

The realization that a vast amount of analytical data would be generated that would have to be linked with demographics information such as: names, addresses, phone numbers, and number of children, lead to the creation of a comprehensive database program. The database program must be able to accept analytical data from numerous sources, link them with demographics information, and health-risk census, and be able to track the progress of the project. A program called LCITY_DB was created by the U.S. EPA/ERT and REAC to address the above issues.

The LCITY_DB program will generate unique sample identification codes, chain of custodys, import data, (manually or electronically), schedule health-risk census appointments, and check for completeness. A major function of the LCITY_DB program is the ability to sort and generate reports by numerous indices, thereby allowing statistical and demographic evaluation of the LEAD City project. One of the reports generated can be downloaded into the U.S. EPA's Integrated Uptake and Bio-Kinetic model (IUBK), to predict blood Pb levels in children.

The LCITY_DB was used by the U.S. EPA Region VIII Technical Assistance Team (TAT), the Utah Department of Environmental Quality (UDEQ), U.S. EPA Region VIII toxicologists, the U.S. EPA/ERT and REAC, from November 1992 to April 1993. The LCITY_DB program is easy to use, with pull-down menus and programmed "hot keys". The LCITY_DB program is a Microsoft DOS executable program which can be ran on a standard XT type computer, with no special computer training required.

Over 500 residences were investigated. At each residence, Pb in exterior soils and Pb-in-Paint (both K and L lines) on interior and exterior surfaces were determined using two different FPXRFs. Pb in tap waters and indoor dust, were determined via atomic absorption or inductively coupled plasma emission spectroscopy, on only those residences with children six and under. For each of these residences, a comprehensive health-risk census was performed. Over 7000 Pb analysis were performed on the various matrices at LEAD City. The LCITY_DB program will be demonstrated with various sorting routines and demographic reports showcased.

¹Currently with R.O.W. Sciences

NEW TECHNIQUES FOR SOIL VAPOR SAMPLING AND ANALYSIS

Byron D. Taylor, TVG Environmental, Inc.

The objective of the presentation is to discuss the research and development that has resulted in the production of a new system for soil gas, extraction, sampling and analysis.

The technology is based on the in-situ increase of soil temperature and the reduction of atmospheric pressure to facilitate the extraction and sampling of soil contaminants heretofore out of reach for soil vapor analysis. Data from numerous field trials and applications will be included in the full presentation.

The concept of inserting probes into the soil to extract and analyze evolved earth gases was reported to have been first employed for analytical applications as early as 1853. It was not until 1975 that the technique was first attempted as a means to detect and trace contaminants.

In order to expand the range of compounds suitable for soil vapor assessment, a new steam heated vapor extraction well-point has been developed. The well-point allows for the introduction of high pressure steam into a sintered, stainless steel diffusing material which provides a heat source within the soil matrix. The introduction of steam in this manner not only adds heat to the subsurface soil structure, but also ensures that drying of the soil does not occur during the vacuum extraction process. In addition, the added moisture prevents the by-passing of atmospheric gasses at the surface from infiltrating down to the vacuum extraction ports. Also, the former limitations of Henry's Law Constant and vapor pressures are reduced by this sampling and extraction process.

In addition the heated sampling tip, the system employs an insertion process that minimizes disturbance and disruption of the sub-surface soil matrix and maximizes the seal around the probe rod. These factors insure the maximum vacuum in the sampling zone and protect against the introduction of diluting, surface air into the sample prior to analysis.

The newly developed steam-heated/vacuum extraction sampling point and Steam-Probe™ system overcame many of the limitations of previous soil vapor assessment techniques. In addition to broadening the range of compounds detectable by the process, it allows for the rapid assessment of a site and the horizontal and vertical plotting of a contaminant plume. Due to the reduced demand for boring equipment and laboratory services, results can be available instantaneously and at a significantly reduced cost.

COMPARISON OF IR AND THERMAL EXTRACTION RESULTS FOR TOTAL RECOVERABLE PETROLEUM HYDROCARBON ANALYSIS

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New Total Petroleum Hydrocarbon (TPH) methodologies are evolving in response to restricted use and possible elimination of chlorinated solvents. New techniques must meet or exceed the current analytical standards for precision, accuracy, and extraction efficiency as defined by method 418.1. This paper investigates the use of thermal extraction and flame ionization detection as an alternative method to the freon extraction followed by IR analysis for soils. (method 418.1, modified).

The noted advantages of thermal extraction are extraction without solvents, rapid extraction and analysis time. With an flame ionization detector (FID) connected as a single unit to the thermal extraction device, analysis and extraction are completed as a single operation with no loss of extracted compounds due to transfer or solvent reduction. Through this form of TPH analysis a thermal chromatogram, or thermogram, is created which provides qualitative as well as quantitative information to potentially classify petroleum contaminated soils.

Both methods are compared using natural soils (sand, high organic, and silt/clay) spiked with various oils at a range of concentrations. In addition, actual environmental samples were analyzed. All samples were analyzed by both techniques in replicate and results were statistically synthesized. Results presented will include the range of compounds applicable for thermal extraction using the proposed method. Matrix effects and interferences as well as extraction effectiveness relative to solvent extraction techniques will be discussed.

FIELD APPLICATIONS OF FLOW CYTOMETRY

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Flow Cytometry (FC) is a spectroscopic technique that allows one to interrogate the individual members of a sample population for information on size, shape, and specific chemical properties. FC has been the workhorse in the clinical hematology laboratory for sorting/typing blood cell populations. It has been proposed that the technique could serve as a possible platform for monitoring the environment for biological materials.

Preliminary results in the laboratory suggest that the technique can be very powerful. One may be able to differentiate among naturally occurring aerosols, live and dead organisms, specific chemical properties, and other properties. In addition, the instrument could serve as a possible biosensor platform for immunological and DNA probe-based detection systems. Several authors have shown the use of this technology as means to monitor water for the effectiveness of treatment for biological materials.

This talk will focus on the applications of flow cytometry in the environmental area, propose some strategies for implementation, and present some of the laboratory and field data to date.

POLYCYCLIC AROMATIC HYDROCARBONS FROM U.S. AND ANTARCTIC SITES ANALYZED WITH SYNCHRONOUS FLUORESCENCE SPECTROMETRY

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The U.S. Environmental Protection Agency (EPA) continues to search for innovative field methods and instrumentation that are fast and inexpensive. EPA's Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV) has sponsored the development of an advanced prototype portable scanning spectrofluorometer. This paper describes recent field performance at an EPA Region IV site where it rapidly analyzed mixed polycyclic aromatic hydrocarbons (PAHs). In each case, soil samples from locations with suspected PAH contaminants were rapidly characterized in the field using the UV-Vis synchronous fluorescence mode of operation. Synchronous scans of the mixed PAHs provided excellent "spectral separation", showing the peaks of component PAHs in the pollutant mixture.

In addition, sediment samples from Winter Quarter Bay, McMurdo Station in Antarctica were analyzed for PAHs. Levels of detection in the laboratory with synchronous mode was about 3 parts per billion for anthracene, a typical PAH. The collaborating group at the Idaho National Engineering Laboratory, whose Antarctic field work was supported by the National Science Foundation, used conventional analytical procedures and these data were compared to the fluorescence data. Attempts were also made to identify the sources of contamination which included diesel and jet fuels, bunker C and lubricating oils.

Based on early field results, synchronous fluorescence shows great promise for use as a field screening method for PAHs. While there are some limitations in utilizing this innovative and rapid field screening technique, synchronous fluorescence offers several major advantages; these are discussed in the paper.

Note: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), prepared this abstract for a proposed oral presentation. It does not necessarily reflect the views of the EPA or ORD.

DEVELOPMENT AND TESTING OF A VOLATILE ORGANICS CONCENTRATOR FOR USE IN WATER QUALITY ANALYSIS

D. Ehntholt, I. Bodek and T. Stolki, Arthur D. Little, Inc., 15 Acorn Park, Cambridge, MA 02140

A new approach to the concentration and analysis of parts-per-billion levels of volatile organic compounds in drinking water will be described. The technology requires only small (10 ml) samples of water and no solvent use. It has been demonstrated using the compounds generally monitored by EPA Method 524.2, using both GC/FID and GC/MS analytical finishes. Test results indicate that the method has comparable detection limits for many compounds, but can analyze a wider range of compounds and in some cases has detection limits which reach a factor of five lower. It is also expected to facilitate field sampling of water sources and to be extendable to field sampling and analysis.

THE DEVELOPMENT OF A SOLVENT-FREE APPROACH FOR THE DETERMINATION OF PETROLEUM HYDROCARBON IN WATER PETROLEUM HYDROCARBONS; WATER ANALYSIS

D. Ehntholt, I. Bodek and E. Miso, Arthur D. Little, Inc., 15 Acorn Park, Cambridge, MA 02140

Efforts to develop methods for the determination of total petroleum hydrocarbon (TPH) content of environmental samples which do not require the use of toxic or hazardous organic solvents are ongoing. Alternate approaches include the use of supercritical fluid solvents such as carbon dioxide, and organic solvents other than the freons.

We have developed a new technology which includes sample collection, concentration, separation (if desired) and analysis steps necessary for the determination of petroleum hydrocarbon content of aqueous samples. No organic solvents are necessary to practice this technology, and sample handling can be automated for use in the field or for laboratory analysis. Analyses have been performed on hydrocarbon mixtures in water which contain carbon lengths up to tetradecane at individual compound concentrations in the low parts-per-million range.

The results of these analyses compare favorably with standard methods currently used for TPH analyses of water samples.

LABORATORY AND FIELD-SCALE TEST METHODOLOGY FOR RELIABLE CHARACTERIZATION OF SOLIDIFIED/STABILIZED HAZARDOUS WASTES

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Laboratory test methodologies for field testing of solidification/stabilization (S/S) systems are described. This research combines the expertise in soil/rock mechanical/flow-through testing capabilities of the Center for Earth Sciences and Engineering (CESE) at the University of Texas and the S/S chemistry capabilities of The Gill Chair of Analytical Chemistry at Lamar University to attack one of the most important shortcomings of S/S remediation technology - the prediction of long-term stability of selected S/S hazardous waste systems currently used or contemplated for use in remediation of mixed wastes at DOE and DoD sites.

The TCLP leach test creates a worst-case environment for all attempts to correlate laboratory leaching tests with field data have been unsuccessful. Leaching in S/S material is due to the movement of water or other solvents through the pore structure, and leaching behavior can differ significantly from that in the equilibrium configuration of a TCLP test. Two procedures have been used to more effectively characterize real-world behavior: 1) tests utilize flowing pore water to replicate field conditions; and 2) reactions are accelerated by adjustments in flow rate, temperature, pressure, and/or solvent chemistry. The laboratory testing systems at the CESE provide for simultaneous measurements of chemical leaching behavior; bulk properties such as strength, mechanical deformation behavior, porosity, and permeability; and physical properties such as wave velocities and electrical conductivity. Measurements of leaching behavior have been carried out for flow through 'intact' specimens and for fracture flow through specimens subjected to failure stresses in the apparatus. These procedures are used to establish: 1) procedures for accelerated testing of selected S/S systems; 2) appropriate measured properties or indicators; 3) failure criteria in addition to acid leaching behavior; 4) a standard methodology for systematic data analysis and development of hypothesis, concepts, and models; and 5) relationships between S/S formulations and durability and longevity.

Results are presented for measurements on test specimens of strontium in Portland cement, with and without added fly ash, which have been subjected to sustained flow of low-pH fluid through laboratory-scale specimens under modest pressure. The analysis of effluent pore fluid chemistry as a function of time provides direct leaching behavior under simulated disposal conditions. Data from the test are used with numerical simulations to provide an indication of correlation or a lack of correlation with current certification standards and to extrapolate short-term laboratory performance to anticipated repository time scales. In addition to the fluid chemistry analysis, analysis of physico-chemical properties on selected pre- and post-test specimens, using wet chemistry and bulk and surface characterization, are being done to determine mechanisms of binding and leaching.

INSTRUMENTATION FOR ON-SITE TRACE ORGANIC ANALYSIS OF SOIL, WATER, AND AIR

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Time-dependent information must be made available for site-assessment and remediation teams to ensure effective decision-making regarding trace organics in soil, water, and air. Automated instrumentation and methods developed over the past decade are shown to provide qualitative and quantitative data for a range of priority pollutants and toxic organics. Sample processing with thermal desorption and/or supercritical fluid extraction (SFE) are directed to target groups or chemical species for rapid (5-30 min) screening or integrated on-line chromatographic and spectral analyses. Analytical systems that focus on purge-and-trap methods for water, thermal desorption for soils, or SFE of sorbed bed air cartridges are used with integral cryotrapping/detectors in automated method sequences.

Commercial instrumentation designed for integral sample processing (thermal and nonthermal) with enrichment/trapping is shown effectively interfaced with established chromatography (GC or SFC) and versatile detectors, including flame ionization, photoionization, infrared, or mass spectral units. Pre-programmed methods for selected organic targets permit in-field use with minimal operator training to detect and to identify species over wide concentration levels (percent to part-per-trillion). Interdisciplinary teams, such as demonstrated in DOE's Expedited Site Characterization program, are thus provided with validated on-site information for needed decision-making. Illustrations will be given for data generation with compact, flexible organic analyzers that are combined with expert system and neural network software packages to demonstrate the advantages of integrated intelligent instrumentation for the environmental sciences.

FIELD TESTING OF A PORTABLE FIBER OPTIC CHEMICAL SENSOR FOR CARBON TETRACHLORIDE, TRICHLOROETHYLENE AND CHLOROFORM

James C. Wells, David A. Gobelt, Harresh Kumar, Purus, Inc., San Jose, California 95134, 2010, and Roger Jenkins, Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831-6120

Purus, Inc. has developed and is testing a field transportable PurSense™ fiber optic chemical sensor. Field evaluation of the PurSense™ unit will begin in July 1994, at Oak Ridge National Laboratory.

The configuration of the PurSense™ fiber optic chemical sensor is based upon a laboratory unit with additional features to accommodate field analysis. The system may be battery operated and consists of a flow optrode, integral VOA vial heater, reagent delivery and recovery system with removable reagent pack, fiber optic transmitter-receiver, embedded micro controller, 2 line by 20 character LCD and communication port. The sensor is contained in a carrying case measuring 46 x 30 x 20 cm. and weighs less than 10 kg. The optrode is a miniature reaction chamber through which a chemical reagent is pumped. The reagent reacts with gaseous halogenated compounds that diffuse in through a gas permeable membrane to form a colored product. The concentration of the product is detected by measuring the rate of change of the transmission of light from a 560 nm light emitting diode. The analysis time is 2.5 minutes at the detection limit of 5 µg/L using a 20 ml aqueous sample.

Measurement methodology, sensor performance and reliability will be evaluated using reagents for the semi-specific determination of ppb concentration levels of carbon tetrachloride, trichloroethylene, and chloroform in water, soil and gaseous samples. The results of this evaluation will be compared with laboratory GC results to demonstrate the viability of using this sensor as a field screening and monitoring tool.

THERMAL DESORPTION ANALYSES OF ENVIRONMENTAL SAMPLES

O.J. Vetter and Y. Ding (NETTER RESEARCH INC, COSTA MESA, CA)

Organic constituents at low concentrations in environmental samples are normally determined by using either "purge & trap" methods or various types of extraction methods followed by GC analysis. Both basic types of analysis method have numerous disadvantages. Many of these principal disadvantages are avoided by a third and novel type of "thermal desorption - trap" method.

The organic constituents of solid samples are thermally desorbed into a trap similar to that of the "purge and trap" method. While this new method offers only marginal advantages over the conventional "purge and trap" methods, this new method is far superior to any analysis method that relies on sample extractions.

We developed the new "thermal desorption - trap" method as a replacement or alternative to US-EPA methods 8020 ("purge & trap") as well as 8080 and 8100 (extraction methods). The new "thermal desorption - trap" method allows us to determine PCBs (8080) and PNAs (8100) much more reliably (better accuracy and precision) and at much lower Method Detection Limits (MDLs) than the more conventional US-EPA methods 8080 and 8100. Combining this new thermal desorption methodology with high resolution capillary gas chromatography, we can easily determine PNAs and PCBs down to lower parts per trillion levels with high accuracy and precision.

We can also determine volatile aromatics (8020 replacement) with this new method. The new method is far superior to US-EPA method 8020 if the solid sample is fairly dry. However, severe technical problems caused by a high water content in the sample has not yet been resolved.

All of these "thermal desorption - trap" methods can easily be performed either in the lab or the field.

NON-PURGEABLE VOLATILE ORGANIC COMPOUNDS RAPIDLY DETERMINED BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING DIRECT AQUEOUS INJECTION

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The ion trap mass spectrometer, a recent commercial innovation, has made possible the detection of organic compounds at the picogram level in the full-scan mode. Using μL sample injection volumes, this translates into potential parts-per-billion sensitivity for the direct injection and analysis of aqueous environmental samples. Direct aqueous injection (DAI) is rapid, simple, and sensitive. It also eliminates the need for waste-solvent disposal and is compatible with EPA pollution prevention policy. Compared with solvent extraction and purge-and-trap preparatory methods, DAI is particularly suited to analyzing aqueous samples for non-purgeable, poorly purgeable, or non-extractable, volatile organic pollutants.

A DAI method was developed for the determination of 19 non-purgeable volatile organic compounds for which no method currently exists. These polar liquids were spiked into distilled water at 1- to 100-ppm levels and analyzed in triplicate using a fused-silica capillary column interfaced to an ion trap mass spectrometer. Using internal standardization, the relative response factors and relative retention times for the 19 compounds were determined. Duplicate data was collected using on-column and split injectors. Accuracy and method detection limits (MDLs) were calculated from 10 replicate injections of 2-ppm standards. For split injection, the average relative standard deviation (%RSD) for the compounds was 19% and the average MDL was 800 ppb; for on-column injection, the respective values were 13% and 800 ppb. Agreement with EPA-established criteria for 4-bromofluorobenzene will also be shown.

Data from the EMSL-LV Analytical Sciences Division will be presented to show method limitations involving injection volume and injector conditions (injection speed, insert type, temperature, etc). Attempts to optimize method precision and peak shape will also be discussed.

*Senior Environmental Employee Program Enrollee hosted by the National Association for Hispanic Elderly.

DESIGN CONCEPTS AND PERFORMANCE CRITERIA IN THE DEVELOPMENT OF A COMPACT, PERSON-PORTABLE GC/MS SYSTEM

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Lawrence Livermore National Laboratory and Viking Instruments Corporation have teamed together to develop an advanced, very compact, person-portable GC/MS system for dual-use field analysis applications, including rapid on-site screening for environmental contaminants. This research and development project is sponsored by the U.S. Department of Energy, Program NN-20.

This compact GC/MS system incorporates a temperature-programmable, rapid-elution, low-thermal-mass gas chromatograph with back-pressure regulation, split/splitless capability, and sorbent cartridge concentrator/thermal desorber; a hybrid ion/sorption vacuum pump developed by Lawrence Livermore Laboratory; and the Hewlett-Packard model 5972 monolithic quadrupole mass analyzer. The analyzer instrument weighs less than 44 pounds, and is packaged in a fabric carrying enclosure the size of a brief case (19 x 8.5 x 15 inches). A compact external laptop computer provides all software and signals for instrument control and data analysis.

This paper reviews the design concepts and performance criteria used in developing this new instrument. Benchmark performance data and parameters are presented and discussed. Target environmental applications include emergency response analysis, soil gas screening, ambient air quality monitoring, and hazardous waste site characterization.

**MODERN THIN-LAYER CHROMATOGRAPHY FOR FIELD-SCREENING:
DETERMINATION OF PHENOLS**

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Thin-layer chromatography (TLC) has been relatively ignored in the U.S. for environmental analysis. Most published work using TLC for environmental samples has involved pesticides. The U.S. EPA Environmental Monitoring Systems Laboratory, Las Vegas, maintains a continuous interest in various separation technologies as part of its methods research program. A number of intrinsic advantages would be associated with field-screening methods based on TLC. Among these advantages is the liquid chromatographic nature of the separation; gas chromatography, for example, depends on the volatility of the analytes and coextractives. Other advantages of TLC include its low cost, simplicity and capability of analyzing 20 to 40 sample extracts simultaneously. Examples are given of the separation of selected phenols by TLC on silica gel plates and their determination using ultraviolet and fluorescence detection.

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), prepared this abstract for a proposed oral presentation. It does not necessarily reflect the views of the EPA or ORD.

ON-SITE ANALYSIS OF LANDFILL GAS AND LANDFILL GAS FIRED BOILER EXHAUST

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Currently samples of landfill gas and landfill gas fired boiler exhaust are analyzed by a time-consuming process of field sampling, sample transportation, and storage, followed by multiple analyses on several different instruments; except for expedited samples this can take more than one day. Samples of landfill gas are analyzed for: BTX by GC/PID, chlorinated compounds by GC/ECD and sulfur compounds by GC/SCD. Use of GC/MS allows simultaneous analysis of all three classes of compounds with equivalent sensitivity. "On-Site" analysis by GC/MS could potentially save many hours of laboratory analysis time while providing additional spectral identity confirmation.

We used a Viking Spectra Trak 620 field-mobile GC/MS to analyze landfill gas and landfill gas fired boiler exhaust on-site at Rio Hondo College with landfill gas from Puente Hills Landfill. We also took samples of the landfill-gas and boiler exhaust for later laboratory GC/PID and laboratory GC/MS analysis at the SCAQMD lab. Comparing our laboratory analyses to on-site GC/MS analysis shows that the field-mobile GC/MS is sufficiently sensitive for landfill gas and boiler exhaust samples.

Analysis of the landfill fuel gas and the boiler exhaust occurred simultaneously with sampling into Tedlar bags. On-site analysis took less time than the taking of samples for later laboratory analysis. The field-mobile GC/MS detected not only BTX, but chlorinated and sulfur containing compounds as well. Several other compounds not routinely analyzed for (limonene, etc.) were also detected by the field-mobile GC/MS.

FIELD EVALUATION OF A PROTOTYPE MAN-PORTABLE GC/MS

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In recent years a man-portable gas chromatography/mass spectrometry (GC/MS) system has been developed based on a Hewlett Packard 5971 MSD and a unique automated vapor sampling transfer line GC system for direct sampling of ambient chemical vapors [1]. The vacuum system and power supplies were replaced to facilitate operation on 24 vdc batteries for up to 4 hours after startup on a transportable docking station. The gas chromatography was performed on a short capillary column under isothermal conditions in a small oven to minimize power usage. Repetitive samples were taken at ~70 s intervals using an automated vapor sampling inlet [2].

In initial testing, the prototype system has been used for monitoring of gasoline vapors. Ambient levels of 6.0 ppm benzene, 4.1 ppm toluene, 0.22 ppm ethylbenzene, 1.1 ppm m- and p-xylene and 0.25 ppm o-xylene were measured near a busy gas station [3]. The gradient mapping or source tracking capabilities of the backpack mounted system have also been demonstrated in preliminary tests with a simulated gasoline leak.

This paper will describe recent work to further evaluate the capabilities and limitations of the prototype system. In tests planned for summer 1994, the instrument will be used to monitor emissions from one or more industrial sites. Results will be described in terms of the practical utility of portable GC/MS for identification and quantitation of unknown vapors.

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VOLATILE ANALYSIS USING MOBILE GC/MS

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Clearly the numerous advantages of field screening methods has increased the interest with those in development of mobile technology and those in the field using these methods. In the past, the choice was clear, if you needed GC/MS analyses, samples were sent to service laboratories. However, advancements made in mobile quadrupole technology now offers those in the field an alternative - real-time GC/MS results on site where the information is needed and can be used for immediate response. The idea of this study is to show that GC/MS in the field can be used for full data analysis and not limited to just field screening.

Analytical challenges in the "field" setting add to the already imposing difficulties in contamination analysis. Using techniques and methods similar to those already developed for a laboratory setting, in the past, seemed impossible. Due to these advances in mobile GC/MS technologies this is not only possible but practical, cost effective, time efficient and easily implemented.

This study includes the analysis of volatile organic compounds using the mobile quadrupole GC/MS. Data illustrates a more complete analysis of volatile compounds rather than a screening of just BTX which, in the past, was all that was typically analyzed on site. The volatile analyses include compounds ranging from chloromethane to 1,1,2,2-tetrachloroethane. Analysis and data evaluation were based on the volatile EPA methodologies, including tuning criteria, calibration and quantitation.

Samples were purged using a simplified purge and trap device and desorbed on to a DB624™ capillary column. The mass spectrometer was tuned and evaluated according to EPA specifications using Bromofluorobenzene. Full scan measurements were used for all analytical runs. Results from the calibration curve and matrix samples will also be illustrated.

FIELD-PROVEN TRANSPORTABLE GC/MS: REAL-WORLD CASE STUDIES AND SUCCESS STORIES

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Gas chromatography coupled with mass spectrometry (GC/MS) has evolved into being the preferred technique for analyzing challenging environmental samples in complex matrices. In this paper we present case studies and success stories in applying a field-proven, fully transportable GC/MS system that is conveniently deployed to perform on-site GC/MS analysis, with results equivalent to benchtop laboratory systems. This on-site GC/MS analytical capability provides real-time, decision-quality data, while reducing sample handling delays, minimizing laboratory analysis costs, and eliminating sample degradation, so analytical results accurately represent actual on-site conditions.

For environmental analysis, this on-site GC/MS capability performs detailed analyses of complex sample matrices, with rigorous decision-quality QA/QC levels. This enables high-volume, accurate, comprehensive analysis of soil, soil gas, air, stack emission, and water samples. Compared to the customary practice of collecting samples and then sending these samples off-site to a laboratory, this transportable on-site GC/MS capability saves time, improves accuracy, and eliminates costly laboratory analysis fees. Site characterization is accelerated, environmental remediation is streamlined, costly delays and remobilization are avoided.

Several real-world case studies are presented, including soil contamination characterization, ground water analysis and plume delineation, emergency response environmental analysis, and air toxics analysis.

A NEW FIELD TEST KIT FOR THE RAPID DETERMINATION OF DIESEL FUEL IN SOIL

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A new test kit is available for evaluating diesel and other fuel contamination in soils. While the Freon extraction/infrared method estimates fuel concentration by measuring aliphatic C-H stretch, approaches designed to replace this such as the immunoassay method and the method involving a Friedel-Crafts alkylation reaction measure the aromatic component of fuel mixtures. Both of these methods measure fuel aromatic content indirectly with visible color development. The new method measures the aromatic component directly with a 254 nm portable field photometer. The method involves a 3-minute extraction of soil with a 10:1 ratio of isopropyl alcohol. Prior to extraction, the soil is treated with an agent to minimize the extraction of humic materials which can interfere. The extract solution is passed through a syringe filter and the absorbance is read at 254 nm. The fuel concentration is calculated based on average relative response factors. Alternatively, the actual contaminant fuel can be used as a standard if it is known. The quantitation limit is about 75 mg/kg diesel in soil. The method has been tested with a variety of soils and fuel types.

USE OF FIELD SCREENING TEST KITS TO DELINEATE HYDROCARBON CONTAMINATION

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Field screening test kits were recently used to delineate hydrocarbon contamination in soils along a 3000-ft-long abandoned fuel line and distribution system. The purpose of using the kits was to minimize characterization expenses while providing reliable and cost-effective information. More than 200 tests were run on soils at depths from surface to 12 ft below the land surface. The total petroleum hydrocarbon (TPH) test used immunoassay technology to detect TPH values below 10 ppm. Fixed-base laboratory analyses were performed on 10% of the samples collected for field screening. A review of the screening results and the fixed-based laboratory results show that the field tests were comparable.

As a result of this screening effort, future site characterization efforts will be able to focus on specific areas of soil contamination. The test kits selected and used were the Hach TPH in Soil Kits that use the Environmental Product, Inc., PETRO RISC® Soil Test. The PETRO RISC® Soil Test has been approved for inclusion in the third update of Test Methods for Solid Waste, SW-846, under EPA Draft Method 4030. Using this technology eliminated the delay in waiting for laboratory results and the higher costs associated with gas chromatography. Immunoassay kits are an important tool in field screening efforts, and they save time and monies. By using a \$150/sample for EPA Method 8020 (BTX), a savings of more than \$20,000 was realized. The time savings was realized by not having any downtime while waiting for laboratory results.

*The authors are assigned to provide hydrogeological support to the Hazardous Waste Remedial Actions Program, Martin Marietta Energy Systems, Inc.

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FIELD SCREENING METHOD FOR PICRIC ACID/AMMONIUM PICRATE IN SOIL AND WATER

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Ammonium picrate (ammonium 2,4,6-trinitrophenoxide) was the principal explosive used in a number of armor-piercing shells, bombs and rocket warheads from the early 1900s until after World War II. While it is no longer manufactured, wastes containing this component are still encountered at military installations. In addition, approximately 8% of the demilitarization inventory contains ammonium picrate. Picric acid (2,4,6-trinitrophenol) is a common industrial chemical with uses that include dyeing fabric and leather, glass coloration and metal etching. Since the pK_a of picric acid is 0.8, both picric acid and ammonium picrate occur as the dissociated picrate ion in the environment. Because of its high water solubility (about 10 g/L), picrate appears to be quite mobile in the soil profile. The objective of the work described here was to develop simple field-screening methods for ammonium picrate/picric acid in soil and water.

For soil a 20-g sample is extracted by manual shaking with 100 mL of acetone in a like manner to the colorimetric screening methods for TNT and RDX. A 20-mL filtered aliquot is mixed with 20 mL of deionized water and passed through a 3-mL SPE-ALUMINA-A (Supelco) cartridge. Picrate is retained on the alumina. Interferences are removed by passing through a 5-mL aliquot of methanol followed by a 2 to 3-mL aliquot of acetone. Picrate is eluted from the cartridge with 5 mL of acetone which has been acidified with 2 drops of concentrated sulfuric acid. The eluent is added to an additional 10 mL of unacidified acetone and the resulting solution should be colorless. The solution is then diluted with 5 mL of deionized water and a change from colorless to yellow is indicative of the presence of picrate at a concentration above 3 µg/g. The picrate concentration can be estimated from the absorbance of the solution at 400 nm with a Method Detection Limit of 0.9 µg/g. The field screening method was tested using several field-contaminated soils and the results compared favorably to the laboratory HPLC method.

For water, a 2-L sample is passed through a 47-mm Empore Anion SPE Membrane (3M Corp.) which retains picrate ion. A 10 mL rinse with methanol removes interferences. If the membrane remains white, there is less than 2 µg/L of picrate. If the membrane turns yellow, it is rinsed with 10 mL of acetone and one drop of a color-forming reagent (EnSys Inc. -TNT reagent) placed on the surface. A pink Meisenheimer anion confirms the presence of picrate. If the membrane retains brown humic materials, the color reagent produces a "rust" colored spot.

QUANTITATIVE FIELD MEASUREMENT OF TRINITROTOLUENE IN SOIL

Doug Peery, Daniel Brown, and Cora Huston, IT Corporation, Knoxville, Tennessee

The U.S. Army Corps of Engineers Cold Regions Research and Engineering Laboratory (CRREL), under the direction of Dr. Thomas F. Jenkins, developed a colorimetric method for the rapid determination of 2,4,6-trinitrotoluene (TNT) in soil. The test involves extracting soils with acetone and initiating a reaction where nitroaromatic compounds in acetone form characteristically-colored solutions in the presence of strong base. This test was used in the field at two former TNT production facilities that were undergoing remedial investigations. Much of the available information on the historical use of this test indicates that it generally produces qualitative data. Matrix interference problems were overcome on these projects to produce data that were quantitative and correlated well with data from a commercial analytical laboratory. The test data were used to direct the efforts of the surface sampling teams and drilling crews in selecting samples to define the levels and extent of TNT contamination at the sites. These data were also used to ensure personnel safety and compliance with Department of Transportation (DOT) shipping regulations for selected samples containing TNT that were sent off site for commercial laboratory analysis. The application of this test in the field to direct project activities and to control project costs will be discussed in this paper. The comparability of the field data with Level III analytical laboratory data will also be discussed.

THE COLORIMETRIC FIELD DETERMINATION OF 2,4-DINITROTOLUENE IN SOIL

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The U.S. Army Corps of Engineers Cold Regions Research and Engineering Laboratory (CRREL), under the direction of Dr. Thomas F. Jenkins, has developed a colorimetric method for the rapid determination of 2,4-dinitrotoluene (2,4-DNT) in soil. This analytical procedure is designed primarily to yield qualitative data in soil matrices based on the intensity of the blue/violet color that develops when indigenous 2,4-DNT chemically reacts with the test reagents. Results from this test method were used to direct field sampling activities during a remedial investigation of an Army Ammunition Plant in Tennessee. During the course of the project, problems were encountered with the method that resulted in non-reproducible data. The test procedures were modified to improve performance of the test on the soil matrices encountered at the site. The modified method produced semi-quantitative test results that were used to select samples for more extensive analytical characterization at a full service off-site laboratory. Consequently, the ability to "screen" samples in the field resulted in reduced off-site laboratory analytical costs. Further, the investigation team was able to gather the data needed to support cleanup feasibility studies and risk assessments at the site using a single field mobilization. This paper addresses the refinements made to the existing field test method for 2,4-DNT and the application of this method to aid in controlling project costs.

PHOTOACTIVATED LUMINESCENCE SPOT TEST FOR RAPID SCREENING OF POLYCHLORINATED BIPHENYLS

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The U.S. Department of Energy and the U.S. Environmental Protection Agency have a strong need for screening capabilities for hazardous materials. Especially, there is a critical demand to have a rapid and simple technique for screening polychlorinated biphenyls (PCBs) under field conditions. The use of field screening analysis allows rapid decisions in a cleanup operation and reduces the need for either return visits to a site by a cleanup crew or extensive and costly laboratory analyses of samples that contain no detectable levels of PCBs. Field screening techniques also reduce the cost of remedial actions by preventing unnecessary excavation of uncontaminated soil.

This paper describes a new method based on enhanced photoactivated luminescence (EPL) for rapid detection of polychlorinated biphenyls (PCBs). The EPL method described here is a unique procedure combining several processes including (i) photoactivation by UV irradiation, (ii) excitation of the photoproduct complex, and (iii) fluorescence detection of the product. The EPL procedure is simple and rapid and can be performed as a spot test method under field conditions. The methodology for determination of total chlorine content of complex PCB mixtures is described.

FIELD DETERMINATION OF VOCs AT HAZARDOUS WASTE SITES

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As the EPA increases its emphasis on actual remediation, there is a growing need for accurate and reliable air-monitoring equipment that can perform on-site to provide air analyses for volatile organic compounds (VOCs). Monitoring requirements are implemented through the Resource Conservation and Recovery Act (RCRA) and the Superfund Amendments and Reauthorization Act (SARA). It is often the case that the monitoring requirements are even more strict because of public pressure from surrounding communities.

Automated gas chromatography provides a reliable means for on-site air monitoring for VOCs. With careful selection of the GC column, column heating parameters, and the GC detector, an automatic GC can be used to monitor for the presence of the target VOCs. This technology has been used at two National Priority List (NPL) sites and in San Juan, Puerto Rico under the Superfund Innovative Technology Evaluation (SITE) program. Details of the monitoring conditions, concentration data, and monitoring system will be discussed.

FIELD SCREENING OF VOLATILE AND SEMI-VOLATILE ORGANIC POLLUTANTS USING A HEATED, PORTABLE MICRO GAS CHROMATOGRAPH

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A heated, portable silicon micromachined gas chromatograph was designed, developed and recently utilized in the characterization of volatile and some semi-volatile organic compound distribution in soil at a site in the Midwest. The use of this system in the field allowed for the rapid, on-site characterization and field screening of gasoline range VOCs and semi-volatiles (e.g., benzene, toluene, ethylbenzene, and xylene; and naphthalene) and chlorinated hydrocarbon solvents in soil and assisted in the geological mapping of environmental "hot spots". The sample inlet and introduction system of the micro GC is heatable to 110°C to further extend the application range of the micro GC to samples with high moisture content, high concentration of condensable vapors, and semi-volatile compounds. Data will be presented on the use of the heated system in the characterization of the site with details provided regarding the advantages of individual compound characterization, sampling for screening purposes, and environmental screening in the field (to locate "hot spots") and in mobile laboratories (to prevent overloading of GC/MS systems).

FIELD VOC ANALYSIS FOR WATER AND SOIL, DEMONSTRATING LOW PPB DETECTION LIMITS BY EMPIRICAL CALIBRATION

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The use of a field gas chromatograph (GC) to determine the concentration of BTEX and halogenated compounds in groundwater is demonstrated, and compared to laboratory analysis. Innovative empirical calibration in the sample matrix, increases the reliability and accuracy of the headspace analysis with a field GC. Close correlation of results with fixed laboratory analysis demonstrate the accuracy of this technique for field screening. Thermal augmentation of the sample is used to increase the headspace concentration and reduce the detection limits to the 1-10 ppb range for water samples.

Methodology for the use of these methods for soil is presented, as will it be performed in cooperation with the R. S. Kerr Environmental Research Laboratory, later this year. The use of sonication and bag headspace extraction amend the accuracy of the method for soil analysis. In addition, the performance of these analysis in a time critical, production oriented, site investigation with excellent results, is well documented. The briefcase size, field GC and auxiliary equipment is capable of providing accurate screening at EPA Quality Level II+ in real time, at a very low cost. This method allows the acceleration of site investigation and well/boring placement, with demonstrated quality.

VOLATILE ORGANIC HYDROCARBON ANALYSIS IN GROUND WATER WITH AN IN SITU SPARGE SAMPLER AND FIELD GAS CHROMATOGRAPH

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A device has been designed and tested which enables a ground water sample to be sparged within the well and purged of volatile organic compounds (patent pending). The purge gas is subsequently collected in an absorbent trap for conventional purge and trap gas chromatograph analysis. This method is applicable as both a rapid screening technique when used in conjunction with an onsite gas chromatograph, as a sample collection procedure where an absorbent trap is transported to a lab rather than a water sample and as part of a long term monitoring system for unattended ground water analysis. This method was tested by comparing on-site gas chromatograph analysis of the sparge gas from the in situ sparger to conventional purge and trap analysis of a ground water sample taken with a standard bailer. No water sample is required to be collected eliminating hazardous waste generation. Analysis can be performed at discrete depths within the well. There is no sample storage or preparation involved, removing several sources of error. Since the objective of the procedure is to liberate volatiles from the water sample, the problems with pumps and handling procedures that cause poor volatile retention in water samples are eliminated. We are currently in the process of developing a commercial product based on this concept.

EVALUATION OF IMMUNOASSAY-BASED FIELD TEST KITS FOR THE DETECTION OF PETROLEUM FUEL HYDROCARBONS IN SOIL

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The objectives of this project are to identify, experimentally evaluate and implement the use of alternative field screening methods that are specific for environmental contaminants of interest and concern to the Department of Energy. Immunochemical techniques are rapidly becoming a significant component in the arsenal of field screening methods. Analytical results obtained by immunoassay have been shown to correlate well with those obtained by traditional laboratory methods. Also, the use of immunoassay-based field screening methods can significantly reduce the cost and time required for environmental assessment. For example, our previous experimental evaluations of immunoassay-based tests for PCBs and mercury showed them to be effective, rapid and economical field screening methods (Methods OS020 and MB100, DOE Methods Compendium). We are currently evaluating the effectiveness of immunoassay-based test kits, from a number of sources, for detecting fuel hydrocarbons (primarily BTEX) in soil. The formats of the kits being evaluated vary significantly - from how the test sample is diluted for assay to how the test color reaction is developed and measured. Dilution schemes have been shown to be crucial, due to the volatility of the analytes; and can result in false negative results. Kits, with which the color reaction development occurs on a membranous filter, are subject to producing erroneous and erratic results if the test samples and/or test reagents are compromised in such a way as to impede the normal flow rate through the filter. Results of these studies, with respect to the accuracy, reproducibility, sensitivity, working range, cost and sample throughput, will be presented.

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ON-SITE SOIL AND WATER ANALYSIS USING D TECH™ IMMUNOASSAYS FOR RDX AND TNT

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The secondary military explosives, trinitrotoluene (TNT) and hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) are commonly found in soils and ground water at munition load and pack operations, and demilitarization facilities. RDX is very mobile in soils, and is often used to indicate the leading edge of an explosives plume, whereas the rate of migration of TNT in the soil is far less. The development of the D TECH field screening methods for RDX and TNT in both soil and ground water have been designed to greatly benefit the explosives remediation effort. The use of these inexpensive field screening methods will reduce the total cost and improve the efficiency of site surveys and remediation projects. Field screening techniques can also circumvent the long turnaround time of laboratory analysis by providing reliable on site results.

The portable RDX and TNT enzyme immunoassays (EIA) are quick, cost effective, highly specific, and user friendly. The components of these EIAs include RDX or TNT specific antibodies (Ab) covalently linked to small latex particles, an RDX or TNT analog which is covalently linked to alkaline phosphatase, and the free explosive in the water sample. The free explosive competes with the enzyme linked analog for the Ab binding sites. The latex particles are then collected on a filter device, washed, and an enzyme substrate is added. The amount of color produced is inversely proportional to the concentration of free explosive in the water or soil sample, and can be determined using a hand held reflectometer, or a color card. These assays have demonstrated 5 ppb sensitivity in water samples and 0.5 ppm sensitivity in soils with minimal cross-reactivity and matrix effects.

The application of these tests in the field has been demonstrated at several sites across the United States. Results from a 42 sample TNT field trial at Army Ammunition Plant reported 2% false positives and no false negative results. When confirmed by EPA SW-846 Method 8330, 74% of the samples were in direct agreement. During a series of RDX field trials, the RDX EIA reported 3% false positives and no false negatives. In all field trials the EIA demonstrated a correlation coefficient of at least 90% when compared with Method 8330. Results from larger field studies have shown these tests to be cost and time effective as well as accurate.

SIMPLIFYING SITE ASSESSMENT OF PAH USING IMMUNOASSAY DIRECT - PCB FIELD SCREENING

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Polycyclic aromatic hydrocarbons (PAH) are carcinogenic and mutagenic chemicals found in combustion products and industrial waste materials. The carcinogenic nature of PAHs has led state and federal agencies to control their levels in the environment. Until recently, site assessment of PAH levels in soil was limited by the high cost and long turnaround time associated with analysis methods used in commercial analytical laboratories. The recent development of PAH immunoassays provides a rapid and cost effective alternative to the traditional analytical methods. One such immunoassay was developed as a part of the D TECH™ line of environmental detection systems. This competitive immunoassay uses anti-PAH antibodies immobilized on latex particles. Antibody binding to an enzyme labeled PAH analog is inhibited by free PAH extracted from soil samples or contained in water samples. The immunoassay detects the majority of compounds on the EPA list of 16 PAH priority pollutants below the ppm level without cross reacting with BTEX, PCBs and other similar compounds. Field samples of PAH were collected at both petrogenic and pyrolytic sites of contamination and the PAH concentrations were determined by both the immunoassay and the EPA SW-846 GC/MS method 8270. Test results correlated between the two methods and the low number of false negative and false positive results confirmed the assay's specificity for PAH. The immunoassay provides the advantages of rapid, on site analysis, and minimum sample preparation. Use of the immunoassay results in a significant savings in the cost of analysis because the cost per assay is lower than commercial analysis and only positive samples require additional confirmation by instrumental techniques. The simple format of the immunoassay allows personnel with minimal training to reliably and accurately determine the PAH content of soils.

PCB DETECTION BY IMMUNOASSAY - A WIPE TEST FOR SURFACE CONTAMINATION

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Immunoassay based field screening methods are gaining acceptance by the environmental diagnostics industry for on-site characterization and remediation monitoring. Polychlorinated Biphenyl's (PCB), a family of molecules classified as potential carcinogens, can be easily detected on-site by immunoassay screening methods. This results in reduced project cost and improved on-site efficiency, since field screening immunoassays short cut the long turn around time of laboratory analysis while providing reliable results. On site wipe test technology for assessing PCB contamination on surfaces such as walls and floors of PCB storage facilities has been developed to supplement the D TECH™ PCB soil assay. This sampling technique can also be used to monitor for transformer leaks, spills and to evaluate equipment decontamination processes. The D TECH PCB wipe test is quick, cost effective, highly specific and user friendly. The surface is sampled by wiping a 100 cm² area with a 1 cm² pad saturated with an extractant. The PCB is extracted from the sampling pad during a short extraction step. The sample is filtered, diluted, and run in the D TECH PCB field screening system. The components of the immunoassay include PCB specific antibodies (Ab) covalently linked to small latex particles, a PCB analog which is covalently linked to alkaline phosphatase, and the free PCB from the sample. The free PCB competes with the enzyme linked analog for the Ab binding sites. The latex particles are then collected on a filter device, washed, and an enzyme substrate is added. The amount of color produced is inversely proportional to the concentration of free PCB on the sample, and can be determined using a hand held reflectometer, or a color card. This assay has demonstrated 5 ug / 100 cm² sensitivity to a broad spectrum of PCB Aroclors. Results from case studies will be discussed.

DETECTION OF PETROLEUM HYDROCARBONS IN SOIL AND WATER BY IMMUNOASSAY

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Volatile aromatic hydrocarbons, which include BTEX (benzene, toluene, ethylbenzene and xylenes), are prevalent contaminants at hazardous wastes sites across the United States as a result of spillage of hydrocarbon fuels and leaking underground storage tanks. It has been estimated that 2 to 5 million underground storage tanks containing petroleum hydrocarbons or chemicals are present in the United States. Identifying the presence of hydrocarbon contamination is a high priority; suspected contaminated sites must be evaluated to map out a contamination plume, to determine if remediation is needed or to determine the effectiveness of remediation.

Groundwater and soil sampling are widely used to assess contamination. Exposure via drinking water, especially groundwater, is of great concern and cause for much of the hydrocarbon and underground storage tank regulations. The collection and analysis of soil and groundwater samples has become a standard practice in site assessment and has gained wide acceptance of regulatory agencies. However, accurate and precise characterization of a region is difficult due to inherent uncertainty in measuring volatile organic compounds (VOCs). Sample collection, handling, storage and transport, as well as large spatial and temporal variations can occur during sampling, which all contribute substantial error to accurate analysis.

A magnetic particle based enzyme immunoassay (EIA) that detects small aromatic hydrocarbons was developed. This assay offers several advantages over traditional testing methods (i.e. GC) including speed, cost effectiveness and portability. The assay can be performed on site in less than one hour. The assay can be coupled with simple extraction methods for analyzing petroleum hydrocarbons in soil.

Several studies were conducted to evaluate the assay's performance in soil and water. In a study conducted on water samples from locations across the U.S., recoveries of spiked Total BTEX averaged greater than 99%, with results ranging from 87 to 119%; one false positive (1.8%) was observed. Soils spiked with Total BTEX at concentrations ranging from 0.25 to 10 ppm Total BTEX were extracted, diluted and evaluated in the immunoassay. Recoveries averaged 113% with results ranging from 104 to 120%. In another study with 30 soil samples spiked with gasoline, jet fuel, kerosene and Total BTEX, the assay and recommended extraction procedure agreed well with results obtained by EPA Method 8020 in determining the presence and degree of contamination. Additional study results and data on the cross reactivity of the assay for various small aromatic hydrocarbons and petroleum fuel mixtures (i.e. gasoline), will be presented.

DETERMINATION OF POLYNUCLEAR AROMATIC HYDROCARBONS (PAHS) IN WATER AND SOIL BY A MAGNETIC PARTICLE-BASED ENZYME IMMUNOASSAY

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Use of immunoassays as field-screening methods to detect environmental contaminants has increased dramatically over the past few years. Immunochemical assays are sensitive, rapid, reliable, cost-effective and can be used for lab or field analysis. A magnetic particle-based immunoassay system has been developed for the quantitation of Polynuclear Aromatic Hydrocarbons (PAHs) in water and soil. Paramagnetic particles used as the solid-phase, allow for the precise addition of antibody and rapid reaction kinetics. The magnetic particle-based immunoassay is ideally suited for on-site investigation and remediation processes to delineate PAHs contamination. This system includes easy-to-use materials for collection, extraction, filtration and dilution of soil samples prior to analysis by immunoassay. Water samples are analyzed directly without sample preparation. The method detects PAHs, including anthracene, chrysene, fluoranthene, phenanthrene, pyrene and benzofluoranthene, at sub-parts per million levels in soil and at less than a part per billion in water. This PAHs ELISA compares favorably to HPLC determinations, and exhibits good precision and accuracy. The assay procedure and detailed performance characteristics including precision, spike recovery and correlation with U.S. EPA methods are discussed.

A QUARTZ CRYSTAL MICROBALANCE BIOSENSOR FOR MEASUREMENT OF 2,4-D IN WATER

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Due to the proportional relationship between vibrational frequency and surface mass, the quartz crystal microbalance (QCM) has been used to detect the accumulation of small quantities of chemical and biological compounds on electrode covered quartz crystals. Classically, these devices have been used to detect gasses and vapors which adhere to chemically selective coatings. Recently, however, increasing attention has been focused on the use of QCMs in aqueous environments. Herein reported is the development of an antibody-based QCM biosensor for measurement of (2,4-dichlorophenoxy)acetic acid (2,4-D) in aqueous systems. The dynamic range for the measurement of 2,4-D is 0.1 to 10 ppm.

A biotinylated-2,4-D coating antigen is immobilized on the gold electrode by means of a spontaneously formed streptavidin monolayer. 2,4-D (analyte) then competes with the surface-bound antibody for binding to anti-2,4-D IgG labeled with alkaline phosphatase. The amount of labeled antibody that binds to the crystal is inversely proportional to the analyte (2,4-D). The mass of the labeled antibody which binds to the crystal is then amplified by the enzyme catalyzed dimenzation of 5-bromo-4-chloroindolylphosphate (BCIP) which precipitates on the crystal surface. Consequently, the observed rate of frequency change is inversely proportional to the concentration of 2,4-D to which the sensor was exposed.

NOTICE: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), has prepared this abstract for a proposed presentation. It does not necessarily reflect the views of the EPA or ORD.

DETECTION OF SMALL MOLECULES BY AN EVANESCENT WAVE FIBER OPTIC BIOSENSOR

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Detection of small molecules is important in environmental analyses as most pollutants are low molecular weight compounds. Utilizing the specificity of antibodies, the rapid signal transduction of optical fibers and the signal-to-noise discrimination of fluorescence, an evanescent wave fiber optic-based biosensor was modified for the detection of these molecules. To facilitate the isolation of personnel and equipment from hazardous environments, long partially clad optical fibers are employed. Antibodies were immobilized on the 10 cm sensing region at the distal portion of the optical fiber probe. A 200 μ l sample chamber was fabricated from a capillary tube. The core of the fiber in the sensing region is tapered for maximum signal recovery. A competitive immunoassay was performed. Basically, the small molecule competes with a fluorescently-labelled analog for the capture antibody binding sites. As the level of analyte increases in the sample, the fluorescent signal decreases. Two assays for the small molecules, trinitrotoluene (TNT) and polychlorinated biphenyl (PCB) will be described. Detection levels of 10 ng/ml TNT (8 ppb) have been achieved with this sensor.

IMMUNOASSAY PANEL PROFILE FOR DETECTING TOTAL PCB CONTENT

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Immunoassay test kits are being widely used to provide rapid, inexpensive screening of soil samples for the presence of PCBs at or above a given threshold value. Currently available immunoassay methods are sensitive to aroclor preparations that contain the more highly chlorinated PCB congeners. The interpretation of these tests is accomplished by comparison to an appropriate aroclor standard. If PCB contamination at a site has undergone significant changes through weathering or biological degradation, or if contamination has occurred from lower chlorinated species, the relative sensitivity of available test methods is reduced. This paper describes the results of a program to develop and demonstrate an Immunoassay Panel Method that normalizes the recovery of PCBs detected and thereby provides an accurate representation of the total PCB content of a sample. The development and validation of this method, and the associative correlation testing data using laboratory and environmental samples, will be discussed.

PCB DETECTION BY THE CONTINUOUS FLOW IMMUNOSENSOR

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Increasingly, pollutants and hazardous waste materials have become a major problem within the environmental community. The monitoring and detection of PCBs (polychlorinated biphenyls) in soil, waste water, and other remote sites have cost the government millions of dollars. This study demonstrates a method of detecting and monitoring PCBs using a continuous flow immunosensor. Such an assay involves the immobilization of antibodies raised against PCBs onto a solid support matrix. The matrix is then saturated with the fluorescently labeled antigen. As the PCB is injected into the system, this labeled antigen is displaced and its presence is monitored downstream. Preliminary results have demonstrated detection sensitivities of less than 10 parts per million. This device and the effects of such factors as antibody density, antibody orientation, and flow rate on its performance and detection capabilities will be presented.

LIPOSOME-ENHANCED IMMUNOMIGRATION STRIPS FOR FIELD SCREENING OF TOXIC CHEMICALS

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The use of liposomes containing encapsulated dye to provide instantaneous enhancement of signals generated in competitive immunoassays is described. The application of this technology to Immunomigration Strip field assays for environmental contaminants is demonstrated, using Alachlor and PCBs as model analytes. The field assay as currently envisaged can be performed in two ways. The first method involves immobilizing antibodies onto a nitrocellulose strip and allowing the sample and analyte-tagged, dye-containing liposomes to migrate through this zone. Liposomes passing through without binding, as a result of competition from the sample analyte, are totally bound in a collection region, and the degree of color in this region is proportional to the analyte concentration. The second design allows the antibody to react in a competitive manner with the analyte and the liposomes in a homogeneous solution into which a test strip is subsequently placed. This test strip contains a liposome capture zone but no antibody zone. The antibodies in solution are capable of causing aggregation of the liposomes inversely proportional to the concentration of competing analyte, and aggregated liposomes do not migrate up the strip. Thus the amount of liposome found in the capture zone is again proportional to the amount of analyte present in the sample. The latter technique appears to be more sensitive, and has the added advantage that the strip is generic rather than specific for a particular analyte.

Using this technology, strip assays have been developed that will measure either the herbicide Alachlor or PCBs at the levels required by current regulations, as demonstrated by dose response data for both analytes. Both of these assays are complete in less than 15 minutes, and multiple assays can run at the same time. At present the quantitation of the colored zone is carried out by scanning the strips with a computer scanner and measuring the greyscale density of the capture zone, but alternate methods of quantitation are actively being investigated.

A possible design for a convenient and user-friendly assay is presented, along with some options for alternate strip designs and detection methodologies.

RAPID DIOXIN SCREENING BY ENZYME IMMUNOASSAY

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A system has been developed for rapid screening of 2,3,7,8-TetraChloroDibenzo-p-Dioxin (TCDD). The system uses a competitive inhibition Enzyme ImmunoAssay (EIA) based on a mouse monoclonal antibody which is specific for TCDD and related congeners. Sample preparation can be performed with a programmable automated extraction and cleanup system which uses disposable Teflon clad columns. The extraction and cleanup system has been extensively validated by GC-MS for a variety of sample types. The sample preparation system allows immunoassay analysis of soil, serum, water, and other matrices by taking each sample type to the same sample preparation endpoint. Concentration factors and endpoint conditions are completely flexible and programmable. Immunoassay analysis is performed by the addition of a prepared sample extract in organic solvent to an antibody coated microwell containing an aqueous sample diluent. This is mixed and incubated for 30 minutes to allow the immobilized antibody to capture analyte from the sample. The liquid is then removed and the well is washed to remove unbound materials. The well is then incubated with a competitor-HRP conjugate capable of binding specifically to the antibody sites not occupied by TCDD. After 30 minutes, the unbound conjugate is washed away and enzyme substrate is added for color development. The color generated is directly related to the amount of competitor-HRP bound in the second step, which is inversely related to the amount of analyte bound in the first step. After 30 minutes, a stop solution is added and the developed color is read on a microplate reader. The total time required for the EIA analysis of a prepared extract is less than 2 hours. Sensitivity for TCDD is better than 0.1 ng/well, allowing sensitive analysis of a variety of environmental matrices. Preliminary results indicate that it is possible to detect 10 ppb 2378-TCDD in soil by direct analysis of crude soil extracts. Work is being directed toward simplification of the extraction procedure and improvement of the interface with the automated sample cleanup system. The data presented here demonstrate that this system should be useful for TCDD screening in many situations for a variety of matrices. The system offers significant improvements in speed, sample throughput, and cost compared to GC-MS.

DEVELOPMENT OF AN ENZYME IMMUNOASSAY FOR THE DETECTION OF TRINITROTOLUENE (TNT) IN SOIL AND WATER

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2,4,6-Trinitrotoluene (TNT) is a toxic and persistent nitroaromatic compound currently found contaminating many government installations used for manufacture, storage or testing of explosives. The estimated 1,100 contaminated sites in the U.S. are the center of a large remediation effort, which could be streamlined by the use of a rapid, field-portable, inexpensive analytical method for TNT.

An enzyme immunoassay (EIA) has been developed which is capable of detecting as little as 0.5 ppb TNT in water and 0.25 ppm TNT in soil. Rabbit polyclonal antibodies were raised against a TNT-derivative coupled to bovine serum albumin. These antibodies are coated on both polystyrene tubes and 96-well microtiter plates. Water samples or soil extracts together with a TNT-horseradish peroxidase conjugate are then added and compete for antibody binding sites. Excess conjugate is washed away in a simple rinse step, and substrate (3,3',5,5'-tetramethylbenzidine) is added; the amount of color developed is inversely proportional to the concentration of TNT in the sample. This competitive EIA is relatively specific: 4-amino-2,6-DNT and 2,6-DNT are only 20% as reactive as TNT, and 2,4-DNT shows approximately 1% cross-reactivity. Other common explosives and important metabolites, such as RDX, HMX, nitroglycerin, and dichlorobenzoic acids, are essentially non-reactive in this assay. The microtiter plate-based assay is complete in 90 minutes and is suitable for laboratory use. The tube-based test is run in less than 30 minutes and is field-portable.

The soil extraction protocol employed for this assay utilizes methanol as the solvent and can be completed in 10 minutes. A simple dilution step is the only sample preparation that is required before running soil extracts in the immunoassay. Water samples require no pre-treatment, concentration or extraction. Correlative data between this immunoassay and conventional analytical techniques will show that the EIA has the potential to serve as a time and money-saving tool for soil and water survey and remediation.

DETERMINATION OF VINYL CHLORIDE IN SOIL-GAS SAMPLES BY GAS CHROMATOGRAPHY COUPLED WITH ION MOBILITY SPECTROMETRY

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The advantages of ion mobility spectrometry (IMS), such as ambient pressure operation, simplicity of design, high sensitivity, speed of response, spectral output, and ability to respond to a large number of compounds, make IMS technology attractive for a variety of field monitoring applications. A quantitative, multicomponent field detector based on IMS technology has not been successful, however, because of technology limitations. For multicomponent samples, multiple ion-molecule reactions result in confusing spectral information. Varying humidity can result in ion-water clusters that cause errors in both the identification and quantification of the target analyte. These limitations could be overcome through the addition of gas chromatography (GC) prior to high-temperature IMS. When coupling GC to IMS, the following need to be considered: (a) Currently available IMS cells have a relatively large internal volume (> 20 mL), reducing the chromatographic resolution; (b) smaller IMS cells are not capable of operating above 100°C; (c) the radioactive ionization cell limits the dynamic range of the device to 2 to 3 orders of magnitude; and (d) most standard miniature cells have a membrane inlet that does not allow direct interface with a GC column. We have overcome some of these difficulties by interfacing GC with Fourier Transform IMS (FTIMS). We will present data on determining vinyl chloride in soil-gas samples by GC-FTIMS.

NOTICE: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), prepared this abstract for a proposed oral presentation. It does not necessarily reflect the views of the EPA or ORD.

APPLICATION OF ION MOBILITY SPECTROMETRY TO DRUG DETECTION IN MARINE ENVIRONMENTS

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One of the U.S. Coast Guard's missions is the search of maritime vessels for the presence of illicit drugs. This mission requires that effective and efficient search and detection systems must have rapid analysis processing capabilities and minimal sample preparation and concentration requirements.

The application of Ion Mobility Spectrometry (IMS) has been determined by previous investigators to be effective in detecting trace quantities of illicit drugs. IMS is one of several technologies that the U.S. Coast Guard R&D Center has been actively investigating since 1991 for use in the maritime environment. The primary emphasis of these studies has been focused on the utility and performance of this technology in realistic field environments and field situations. These research studies have involved the use of IMS deployed in vans, mobile laboratories, and onboard U.S. Coast Guard vessels.

Results of these field studies, in which greater than 7,000 samples from various types of vessels, containerers, and cargo were collected and analyzed, will be presented.

ION MOBILITY SPECTROMETRY (IMS) FIELD SCREENING METHODS AND ANALYSIS OF EXPLOSIVES IN CONTAMINATED SOILS

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The rapid on-site and lab analysis of soil samples contaminated with DNT, TNT, and RDX is explored with IMS detection and various sample preparation methods. Field screening for explosives is presently time consuming and generates considerable amounts of additional chemical wastes. The field method is also limited in the number of different explosives that can be analyzed.

The use of a Barringer Instruments IONSCAN IMS system, which is comprised of a thermal desorption sample introduction system directly plumbed to the IMS reaction region and drift tube assembly permits novel methods of sample prep and sample introduction to be employed. Direct sampling, sample sublation, and sample extraction are all investigated and reported. Benefits of utilizing an IONSCAN in the analysis of soil samples is the ability to simultaneously analyze up to 18 analytes in each sample in 4.8 seconds! Therefore it is feasible to analyze for degradation products as well as the target parent explosive compounds.

The IONSCAN analyses were typically performed using 100 mg samples of soil or 100 ul aliquot's of the extracted sample solution directly deposited onto a PTFE filter and then desorbed. Additionally 100 mg samples of soil were directly solvated with assorted solvents on the sample filter with varied success. Performing a 3 minute acetone extraction on the soil samples has yielded the best overall results thus far with respect to limits of detection, and time required for sample preparation. 10 PPM or better limits of detection are achieved with this methodology and an IONSCAN for DNT, TNT, and RDX. Based on these preliminary results this work is being continued in both the lab and on-site with soil and water samples.

CHALLENGES OF DEVELOPING A GC/IMS BASED PERSONAL CHEMICAL HAZARD DETECTOR

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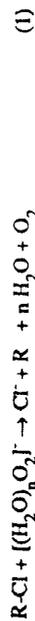
Hand-portable gas chromatography/ion mobility spectrometry (GC/IMS) techniques have been identified as a promising area of development for field and process related analytical problems. Recent development of extremely small (15 in³) personal IMS detection devices offer new prospects for creating a miniaturized GC/IMS unit. If a number of critical engineering obstacles can be overcome, these units will find new applications for monitoring individual exposure to chemical hazards based on having considerable size and cost advantages over their predecessors. The successful development of a combined GC/IMS personal detection system will require the combination of various innovative solutions which have been suggested or applied to various chemical sensor, GC and IMS technologies.

This paper provides a feasibility examination of personal GC/IMS based on the automated vapor sampling-transfer line gas chromatography (AVS-TLGC) approach previously utilized in developing hand-portable GC/IMS instrumentation. Special focus is placed upon developing low power strategies for implementing this approach. The means for achieving low power operation for extended personal use has focused on pulsed, on-demand operation of the sampling system, low (ambient) temperature GC operation, reduced GC flow rates and pressure drops and modification of the internal IMS system flows. Development efforts to date have utilized an existing prototype miniature IMS device, the GI-MINI from Graseby Ionics coupled with modified Enviroprobe AVS-TLGC equipment developed by FerroScan. Effect of parameters such as GC column position, temperature and carrier gas flow as well as IMS purge flows are examined for their effects upon sensitivity and resolution in breadboard GC/IMS test instrument. An evaluation of the remaining engineering hurdles limiting the development of personal GC/IMS are also presented.

FIELD SCREENING OF VOLATILE ORGANOCHLORINE COMPOUNDS USING ION MOBILITY SPECTROMETRY

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The most common ion source used in ion mobility spectrometry (IMS) is still a ⁶³Ni foil placed inside the reaction region of the measuring cell. In atmospheric pressure ionization processes, water clusters of the composition [H₂O]_nH⁺ or [(H₂O)_nO₂]⁺ are formed. These reactant ions undergo further ion-molecule reactions with neutral gas-phase analytes to produce analyte "product ions". In most cases, the positive or negative product ions formed are pseudo-molecular ions. In the negative ion mode, especially halogenated compounds obey a typical dissociative electron capture reaction (Eq. 1).



This reaction can be used to determine volatile organic compounds in different samples.

In this paper, we report applications of hand-held BRUKER RAID-1 ion mobility spectrometers for the determination of the total amount of volatile organic halides in air samples, soil air, sludge, waste water and on waste disposal sites. The measurements are compared with the results obtained by mobile GC/MS - techniques and with the total amount of adsorbable or extractable organic halogens (AOX/EOX) using the German standard method for the examination of water, waste water and sludge (DIN 38 409, H 14). The results suggest that the obtained sum parameter correlates in most cases with the well-known AOX/EOX value.

ON-SITE ANALYSIS OF OLD DEPOSITED CHEMICAL WARFARE AGENTS BY COMBINED USE OF ION MOBILITY SPECTROMETRY AND MASS SPECTROMETRY

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Over the last decade, Ion Mobility Spectrometry (IMS) has evolved into an inexpensive and powerful technique for the detection of trace compounds. Most of the ion mobility spectrometers are rugged and can be used as hand-held instruments in field analyses. If equipped with a ⁶³Ni radioactive source, especially compounds with a high proton or electron affinity can be detected at trace levels. In general, a high proton or electron affinity is typical for toxic compounds. Therefore, on-site detection of hazardous substances is a typical application for IMS because

- identification and quantification of hazardous compounds is possible within a few seconds, which is essential for on-site personnel protection,
- the method can be used for a very fast field screening as well as for measurements of indoor contaminations or inspections of production sites and
- application of IMS reduces, as one result of the screening, the number of samples which have to be investigated by standard laboratory methods, e.g., GC/MS.

The BRUKER RAID-1 ion mobility spectrometer can either be used directly as an automatic warning instrument programmable for up to ten compounds or with the aid of a data system if unknown compounds have to be detected.

Examples given for the application of IMS instruments result from investigations of old production sites of chemical warfare agents, ammunition plants or training areas. Chemical warfare agents and their degradation products were analyzed in residues of the production processes, in the atmosphere of bunkers used for storage, in soil air and in soil samples using a simple head space technique. In most cases, IMS measurements were combined with GC/MS investigations. The results clearly indicate, that GC/MS investigations confirm the findings of the IMS.

UXO DETECTION AT THE JEFFERSON PROVING GROUND, MADISON, IN

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OBJECTIVE

The objective of this project is to quantitatively evaluate the effectiveness of high spatial frequency sampling with the USRADs System and the Minefield Reconnaissance and Detector (MIRADOR) System for locating, mapping and identifying subsurface unexploded ordnance (UXO), consisting of bombs, missiles, mines, projectiles, submunitions and rockets.

TYPE OF TESTING AND EVALUATION TO BE CONDUCTED

A drive-over survey of the 40 acre Jefferson Proving Ground (JPG) test site will be conducted. The USRADs System will record the MIRADOR's location each second to an accuracy of +/- 6". Markers will be placed in the MIRADOR's data files to correlate data with location.

MIRADOR beneficially combines the outputs of multiple sensors, three Ground Penetrating Radars (GPR's) and three Metal Detectors (MD's), to produce a composite result that is better than the result from any single sensor. This data fusion process accommodates multiple sensor output data containing gaps, noise, dropouts, and other flaws, and still produces a good output result. Further, it enhances detection and location of target objects.

Data from MIRADOR will be combined with results from other sensor technologies such as terrain conductivity meters and various magnetometers (as selected by the Selection Committee from other Chemrad technology demonstration proposals) to produce the best possible result.

These anomaly identifications will then be compared with the target information provided by the Naval Explosive Ordnance Disposal Technology Division, Manager of JPG, to measure the effectiveness of this approach for locating buried, unexploded ordnance.

PRELIMINARY RESULTS FROM A FIELD SURVEY CONDUCTED WITH THE AUTOMATED SUBSURFACE CHARACTERIZATION SYSTEM (ASCS)

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OBJECTIVE

The Automated Subsurface Characterization System (ASCS) is a portable system that uses a ground conductivity sensor and a ground penetrating radar sensor to survey and characterize subsurface objects, conditions, and geology. The objective is to demonstrate the system's capabilities by performing an actual field survey and reducing and reporting the results. The concepts and technologies employed are applicable to a wide range of hazardous waste, ordnance detection, and site survey and monitoring tasks.

TYPE OF TESTING AND EVALUATION TO BE CONDUCTED

The specific application reported here is a survey of a chemical waste landfill site performed both before and after injection of a subsurface grout barrier. The site, which has been used for various chemical spill/burial applications, has been studied in detail and the gross physical and geological properties are known. Scientists from Sandia National Laboratories Albuquerque will inject a combination of various grouts and waxes at selected locations and depths. The barriers will be injected through boreholes for the purpose of minimizing the site subsurface permeability and the subsequent migration of chemical waste material and groundwater. After injection and thorough curing of the barriers, each injection location will be excavated to determine the integrity, continuity, uniformity, and physical extent of the injected barriers.

A survey of the chemical waste site, performed both before and after the injection of the barrier, will be compared with the ASCS. The results and findings (barrier characterization) from this survey will be compared and contrasted with actual barrier conditions determined after excavation. In this way, the suitability and applicability of an ASCS-like system that is portable, has hand-carried or man pulled sensors, and which carries out data reduction essentially immediately will be demonstrated.

INTERLABORATORY EVALUATION OF VOLATILE ORGANIC COMPOUND DETERMINATIONS IN SOILS PREPARED BY VAPOR FORTIFICATION

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Despite the large number of vadose-zone soil samples routinely analyzed for volatile organic compounds (VOCs), no secondary reference soils are available for procedures such as calibration evaluation, quality assurance/quality control (QA/QC), and method comparisons. Currently the accuracy of VOC determinations is usually estimated via solution spike and recovery tests. This approach fails to mimic the extraction of real samples because of the large amount of carrier solvent and the insufficient contact time for natural sorption processes to occur.

Vapor equilibration for several days in a closed system was used to produce fortified soil samples contained in 1.0-mL glass ampoules. This preparation method was soil specific, precise within and between batches, and analyte concentration stability was demonstrated to exceed a 60-day holding period. With such samples we were able for the first time, to our knowledge, to distribute samples for interlaboratory analysis where both extraction and determinative accuracy were evaluated together. Each laboratory was sent duplicate sets of three different soil subsamples, each fortified with trans-1,2-dichloroethylene (TDCE), trichloroethylene (TCE), benzene (Ben) and toluene (Tol). Each soil set consisting of subsamples from two separate fortification batches. Twelve laboratories submitted analyte concentration estimates for these soils using methanol extraction purge-and-trap gas chromatography/mass spectrometry analysis (EPA, SW 846, Method 8240).

Statistical analysis resulted in the exclusion of all data from two of the laboratories due to substantial systematic error. A improperly sealed ampoule necessitated exclusion of one data set for soil A, and one set was excluded from soil B due to a very large (statistically significant) variance. Consequently soils A and B were characterized using all the results from nine laboratories, while soil C data were from ten laboratories. There were no significant differences ($\alpha=0.05$) between batches for any analyte in any soil. The relative standard deviation, including random batch variation, ranged from 8.5% to 28.2% for the 12 analyte/soil combinations. Half of the RSDs were less than 10.0%, and the average was 12.6%. The best results were for Ben, while TDCE produced the largest uncertainties.

This round-robin study has confirmed that VOC vapor fortified soil subsamples can be reproducibly prepared and, once sealed in glass ampoules, are stable during distribution so they can be used as secondary standards for various performance evaluation applications.

A DATABASE FOR EVALUATING FIELD-PORTABLE XRF PERFORMANCE INFORMATION FROM HAZARDOUS WASTE SITES

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The U.S. Environmental Protection Agency's (U.S. EPA)/Environmental Response Team (ERT) has used field-portable X-ray fluorescence (FPXRF) instruments extensively to characterize metallic contamination in soils and sediment. Rapid, on-site, multi-element analysis of large numbers of in-situ and prepared samples provides near real-time data to guide cleanup/removal activities at hazardous waste sites. Currently, there is a high level of interest in FPXRF Quality Assurance/Quality Control (QA/QC) results, since FPXRF is a relatively new environmental analytical technique.

Managing large amounts of FPXRF QA/QC data from many sites is difficult because it is typically stored in separate project reports. Electronic management and evaluation of FPXRF performance is desirable. A database, providing a means to store, summarize, compare, and manipulate FPXRF QA/QC performance, is described. Data is entered from the keyboard, through picklists, or by import of electronic files from other sources. Database functions provide easy access to, manipulation, and sorting of, FPXRF QA/QC data. Additionally, an output menu provides a variety of pre-defined report formats. This database quickly provides site-managers with realistic estimates of FPXRF capabilities.

FIELD SCREENING VS. FIELD ANALYSIS: DATA QUALITY OBJECTIVES IN THE AGE OF ADVANCED ON-SITE INSTRUMENTATION

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Field analytical efforts range from true screening instruments such as hand-carried organic vapor analyzers to full on-site laboratory-grade GC/MS instruments. Frequently, the end use of the data is either unknown, or ill-defined, resulting in an inappropriate analytical technical approach. Increasingly, data users are intending to use field data for all possible uses, from determining the depth for bore-holes to modelling and risk assessment. This broad mandate presents severe challenges to the field analysis effort.

One of these challenges is to educate data users who may not be accustomed to analytical methodology and its terminology on the meaning of data quality. Often the term "screening" carries a negative connotation for many users—implying much lower quality than laboratory analytical data. An understanding of the data quality that is attainable in the field from an EPA method context will counteract that impression.

Another challenge lies in the need for sophisticated data reporting, often including input into databases. Most field instruments are not amenable to more than simple reporting. This simple reporting limitation can extend to the total data quality, since additional quality assurance data may not be easily incorporated into field reporting without significant additional effort.

The paper will review the EPA Data Quality Objective process vis-a-vis currently available field instrumentation. Examples of the data quality obtainable from field instruments will be presented: from routine field analysis to a one-analysis approach for all data uses. The data will show typical limits to the quality of field generated data when the objective is to adhere to EPA method protocols.

SCREENING OF ALPHA CONTAMINATED SOILS USING LOW-RESOLUTION ALPHA SPECTROSCOPY OF THICK SOIL SAMPLES

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High-resolution alpha spectroscopy has been used extensively as an analytical tool for identifying unknown radiological contaminants in a variety of matrices. It has, however, been an expensive and time-consuming procedure, primarily limited to the laboratory setting, due to the need to fabricate very thin uniform "weightless" samples in order to preserve the high energy resolution of the system. An alternative approach, using low-resolution alpha spectroscopy of thick samples, has been suggested by A. C. Lucas (Annual Meeting of the Health Physics Society, Atlanta, GA, June 1993). For the case of thick samples, the energy spectrum of emitted particles is broad and rectangular in shape, rather than being sharply peaked as in the conventional case. The sharp high-energy edge of the energy spectrum corresponds to the discrete alpha energy usually associated with the radionuclide of concern. Therefore, the spectrum analysis software may be configured to search for discrete edges in the energy spectrum rather than sharp peaks. We have demonstrated that as many as eight different radionuclides and progeny may be identified in a complicated spectrum. The advantages of this approach over high-resolution spectroscopy include (1) low capital cost of equipment, (2) small footprint of equipment, allowing for deployment in a field laboratory, and (3) minimal sample preparation, leading to high throughputs. We have used this approach to analyze quantitatively National Reference Material soils from Grand Junction and field samples from Fernald, Ohio (for U and Th), and contaminated soils from the Nevada Test Site and the Rocky Flats Plant (for Pu and Am).

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NEW FIELD-DEPLOYABLE IMAGE PROCESSING SYSTEM FOR SAME-DAY ANALYSIS OF NUCLEAR TRACK DETECTORS

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Nuclear Track Detectors (NTD's) can be used for detecting alpha-emitting radiological contamination (e.g. Pu, Am, U, Th) on surfaces and in soils. The primary disadvantage of this approach for field screening applications is the time required for analysis of the detectors. At present it is necessary to ship the detectors to a vendor, where they are etched and optically scanned, which incurs a time delay between retrieval and results of typically 2-7 days. A R&D team at ORNL has developed a prototype analysis system which is sufficiently compact to be deployed in a van or on-site laboratory. The complete system consists of etch/rinse tanks, optical microscope, stepping motors, CCD camera, frame-grabber, and an image analysis software package. In addition to conventional gross counting of etch pit densities, two new capabilities are under investigation. Hot particles are radioactive particles of high specific activity and small size. They pose a significant health hazard and are difficult to detect. Alpha particles are emitted radially from hot particles, and therefore hot particles generate 'starburst' clusters of tracks in a track detector with very characteristic patterns. The image analysis system can be configured to search for and analyze these patterns, yielding quantitative size distributions of hot particle contaminants. This information could be useful input to the site remediation plan. Using this approach, the presence of hot particles has been determined in soil samples from the Nevada Test Site and Johnston Atoll. The second capability involves identification of radiological contaminants. A group at the University of Bristol, UK, has demonstrated that detailed analysis of the size and shape of individual alpha tracks can lead to a determination of the energy of the alpha particle that generated the track. Alpha particles are emitted at discrete energies from different radionuclides, and therefore inexpensive and fast alpha spectroscopy can be carried out to identify unknown alpha-emitting contaminants. Progress on optimizing the image analysis system for this application will be reported.

*Research sponsored by the U.S. Department of Energy, under contract No. DE-AC05-84OR21400 with Martin Marietta Energy Systems Inc.

AN ANALYTICAL PROGRAM FOR DETERMINATION AND CONFIRMATION OF ARBORNE LEVELS OF CHEMICAL AGENT IN THE EVENT OF A SUSPECTED RELEASE

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The Monitoring Branch Laboratory of the U.S. Army Operations Directorate, Chemical Support Division is uniquely outfitted to respond quickly when the need arises to identify and measure atmospheric contamination levels of military unique compounds. Samples collected with a vacuum pump on solid sorbent tubes are initially screened using gas chromatography (GC) and a sulfurphosphorus specific detector to determine and quantify the presence or absence of contaminants. Positive samples from the first stage of testing are subjected to subsequent analysis with different detectors to confirm genuine positives and eliminate false positives. Subsequent testing provides information during follow-up and site remediation activities through screening of soil and other environmental samples.

Monitoring Branch uses Depot Area Air Monitoring Systems (DAAMS) technology in which a sample is collected onto a solid sorbent tube, and subsequently analyzed through thermal desorption of the sample into a gas chromatograph equipped with a simultaneous dual Flame Photometric Detector (FPD) for sulfur and phosphorus detection. Confirmation is accomplished using the same solid sorbent/thermal desorption/GC instrumentation with Atomic Emission Detection to confirm through elemental analysis and Mass Spectrophotometric Detection for structural confirmation. Soil and other environmental samples such as building debris are extracted manually using an organic solvent and analyzed using DAAMS technology and the same variety of detectors.

Data generated during implementation of QA/QC programs for sample collection and analysis indicates system responses are linear within a wide dynamic range. Results of precision and accuracy trials as well as field tests during hazardous response activities are included in this presentation. A single basic instrument system consisting of solid sorbent cartridges for sampling, concentration and injection, with multiple GC detectors for screening and confirmation through elemental and structural analysis, is able to carry out all monitoring activities, from initial confirmation of contamination through characterization of the extent of the release and follow-up during remediation.

THERMAL SAMPLING HEAD AND SYSTEM FOR CONCRETE WALLS

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The objective of this work is to make available a means for rapid, non-destructive sampling of surface and near surface semi-volatile contaminants on porous construction materials such as concrete. The sampling head is designed to heat a 100 cm² area of a typical surface to a temperature of 250 °C in 60-90 seconds. This head interfaces with a quick look detector (photoionization detector) and a multi-sample sorption tube collector to allow for sample recovery and laboratory analysis. First order models of the thermal and diffusion properties of concrete suggest that the heated and sampled regions extend several millimeters into the surface. The thermal performance of the sampling head has been evaluated using buried thermocouples and an imaging pyrometer. The results of initial sampling experiments on controlled laboratory samples and environmental surfaces will be discussed in terms of sampling efficiency and model predictions.

A MAN-PORTABLE SUPERCRITICAL FLUID EXTRACTION SYSTEM

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Supercritical Fluid Extraction (SFE) is gaining acceptance as an alternative sample preparation method. Current extraction methods, which include Soxhlet and sonication methods, are time-consuming and require relatively large volumes of organic solvents which generate hazardous waste. The liquid-like solvating power and rapid mass-transfer properties of supercritical fluids provide for faster and more efficient extraction than is feasible with liquids. Because carbon dioxide or carbon dioxide mixed with low percentages (e.g., <10%) of polar modifiers exhibit sufficient solvating power to extract many analytes of interest, only small volumes of liquid solvent are required for SFE. In addition, because SFE is amenable to automation, the technique is attractive for implementation in the field.

A new prototype SFE instrument has been developed for the on-site preparation of solid samples for subsequent analysis. The instrument is comprised of two parts, each of which satisfy weight and size requirements for portability. They are, a generator module that supplies high pressure carbon dioxide and an instrument briefcase containing extraction cells, flow restriction hardware, equipment for three different methods of collection, control apparatus, and operating supplies. The generator module provides carbon dioxide at pressures to 2 kbar and incorporates no moving parts. The module allows rapid loading without tools, due to novel finger-tight closures. Fluid from the generator is throttled to extraction pressures not to exceed 700 bar, and is preheated for introduction to each of three extraction cells. Flow may be switched between each of the three extraction cells, allowing operation of one cell while depressurizing another and loading the third with sample. Extraction fluid modifiers may be added to the extraction cells or introduced to the fluid flow from the generator. The heated extraction cells feature rapid, finger-tight closures, which allow swift turnover in sample processing. Extraction efficiency is enhanced by radial flow of the extraction medium through the sample, rather than the typical axial flow. Samples are contained in sintered inserts that fit within the extraction cells. Analyte collection may be accomplished using liquid solvent collection, adsorbent collection, or restrictionless rapid depressurization collection. Robust control schemes allow operation at a wide variety of pressures and temperatures and the use of static, dynamic, or hybrid static dynamic extraction strategies.

The technical performance of the prototype instrument will be evaluated by comparison to Soxhlet extraction using representative analytes and matrices. Operation aspects will be summarized based on preliminary field demonstrations. Field ruggedness evaluations of the prototype instrument will also be described.

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*Pacific Northwest Laboratory is operated by Battelle Memorial Institute.

PREPARATION OF TEDLAR® BAG WHOLE AIR STANDARD WITH A SUMMA® CANISTER FOR FIELD VOC ANALYSIS

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As the application of field analyses of Tedlar® bag samples is growing, the standard preparation has become more critical. Traditionally Tedlar® bag standards were prepared by directly injecting a target compound mixture into a Tedlar® bag pre-filled with zero air or nitrogen. It is very time consuming because standards must be prepared daily due to limited stabilities of volatile organic compounds (VOCs) in a Tedlar® bag. It also introduces several variables to the process of standard preparation, such as the accuracy of the volume of zero air or nitrogen in the bag and the injection volume of standard mixture. A new application of Tedlar® bag standard preparation in the field will be introduced in this paper. A pre-prepared SUMMA® canister standard with targeted VOCs is used to fill a Tedlar® bag in the field each day. It takes advantage of long term stabilities of VOCs in a SUMMA® canister, simplifies the process of Tedlar® bag standard preparation in the field and limits the variables in the process to increase the reproducibility. This paper describes the recent study of this method. Its comparison with traditional method of Tedlar® bag preparation is discussed. The results on reproducibility, humidity effects, temperature dependency and time dependent stabilities will be presented.

OPTIMIZATION OF FUNDAMENTAL PARAMETER METHODS FOR ANALYSIS OF HAZARDOUS MATERIALS WITH FIELD PORTABLE XRF ANALYZERS

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Field portable X-ray Fluorescence (XRF) instruments have been widely utilized by the U.S. Environmental Protection Agency's (U.S. EPA) Environmental Response Team (ERT) to characterize metallic waste contamination in soil and sediment. Rapid, on-site XRF analysis provides near real-time data which may be used to guide clean-up/removal activities at hazardous waste sites. Historically, XRF analyses based on site-specific calibration standards have not been useful for more than one site and/or sample matrix. Fundamental Parameters (FP) techniques provide multi-site capabilities by eliminating the requirement for site-specific standards. Inter-element matrix effects are predicted theoretically and intensity response is normalized to a set of pure element standards. The resultant application is, in principle, suitable for analysis of target elements for a given sample type (soil, water, oil, thin films, etc.) at any site. However, uncertainties in the data used to generate theoretical coefficients may lead to errors and biases in FP analytical models based on them. Therefore, adjustments based on assayed materials (such as NIST Standard Reference Materials, SRMs) may be necessary to "fine-tune" results. This paper describes methods to "fine-tune" FP models for analysis of hazardous materials in soil, sediment, and other matrices. The effectiveness of the approach will be demonstrated utilizing a commercially available field portable XRF analyzer and associated PC based software.

A NEW XRF METHOD FOR MEASURING THE CONCENTRATION OF BURIED LEAD IN PAINT USING L X-RAYS

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A new method has been developed that makes it possible to accurately and quickly determine the absolute concentration of lead in lead paint (in mg/cm^2) without knowledge of the composition or thickness of the layers overlying the lead. The invention makes use of the fact that the ratio of the mass attenuation coefficients for the L_{α} at 10.5 keV to the L_{β} at 12.6 keV is effectively independent of the Z of paint layers. The theory and confirming experimental data will be presented for a lead layer of $1 \text{ mg}/\text{cm}^2$ buried by up to $200 \text{ mg}/\text{cm}^2$ of non-lead paint (more than 1 mm thick). The new method also gives a measure of the depth of the lead beneath the surface. We will describe the NITON XL, which uses the invention to give the lead concentration and its depth index.

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