Task 1.11 - Spectroscopic Field Screening of Hazardous Waste and Toxic Spills

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TASK 1.11 - SPECTROSCOPIC FIELD SCREENING OF HAZARDOUS WASTE AND TOXIC SPILLS

1.0 INTRODUCTION

Techniques for the field characterization of soil contamination due to spillage of hazardous waste or toxic chemicals are time-consuming and expensive. Thus, more economical, less time-intensive methods are needed to facilitate rapid field screening of contaminated sites. In situ detection of toxic chemicals in soil offers both time and cost advantages for field screening with additional application to real-time site monitoring.

One method to assess soil contamination by hydrocarbons is soil gas survey. Soil gas measurements offer a rapid and relatively inexpensive means of site characterization. However, soil gas methods are subject to influences from size and age of the contaminant source, moisture and organic content of the unsaturated zone, volatility and solubility of the volatile organic carbon (VOC) source (1). Soil gas surveys also suffer from the effects of biochemical processes, since biodegradation may remove the low molecular weight VOC vapors in the vadose zone (2). In addition, soil gas surveys also have inherent limitations in their spatial resolution (3).

Lieberman and coworkers have demonstrated the combination of laser-induced fluorescence spectroscopy with cone penetrometry (LIF-CPT) as a means to estimate the extent to which a region of soil has been contaminated with petroleum hydrocarbons (4). The LIF-CPT method has also been shown to overcome some of the spatial resolution limitations of soil gas measurements (5). The LIF-CPT method however, is currently limited to detection of higher molecular weight aromatic hydrocarbons that fluoresce and is not sensitive to detection of lighter, more volatile compounds such as benzene, toluene, ethylbenzene, and xylene (BTEX) (5).

Fourier-transform infrared (FT-IR) spectroscopy coupled with evanescent mode fiber-optic sensors has been demonstrated as a means to remotely detect and classify petroleum products in water using mid-infrared (MIR) optical fibers (6). This work demonstrated that a fiber-optic evanescent field absorbance sensor (EFAS) could be used to classify petroleum contamination into categories such as crude oil, kerosene, No. 2 fuel and residual distillates using the MIR spectral range. Conzen, and coworkers, utilized silicone-clad silica optical fibers coupled with near-infrared (NIR) spectroscopy to remotely detect dichloromethane, chloroform, and trichloroethylene in aqueous solution (7). Klunder and Russo used NIR spectroscopy with optical fibers to monitor diffusion of VOCs into a silicone rubber cylinder (8). They obtained both qualitative and quantitative information on trichloroethylene, 1,1-dichloroethylene, and benzene in aqueous solution and vapor phase.

An in situ method based on the concepts presented above would employ an EFAS similar in design to that proposed by Conzen, and coworkers, but coated with a polymer to allow solid-phase extraction of analytes to improve the method's sensitivity. The fiber optic will have cladding that is hydrophobic and oleophilic such as that suggested by Klunder and Russo [8]. Additionally, the process by which the detection system of the EFAS collects VOCs should be quantitative and if possible, reusable. Work has shown that the partitioning of organic species into thin polymeric films are reversible, quantitative, and possess the selectivity for extraction of VOCs from aqueous solution and vapor phase when used in solid-phase microextraction (SPME) (9-11). Spectroscopic

studies in the mid-infrared range have shown polymeric materials to be useful coatings for infrared attenuated total reflectance (IR-ATR) measurements (12,13) of chlorinated hydrocarbons in aqueous solution.

The work cited above strongly suggests that an EFAS sensor can be developed that employs MIR spectroscopy for detection of VOCs in both aqueous and vapor phases. Such a sensor can prove useful in the application of in situ field screening when coupled with a delivery method such as cone penetrometry.

2.0 OBJECTIVES

The overall objective of this project is to study the feasibility of using an EFAS FT-IR spectroscopic sensor coupled with cone penetrometry as a field screening method. The Fourier transform infrared cone penetrometry method (FT-IR-CPT) will be developed by building on the work cited above. The specific objectives of this project are:

- Design an accessory for use with FT-IR that interfaces the spectrometer to a cone penetrometer.
- Characterize the response of the FT-IR accessory to selected hydrocarbons in a laboratory-simulated field environment.
- Determine the ability of the FT-IR-CPT instrument to measure hydrocarbon contamination in soil by direct comparison with a reference method (e.g. Soxhlet extraction followed by gas chromatography) to quantify hydrocarbons from the same soil.

3.0 WORK PERFORMED AND RESULTS

Work performed during the first two quarters was focused on three areas: compilation of current literature available relating to the project objectives with review of reported work, development of a conceptual design for the EFAS sensor, and characterization of a candidate polymeric film for use in SPME of analytes onto the sensor.

3.1 Review of Published Work

A literature search was done to obtain a list of published work relating to the project objectives. Bibliographic information for the articles currently on file are stored in a computer database. A complete listing is contained in Appendix A.

Recently published work on spectroscopic applications using optical fibers and SPME has been reviewed in order to apply this knowledge to the project goals.

A member of the project team attended the Fourth International Symposium on Field Screening Methods for Hazardous Wastes and Toxic Chemicals organized by the U.S.

¹Environmental Protection Agency (EPA) and the Air and Waste Management Association. Several presentations at this conference described either development or application of field screening methods employing fiber-optic technology with spectroscopic methods. A summary of the sessions attended is included in Appendix B. Proceedings from this conference will be available near the end of July 1995.

3.2 EFAS Concept

The proposed sensor will use fiber-optic evanescent field absorption as a means of spectroscopic detection. The EFAS will have a polymeric cladding in the active sensor region that possesses the desired characteristics described above. Thus, the EFAS will utilize SPME to extract and concentrate analytes on the EFAS, allowing MIR spectroscopic detection of VOCs in aqueous and vapor phases. The sensor is being developed with the goal of being able to mount the EFAS into commercially available cone penetrometers. This will allow the EFAS to be deployed in the field for in situ screening of hazardous and toxic chemical spills.

3.3 Evaluation of Polymeric Solid Phase for SPME

A candidate material for cladding the EFAS has been selected based on a previous study of its capability for direct SPME of VOCs in aqueous solution (14). The material is an inexpensive commercially available film typically used for sealing laboratory sample vials and jars¹. Work performed under this contract has collected data on the extraction capabilities of Parafilm-M[®] for aqueous solutions of trichloroethylene (TCE), ethylbenzene, and *m*-xylene.

An experiment similar in principle to that of Zhang and Pawliszyn [10] was devised to evaluate the characteristics of Parafilm-M[®] for use as an SPME solid phase for headspace sampling. Our experiment differed in the geometry and type of the solid phase (Parafilm-M[®]). We extract the VOCs from the headspace with the Parafilm-M[®] and analyze the components directly in the solid phase via MIR absorbance spectroscopy. This is unlike conventional SPME, where the analytes are thermally desorbed from the solid phase directly into an instrument such as gas chromatography-flame ionization detection (GC-FID) or GC-mass spectroscopy (MS).

For each of the test compounds TCE, ethylbenzene, and *m*-xylene, calibration curves were determined. These data sets represent the ability of Parafilm-M[®] to quantitatively extract analytes in the headspace over aqueous solutions. For all three compounds, the regression of analyte concentration against MIR absorbance shows excellent linearity and repeatability, as shown by the data of Table 1.

It being established that quantitative extraction is possible with Parafilm-M[®], work was initiated to determine the optimum temperature at which the solid phase collects analytes. This work used the same general procedures as that of the calibration experiments. MIR absorbance versus temperature curves were collected in headspace over aqueous solutions of 10 ppm for TCE, ethylbenzene, and *m*-xylene using an exposure time of 90 minutes. Data were collected at five temperatures ranging from 20°C to 50°C. A plot of absorbance versus temperature for all analytes is shown in Figure 1. In order to plot the data for all three compounds on a common set of axes, the MIR absorbance was normalized to the maximum value obtained.

¹PARAFILM-M[®] - American National Can, Greenwich, CT 06836.

TABLE 1

Calibration Data			
Parameter	<i>m</i> -Xylene	Ethylbenzene	Trichloroethylene
Calibration Range (ppm)	1-35	2-50	5-100
R Square	0.9988	0.9995	0.9997
Standard Error	0.5088	0.4316	0.6561
No. of Observations	7	7	7
Intercept (ppm)	0.47	1.65	0.49
Slope (ppm/AU)	209.16	500.13	1158.17
%RSD @ Concentration	0.47 @ 1.75	1.22 @ 4.99	2.41 @ 10

Figure 1. Analyte partitioning versus temperature.

As shown in Figure 1, as the temperature increases, the extraction efficiency increases in direct proportion to temperature up to 37°C. Detailed analysis of the MIR spectra obtained from these experiments revealed that at approximately 50°C, thermally induced structural changes in the solid phase occur, causing distortions in the spectrum baseline. Therefore, 37°C has been selected for a temperature at which an acceptable compromise is reached between extraction efficiency and thermal degradation effects in the stationary phase.

Since SPME is a diffusion-based method, it will show a strong dependence on temperature and time. Experiments were carried out to determine partitioning of analytes into Parafilm-M® as a function of exposure time at a fixed temperature and analyte concentration. Partitioning curve data (MIR absorbance versus time) were collected for TCE, ethylbenzene, and *m*-xylene at a temperature of 37°C and a fixed concentration (TCE = 10 ppm, ethylbenzene = 10 ppm, *m*-xylene = 5 ppm). A graph depicting the data collected is shown in Figure 2. As with the temperature data of Figure 1, the data are calculated to a common Y-axis by normalization to the maximum value obtained for the compound.

As with the extraction temperature study, an exposure time was selected based on a compromise between practical and theoretical considerations. For practical reasons, the extraction time should be minimized. However, if too small an extraction time is chosen, analyte detection limits will increase, reducing measurement sensitivity. Figure 2 shows that as expected, the amount of analyte that has diffused into the Parafilm-M® increases with time. A subtle break appears in the partitioning curves of Figure 2 at approximately 180 to 200 minutes exposure time. Past this point, the slope of the partitioning curves decrease. Therefore, 180 minutes has been chosen as an extraction time in this preliminary work.

Figure 2. Analyte partitioning versus time.

4.0 CONCLUSIONS

Past work by other researchers has shown that construction of a spectroscopic method utilizing an evanescent field absorption sensor based on fiber optics and solid-phase microextraction is possible. Further development of this technology will allow evaluation of its use for field screening applications using cone penetrometry.

The data presented here show Parafilm-M® is a useful solid phase for extraction of hydrocarbons from the headspace above aqueous solutions. The data also show that mid-infrared spectroscopy is applicable to analysis of the analytes collected on the solid phase.

5.0 FUTURE WORK

Continued development of the EFAS sensor using MIR optical fibers and Parafilm-M® will be carried out over the next quarter. The EFAS sensor response to analytes will be characterized in the headspace over aqueous solution, hydrocarbon-spiked sand, and hydrocarbon-spiked soil. Further sensor development will involve incorporation of the unit into a cone penetrometer assembly, which will be evaluated as a soil probe to determine hydrocarbon contamination in a laboratory-simulated field environment.

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APPENDIX B

FIELD SCREENING SYMPOSIUM SUMMARY

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