

Characterization, Monitoring and Sensor Technology Integrated Program

Poster Session Handout

R&D/DT&E Mid-Year Program Review
March 22 - 25, 1993
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Office of Technology Development
Environmental Restoration and Waste Management
U. S. Department of Energy



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AMES
LABORATORY

Characterization,
Monitoring, and
Sensor Technology
Integrated Program

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PREFACE

This booklet contains summary sheets that describe FY 1993 characterization, monitoring, and sensor technology (CMST) development projects. Currently, 32 projects are funded, 22 through the OTD Characterization, Monitoring, and Sensor Technology Integrated Program (CMST-IP), 8 through the OTD Program Research and Development Announcement (PRDA) activity managed by the Morgantown Energy Technology Center (METC), and 2 through Interagency Agreements (IAGs). The summary sheet footers indicate whether a project is principally a CMST-IP or METC responsibility. More detailed information regarding these projects can be obtained by contacting the Principal Investigator. Contact information is included in each project summary sheet.

This booklet is not inclusive of those CMST projects which are funded through Integrated Demonstrations (IDs) and other Integrated Programs (IPs).

The projects are in six areas:

Expedited Site Characterization;

IS--5096

Contaminants in Soils and Groundwater;

Geophysical and Hydrogeological Measurements;

DE93 011563

Mixed Wastes in Drums, Burial Grounds, and USTs;

Remediation, D&D, and Waste Process Monitoring; and

Performance Specifications and Program Support.

Projects that have application in more than one area are listed in their primary area only. The Table of Contents provides the complete list of projects.

The Ames Laboratory CMST group, together with OTD Headquarters and METC personnel, is responsible for the CMST-IP poster session and this booklet for the 1993 RDDT&E Mid-Year Program Review. On behalf of all, we thank the Principal Investigators and their Technical Project Managers, who supplied us with the information for both.

We hope this information will link technology developers with customers to help promote appropriate technology development and, ultimately, application and adoption of improved or previously unavailable technology. Potential customers or technology appliers, we invite your comments on how to better serve your needs. Please address your comments to any one of the following.

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Characterization, Monitoring and Sensor Technology - Integrated Program

Task Description

This task provides field coordination of Characterization, Monitoring, and Sensor Technology Integrated Program (CMST-IP) activities. It involves and contributes to technology needs identification, assessment of technology requirements, capabilities, and limitations, promotion of technology integration, assessment of technology development opportunities, and program planning and implementation. The Ames Laboratory and DOE Headquarters CMST-IP teams are partners in all these activities.

Technology Needs

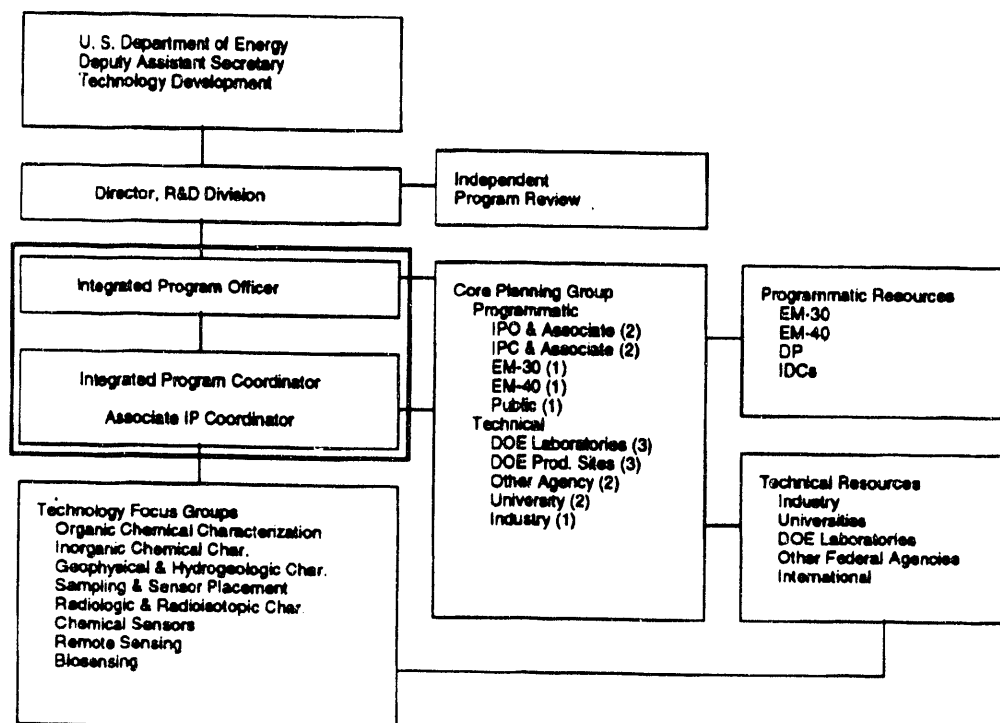
The rapidly changing and cross-cutting nature, wide diversity, and large potential cost savings to be realized from improved characterization, monitoring, and sensor technologies (CMST) mandates special treatment of those technologies for application in accomplishing the Environmental Restoration and Waste Management (EM) mission. Indeed, a CMST-IP driven by the needs of the EM program is essential for sensible and cost effective resource allocation and management.

Accomplishments

Ames Laboratory CMST-IP staff (Bill Haas, Martin Edelson, Paul Wang, and Greg Junk) contributed to CMST needs assessments, technology evaluations and technology selections in support of numerous EM-30, EM-40, and EM-50 activities throughout the DOE Complex. Examples include participation in: Characterization Technical Support Group activities for all but one of the EM-50 Integrated Demonstrations; the Characterization Working Group of the Hanford Tank Waste Remediation System (TWRS) activity; Hanford Single Shell Tank Instrumentation Program Review; Hanford Tank Vapor activities; and the EM-40 Field Area Technology Representatives (FATR) group in support of the Technology Information Exchange meetings. These activities contribute to the effective coordination and integration of CMST application and R&D/DT&E efforts and help the CMST-IP team and others stay up to date regarding EM needs and lessons learned.

Several CMST-related topical reports were distributed. These included: an overview of VOC-monitoring technologies (IS-5082); non-radiometric methods for Pu determination (IS-5085); and an evaluation of technologies for the non-destructive analysis of transuranic elements in drummed and boxed wastes (IS-5095).

A report on the Chemical Sensors Technology Development Planning Workshop (IS-5093) will be distributed in March 1993.



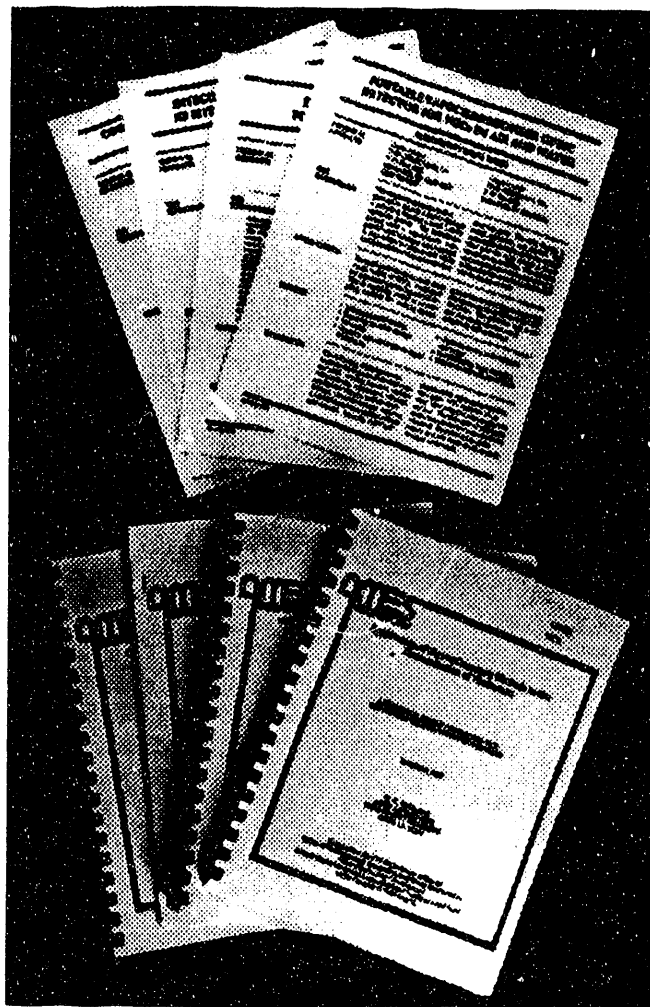
The Ames staff assembled teams of technical experts to compare validated CMST needs submitted by EM-30/-40/-50 organizations against their knowledge of existing CMST. This comparison led to the generation of CMST development needs, written primarily by the technical experts, that formed the core of the first Program Research and Development Announcement (PRDA) and CMST-IP Calls for Proposals.

The Ames team coordinated the review of over 300 technical task proposals (TTPs) submitted by DOE and other federal proposers. The proposals were evaluated by the technical experts cited above as members of the CMST-IP Technology Focus Groups. Copies of the evaluations were distributed to EM-50 Technical Program Managers for relay to the proposers from their sites. Ames Laboratory CMST-IP staff teamed with the CMST-IP Program Manager and headquarters staff in preparation and implementation of the CMST-IP R&D program for fiscal year 1993.

The technology integration action described in detail below was initiated.

Collaboration/Technology Transfer

The application of CMST to environmental restoration and waste management problems is a rapidly growing endeavor. So too, is the commercial, academic, and federal laboratory development of new and improved CMST. Through CMST-IP participation in the EM-40 Technology Information Exchange meetings, it was determined that EM-40 field personnel would appreciate receiving brief (one-page) alerts on new and emerging CMST applicable to the problems they faced. This led to the initiation of a new CMST-IP product, the "Technology Data Sheet," designed to improve communication between EM field workers and CMST providers in EM-50 and elsewhere. Over twenty Technology Data Sheets, providing quantitative descriptions of the capabilities and limitations of new and emerging CMST, have been prepared and hundreds of copies have been distributed to interested parties, both within and outside of EM.



Technology Data Sheets (TDSs) and Ames' CMST Publications

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Expedited Site Characterization

Expedited Site Characterization:

Application and Continued Development of Rapid, Focused Site Characterization Methodology for Federal Facilities

Task Description

Argonne National Laboratory has developed a methodology and procedure for remedial site characterization that has greatly decreased cost, time, and intrusive activities at Department of Interior and Department of Agriculture sites. The purpose of this project is to apply and continue to develop this approach for DOE. The continued development and transfer of this approach will result in considerable cost and time savings to DOE and will allow needed remedial actions to proceed expeditiously.

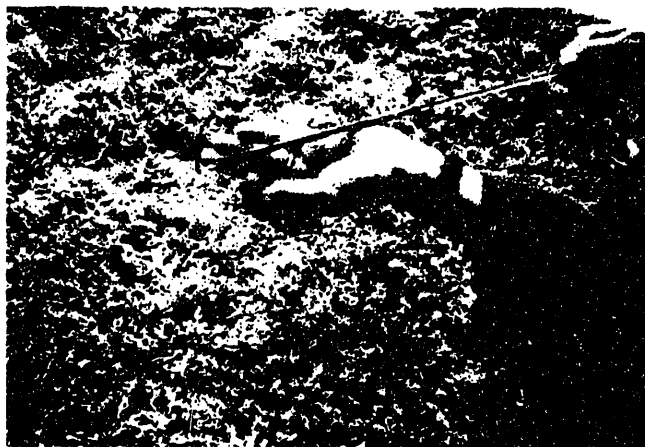
Argonne will develop and demonstrate this expedited site characterization (ESC) process at a federal facility chosen in conjunction with EM-50 staff. Prior to field demonstration of the methodology, Argonne will develop an ESC program specific to the chosen site. The program will emphasize the maximum use of nonintrusive technologies (e.g., surface geophysics, electronic cone penetrometer [ECPT], HydroPunch, vegetation sampling), field analytical procedures, and daily integration of data via expert analysis and various computer programs. Private industry and universities will be used in the demonstration as needed to provide certain services such as ECPT, auger drilling, and field analytics. Industry and DOE will be invited to the field demonstrations. The field demonstration is expected to last for two or three weeks, depending on the complexity of the site.



Using HydroPunch sampling allows delineation of the contaminant plume without installation of monitoring wells.

Technology Needs

Ongoing application of existing and emerging technologies is a vital part of this program. Results from the program are used to give guidance to technology developers whenever possible. The program emphasizes nonintrusive and minimally intrusive technologies such as surface geophysics and ECPT, respectively. A mobile field laboratory is utilized as well as different computer programs for on-site data analysis.



Surface vegetation sampling may be used to detect subsurface contamination.

Accomplishments

New program for DOE (2/93). However, this approach has been successfully applied at Department of Interior (DOI) Bureau of Land Management landfills and U.S. Dept. of Agriculture (USDA) sites contaminated with carbon tetrachloride in Nebraska and Kansas. The approach resulted in significant cost and time savings for both the DOI and USDA.

Collaboration/Technology Transfer

After successful implementation of the field demonstration, Argonne will assist EM-50 in developing a request for proposals (RFP) for industry to perform a similar ESC program. Argonne will provide guidance, assistance, and oversight to DOE throughout the technology transfer process, to ensure correct implementation of the ESC methodology. This will include assistance with the RFP, site visits and field instruction, field data review, laboratory data review, and document review.

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Innovative Directional and Position Specific Sampling Technique

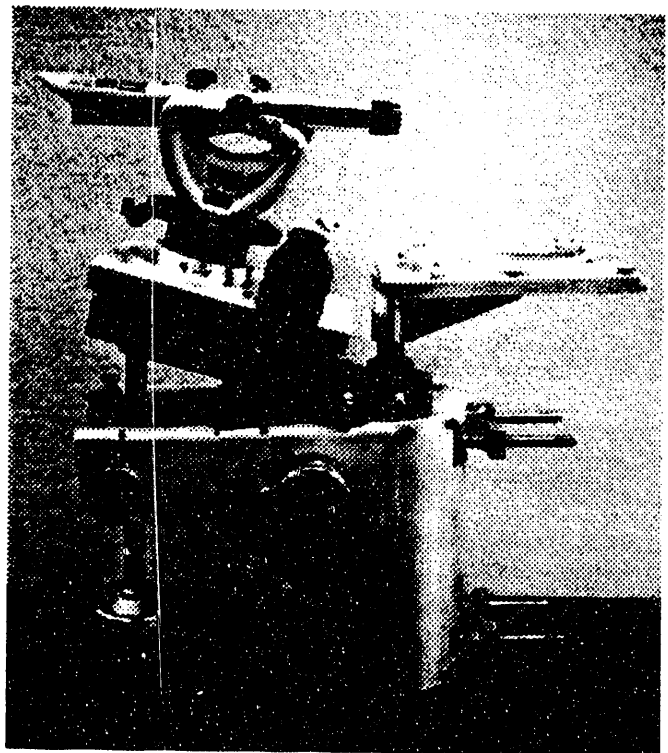
Task Description

A strain-based position location (POLO) determination system for use on penetrometers and lysimeters will provide accurate, downhole, real-time directional control of sampling devices. Conceptually, the POLO system defines the trajectory of a hole in the ground by navigating from point to point along its length and describing the shape and orientation of the centerline between each set of points. Measurement is made by first inserting an instrumented pipe section (measurement module), whose centerline corresponds to the centerline of the hole, and then making strain gauge measurements on the walls of the measurement module. The measurement module, incorporated in a penetrometer rod, advances as the penetrometer rod is pushed into the hole. The module traverses the length of the hole and measures the shape of the hole axis at successive points along the trajectory. A mapping algorithm makes use of measurements spaced at a distance which allows the shape of the hole path to be approximated by a planar radius of curvature. The technology will be applied for mapping existing well and real time mapping of penetrometer and lysimeter paths.

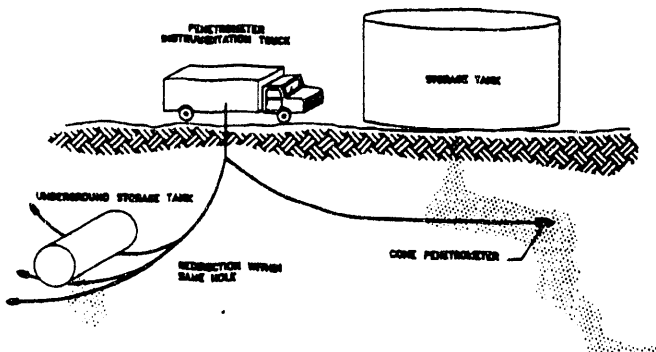
POLO is small enough to be used in real time installation of lysimeters, penetrometers or directional drilling of wells without severely limiting space needed for other instrumentation or drilling fluids during the installation process. Its immunity to nearby magnetic materials provides the additional benefit over commercially existing equipment in that it can provide position location while installing, or mapping existing installations, adjacent to and beneath underground or surface storage tanks; in, adjacent to, and beneath landfills which may include magnetic material such as steel barrels; and adjacent to and beneath

building foundations which include steel reinforcement.

The POLO system will be field tested by using a penetrometer thruster to push the module into the soil of a non-contaminated site and a wedge-shaped nose to control the direction of the penetration.



The Phase I POLO initializer provides elevation, azimuth and rotation angle of the POLO module at the beginning of a penetrometer insertion.



POLO depicted as conceptually providing position location for the directional insertion of a penetrometer for sampling beneath storage tanks. Commercialization of POLO will include applications which will dramatically enhance current environmental sampling, monitoring and remediation.

Technology Needs

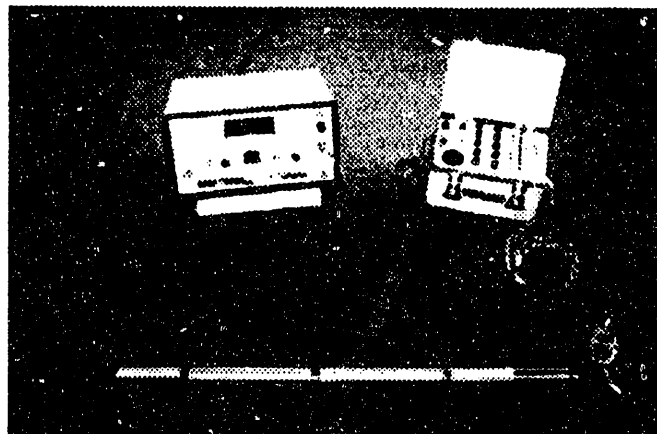
Current sampling strategies in characterizing soil and groundwater contamination often are limited by the cost of installing monitoring wells. Sampling beneath and adjacent to buildings, storage tanks, and landfills with monitoring wells is inefficient and, in some cases, current technology will not meet the need. Penetrometers and lysimeters offer the potential for increasing the number of samples that can be obtained from a site over the number available at the same cost from monitoring wells. With all of these methods, however, the alignment of the well or the device inserted into the soil is conducted on an aim and shoot basis with only broad approximations of actual location of sampling points. Unfortunately, even small errors in approximations of sampling location can have a significant impact on the interpretation of plume origins and other characteristics. The POLO system provides a new opportunity for accuracy in this field.

Accomplishments

Module construction is complete and testing is underway. Preliminary results indicate high resolution in measurement of module deflections. The mapping algorithm which integrates module deflections over the total distance traveled, to determine position location, continues to undergo evaluation and exhibits excellent performance. An initializer has been designed and constructed to provide initial POLO orientation information at the start of a penetrometer insertion, and the device has also met required accuracy capability.

Collaboration/Technology Transfer

UTD Incorporated has been issued a patent on the underlying principles of the POLO system. Commercialization of the POLO system for use in the penetrometer industry is scheduled for the spring of 1994.



Laboratory grade signal conditioning units are used in testing the deflection measurement accuracy of the POLO module.

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Direct Sampling Ion Trap Mass Spectrometry Methods for Organics in Water, Soil, and Waste

Task Description

This task is an extension of the successful demonstration at Savannah River and Portsmouth Gaseous Diffusion Plant of Direct Sampling Ion Trap Mass Spectroscopy (DSITMS) for the rapid determination of organic pollutants in water and soil. The objective of this work is to convert the demonstrations into standardized analytical methods which are acceptable to regulatory agencies and/or can be routinely deployed at multiple DOE sites. This involves developing operating conditions optimized for the intended purpose of the method, generating and statistically analyzing the data to document the performance of the method, and preparing the documentation required to solicit regulatory agency acceptance. This task also seeks to test the applicability of DSITMS to the characterization of wastes. Specifically, methods will be developed for the multicomponent screening of the 34 Volatile Organic Chemicals (VOCs) of the EPA Target Compound List (TCL) at the EPA action limit concentrations (both water and soil), and for quantitatively determining high priority organohalogen pollutants commonly encountered at DOE facilities.

DSITMS methods for the analysis of VOCs in water and soil are based on direct helium purge into the ion trap mass spectrometer. No extensive sample preparation, preconcentration, or prior chromatographic separation is required. The rapid response of this method provides a real-time visualization of the mass spectra for the compounds present in the sample. Both electron impact (EI) and chemical ionization (CI) spectra are obtained for a sample by rapidly alternating ionization conditions in the ion trap every 0.5 seconds. The CI spectra improve the detection limits for water soluble compounds including aldehydes, ketones, and alcohols and also improve the ability to differentiate compounds which have

similar EI fragmentation spectra such as alkyl aromatics. Accurate quantitative results are achieved by collecting data for 2 to 3 minutes in order to average out variations. Integrated responses for specific masses corresponding to target analytes are quantified relative to working curves generated using a series of standards.

Replicate #	Concentration in ppm		
	Field DSITMS	Lab DSITMS	GC/MS
1	29	28	27
2	30	29	28
3	31	30	31
Mean	30	29	29

Table 1. Comparison of TCE quantification in a groundwater sample by field DSITMS, laboratory DSITMS and GC/MS using an EPA approved method.

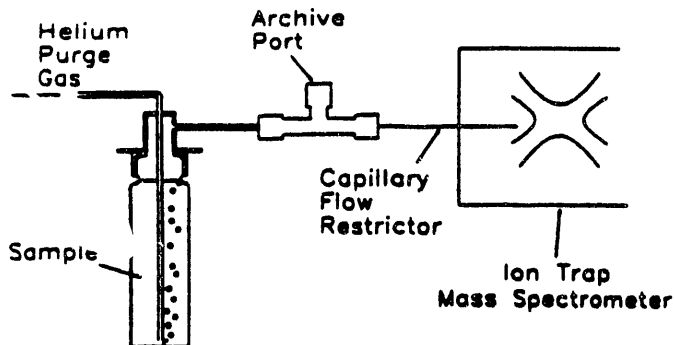


Figure 1. Direct purge sample inlet used for DSITMS analysis of volatile organic chemicals in water and soil samples.

Technology Needs

This technology is needed to reduce the cost and to increase the response time associated with organic analytical support of site characterization and remedial action monitoring activities throughout the DOE. Improved emergency response capabilities also result from this work. The technology compliments fiber optic methods for rapid analysis by being applicable to soils and to multiple component analysis.

Accomplishments

The primary accomplishments of this task have been the development of a method for DSITMS screening of the 34 EPA TCL VOCs in water and the generation of the data necessary

for submission to the regulatory agencies for their approval. The method involves direct purge of water samples using a helium flow rate of 100 mL/min. Approximately 1.5 mL/min (1.5%) of the effluent enters the mass spectrometer through the splitter/capillary restrictor while the bulk of the effluent (98.5%) is directed to a vent port where it can be collected on a sorbent cartridge for further analysis. Internal standards are also added to the sample for improved quantification accuracy and instrument performance standards are employed for quality assurance purposes. Statistical methods are applied to calibration curves in order to establish the USEPA Method Detection Limit (MDL) and the USAEC (the U.S. Army Environmental Center, formerly known as USATHAMA) Certified Reporting Limit (CRL). These limits take into account the errors introduced throughout the method and are higher than the instrument detection limits. Analytical methods for the determination of VOCs in soil are also under development. This work is more complicated due to significantly different VOC purge characteristics for various soil types. The behavior of representative VOCs in different soil types will be examined in order to establish a protocol for soil analysis.

In addition to the methods for VOCs initial work is also underway for developing methods for semivolatile compounds such as PCBs and pesticides. Preliminary data on PCBs in soil indicate that a fast screening method at or above 1 ppm should be possible.

<u>Compound</u>	<u>Ionization Mode^a</u>	<u>Conc. in ppb</u>	
		<u>MDL^b</u>	<u>CRL^c</u>
Acetone	CI	2.9	4.1
Benzene	CI	1.1	1.5
Carbon Tetrachloride	EI	0.7	0.9
Chloroform	EI	0.9	1.3
1,2-Dichloroethane	EI	1.4	1.9
Methyl-Ethyl Ketone	CI	1.9	2.6
Perchloroethylene	EI	0.5	0.7
Toluene	CI	0.6	0.9
Trichloroethylene	EI	0.7	0.9
Xylenes	CI	1.1	1.5

^a Ionization mode used for optimum detection of compound

^b Method Detection Limit as defined by U.S. Environmental Protection Agency

^c Certified Reporting Limit as defined by U.S. Army Environmental Center

Table 2. Example MDLs and CRLs for VOCs in water by direct purge into the ion trap mass spectrometer.

Collaboration/Technology Transfer

Technology transfer within DOE will occur through the OTD FAST committee and through ongoing contacts with DOE environmental services laboratories. Contacts with environmental remediation and waste management offices will be maintained through the ORNL OTD Program Office. Inter-agency transfer will occur as a result of briefings and progress reports to the DOE/AEC, DOD/PMRMA, USEPA/OSW, and NIH/NCI. Scientific technology transfer occurs through participation in scientific symposia and open literature publications. Where resources are available, ORNL staff will assist other DOE laboratories in methods development.

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Direct Sampling Ion Trap Mass Spectrometry Instrumentation for Organics in Water, Soil, and Waste

Task Description

This task is part of an overall program, in cooperation with the US Army Environmental Center (USAEC, formerly U.S. Army Toxic and Hazardous Materials Agency), focused on the development, demonstration, and deployment of faster and less expensive methods for environmental analysis. The technology utilized for this project is Direct Sampling Ion Trap Mass Spectroscopy (DSITMS) which has shown significant promise for rapid analysis for environmental samples, both in the lab and in the field. This work builds upon USAEC sponsored observations that DSITMS is capable of the quantitative determination of EPA-targeted volatile organic chemicals (VOC's) in water and soil at regulated levels in less than five minutes. This work has resulted in the development of laboratory and field DSITMS instruments and a highly versatile direct sampling interface for the analysis of air, water and soil samples. Field testing of the transportable instrument has been performed at the Savannah River Integrated Demonstration Site and at the Portsmouth Gaseous Diffusion Plant.

Research efforts under this TTP focus primarily on the devel-



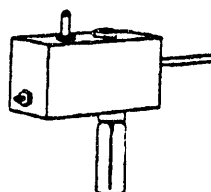
Figure 1. Field transportable ion trap mass spectrometer.

opment and refinement of the hardware and software needed to address specific analytical challenges related to the analysis of organic pollutants at DOE sites. This includes improvement of both the lab and field DSITMS instruments to provide better multicomponent analysis, greater sensitivity for ultra-trace pollutants and increased specificity for target compound analysis in the presence of interferences. In addition, the field instrument will be modified to enable operation in a moving vehicle and will undergo further improvements in terms of size, weight and power reduction. Existing sample introduction hardware will be made more reliable and new sample introduction modules will be developed for custom applications as needed. Software modifications will be aimed at improved instrument control, operator convenience and real-time data interpretation and quantification.

THERMAL DESORBER



WATER/SOIL PURGE



ARCHIVE PORT



TRANSFER LINE

REAL-TIME AIR SAMPLER

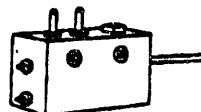


Figure 2. DSITMS interface and sampling modules.

Technology Needs

Rapid analytical technology is needed to reduce the cost and to improve the response time associated with site characterization and remedial action monitoring throughout the DOE. This task extends the benefits of direct sampling ion trap mass

spectrometry to field applications, quantitative analyses and the determination of priority pollutants.

Accomplishments

Work funded under this and related TTPs has resulted in the development and deployment of prototype Direct Sampling Ion Trap Mass Spectrometers for laboratory and field applications. These instruments have been based on modified versions of commercially available (Finnigan-MAT) ion trap mass spectrometers in order to deploy, test and evaluate the general capabilities of the technology in a timely manner. The current field instrument requires approximately 500 watts of power, weighs 120 pounds and is approximately 8 ft³ in size. It is operated using a Compaq 486-C portable computer and is mounted on a shock-absorbing base in order to improve transportability.

Field testing of the DSITMS technology at the Savannah River Integrated Demonstration Site and Portsmouth Gaseous Diffusion Plant have in general been very successful with respect to demonstrating the sensitivity, analytical speed, and versatility of the DSITMS technology. These tests have primarily dealt with the analysis of trichloroethylene (TCE), perchloroethylene (PCE) and related chlorinated solvents which are the main

volatile organic contaminants at these sites. Accurate quantification of TCE and PCE has been demonstrated in groundwater samples at ppb and ppm levels with a sample throughput of up to 24 samples per hour. Further, the real-time air monitoring capabilities have been demonstrated for measuring TCE and PCE in soil gas and the headspace of monitoring wells at concentrations ranging from low ppbv to low ppmv.

As part of the field trials, the ruggedness of the instrument with respect to transport in a vehicle and operation under extreme environmental conditions has been demonstrated. Improvements in the design of the field instrument including reductions in power consumption, lighter weight vacuum pumps, and integration of external components and power supplies into the main instrument housing have been largely a direct result of lessons learned during the field deployment tests.

Collaboration/Technology Transfer

Technology transfer is expected to occur through the OTD FAST committee, the ORNL/OTD Program Office, and collaborating Dept. of Army program offices. Special attention will be given to contracts with the EPA/OSW, the DOE/OTD Integrated Demonstration Program, and the manufacturer of ion trap instrumentation. Where resources are available, ORNL staff will assist other DOE laboratories in acquiring the necessary instrumentation and will train operating staff.

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Sample No.	Conc. in ppb	
	EPA Method	Field Ion Trap
MHM-1	7088	> 10,000
MHT-1D	360	274
MHV-6	666	738
MHV-7	4178	9000
MHT-10D	1180	1273
MHT-14D	160	102
MHT-1C	351	324
MHT-3C	435	461
MHT-4C	63	64
MHT-5C	254	257
MHT-6C	159	167
MHT-8D	-	1366
MHT-13D	-	305

Table 1. Comparison of results for TCE in groundwater at Savannah River Integrated Demonstration Site.

Field-Deployable VOC Analyzer: On-Site, Real-Time VOC Analyzer Systems for Air, Soil, and Water

Task Description

This project is developing field-deployable analyzer systems for environmental air, water, and soil samples based on infrared spectroscopy. The systems will provide in-the-field, real-time analysis for environmental remediation and monitoring. Present off-site analysis methods entail much higher costs, delays, and environmental risks. The analyzers will consist of hand-carried modules that can be mated together to produce systems tailored to the specific needs of a site.

Most of the systems will use one of two commercially available infrared photoacoustic gas-analysis modules. One of these modules uses filters to monitor infrared absorption in narrow wavelength ranges and thereby measure up to five target gases (plus water vapor), and the other contains a complete Fourier-transform spectrometer to provide a full mid-infrared spectrum. The full-spectrum device is the more versatile and can identify unknown gases, but the filter unit has somewhat better sensitivity. Figure 1 shows how the various modules combine to form complete analyzer systems. A laptop computer can attach to the commercial units to augment control and data storage, analysis, and display capabilities. Sampling and sample preparation modules developed in this project will also attach to the commercial units. These will provide automatic gas sampling from

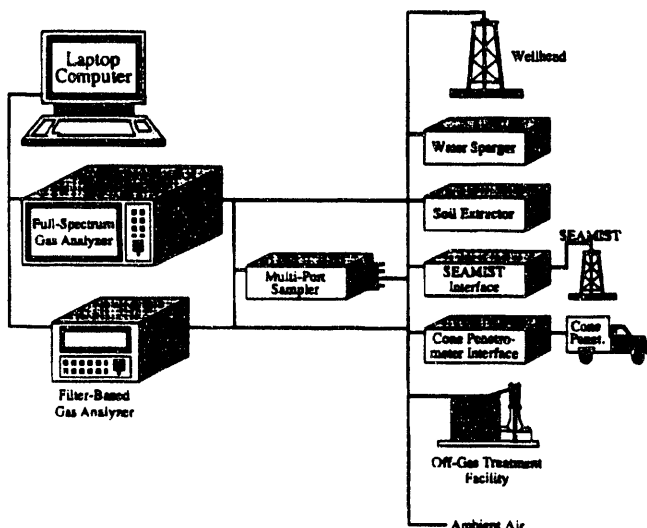


Figure 1. The modular structure of the analyzer systems. A filter-based or a full-spectrum gas analyzer is the heart of each system. A laptop computer augments control and data handling. Any of a variety of sampling or sample preparation modules can be attached to the gas analyzers either directly or in parallel through a multi-port sampler.

several points, preparation of soil and water samples for VOC measurement by the gas analyzers, or interfacing to deployment and retrieval systems such as SEAMIST and cone penetrometers. Demonstrations of the analyzer systems began in 1992.

Simplified analyzers for measuring single contaminants (or perhaps two) are also being developed. These single-contaminant analyzers will also make photoacoustic-based measurements of infrared absorption by gases, but they will be considerably simpler in design than the commercial units described above. These single-contaminant analyzers will provide inexpensive, long-term monitoring of the target vapor during extended remediation efforts.

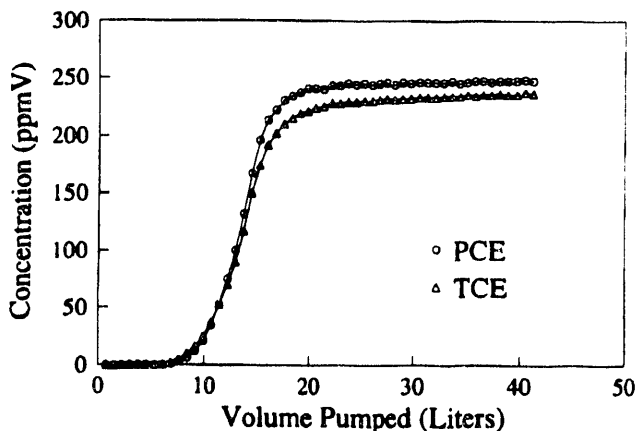


Figure 2. Trichloroethylene (TCE) and perchloroethylene (PCE) concentrations determined simultaneously with the filter-based gas analyzer attached to the head of a monitoring well at Savannah River while air from the well was slowly purged out.

Technology Needs

VOCs are the most widespread chemical contaminants in the DOE complex. The versatility inherent in the modular structure of the VOC analyzers gives them a very wide range of applicability. The proper combination of modules will allow the measurement of VOCs in almost all environments. The instrumentation can be used to monitor worker exposure to ambient-air VOC releases, to determine soil-gas contamination, to monitor off-gas treatment operation, to provide real-time guidance for site characterization tasks, or to determine depth-specific contamination levels in monitoring wells.

Accomplishments

Versions of the analyzer system are now ready for demonstration. The base analyzer system was demonstrated in 1992 for analyzing monitoring wells at Savannah River (Non-Arid VOC ID site). Figure 2 shows the contaminant concentrations measured at one wellhead while the well was being slowly pumped out. All of the measurements shown were made in less than two hours. The sampling and measurement time for gases is generally less than 2 minutes, irrespective of the number of contaminants to be determined. Analysis of water and soil takes longer (5 to 20 minutes) because of the time required to extract the VOCs, but by connecting several sample preparation modules in parallel through the multi-port sampler, the time required per sample can be greatly reduced.

The unaided commercial analyzers have limits of detection for most VOCs of from 100 ppbV to 10 ppmV in air. Attachment of the proper sampling device can extend these limits of detection to whatever level is required. Figure 3 shows a laboratory test of the ability of the technology to determine trichloroethylene contamination in water. The most dilute sample shown (10 ppbV, or 15 ppbW) was determined with a $\pm 5\%$ precision, thus it is not the final limit of the technology.

The systems consist of hand-carried modules and require no infrastructure other than electrical power (A battery pack for the simpler systems is in development.) and a covering to keep off precipitation and direct sun, thus they can be used in many locations where bulkier or more delicate equipment cannot. The systems can be set up and ready for use in under 10 minutes, thus the systems can be used efficiently to make measurements at dispersed locations.

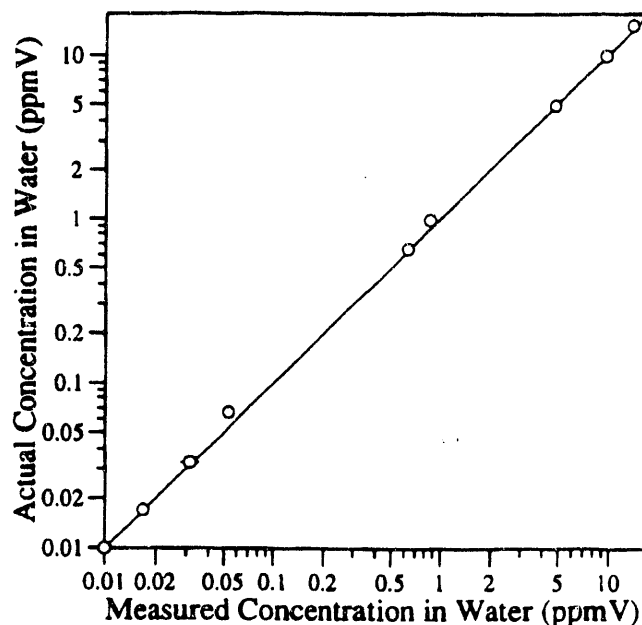


Figure 3. Laboratory measurements of trichloroethylene in water determined with a water-sparging sampler attached to the filter-based gas analyzer.

For more information, please contact:

Collaboration/Technology Transfer

A close working relationship has been established with Bruel and Kjaer (Marlborough, MA), which manufactures the commercial photoacoustic gas analyzers used in the analyzer systems. Ames Laboratory has been acting as a beta test site for the full-spectrum analyzer, which was released to the market earlier this month. The simplified, single-contaminant analyzer being developed in the project is scheduled for transfer to the private sector in 1995. The project staff is interested in collaborating with potential end-users of the technology.

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Field-Usable Portable Analyzer for Chlorinated Organic Compounds

Task Description

A solid-state sensor based chlorinated organic (R-Cl) monitor has been shown to be highly selective for the detection of hydrogen chloride and chlorinated organic compounds. The sensor consists of two platinum electrodes encapsulated in a rare earth salt. The conductivity of the rare earth salt, which corresponds to the level of chlorinated solvent in the vapor phase, is measured across the two electrodes. The sensor can also be used to detect brominated compounds, but produces a weaker response, and still weaker responses to fluorinated and iodinated compounds. The advantage of the R-Cl monitor is its selectivity since it is not sensitive to hydrocarbons, oxygenated compounds, nitrogenated compounds, or elemental chlorine.

The sensor has several other desirable properties that render it suitable for immediate application to environmental and industrial pollutant-monitoring problems, including simple operation, ease of maintenance, and low cost. This sensor has been fitted into an existing instrument system, and is currently in operational use at the DOE Hanford site and several other non-DOE facilities. Based on these operational experiences, design improvements have been identified that will significantly improve performance. The monitor system will be modified to extend the dynamic analytical range for carbon tetrachloride, trichloroethylene (TCE), and perchloroethylene (PCE); and to produce a user friendly system for *in-situ*, real-time measurements at DOE sites. These modifications follow logically from the field performance

of this most promising real-time sensor. Field-deployable prototype designs are expected within 18 months of development.

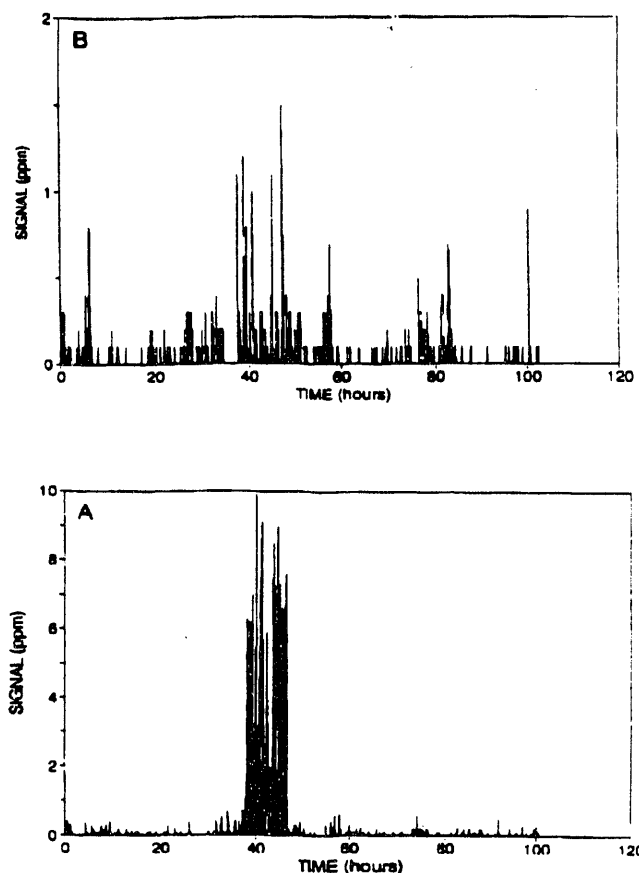


Figure 2. Temporal concentration profiles of chlorinated organic vapors obtained through continuous "around the clock" monitoring with the current version of the chlorinated vapor monitor at two locations around a site with subsurface soil contamination. A) Near the contaminated site. B) Approximately 1000m away. Note the difference in sensitivity. The sudden increase in vapor levels was attributed to a sudden decrease in barometric pressure which effectively provided a natural vacuum extraction of subsurface contamination. The remote monitor detects lower vapor levels with lag time compared to the proximal monitor.

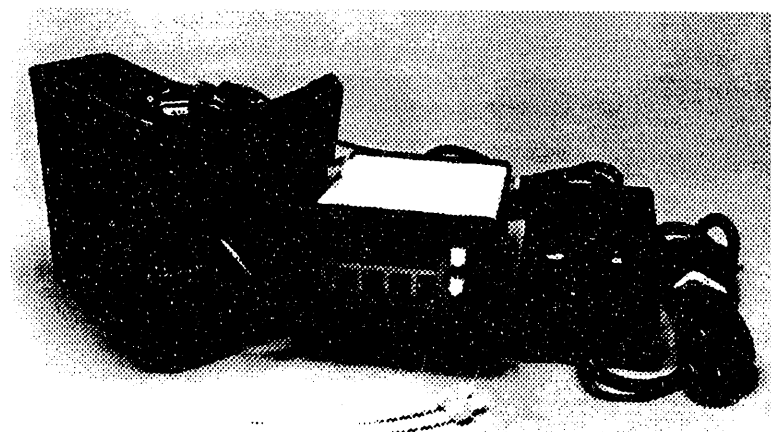


Figure 1. Photograph of the current version of the chlorinated vapor monitor. This instrument is extensively deployed in clean up operations at the Westinghouse Hanford facility. Applications include perimeter monitoring of a contaminated zone and process control of vacuum extraction operations.

Technology Needs

Chlorinated hydrocarbon contamination of soil and ground water is a major problem at numerous DOE sites, including the Hanford Reservation in Richland, WA and the Savannah River Plant in Aiken, SC. Both facilities have ongoing remediation programs which include the removal of R-Cl contamination from subsurface soils and water supplies. During the remediation process, chemical monitoring is necessary to ensure worker safety, to verify environmental compliance of emissions, and to track the actual cleanup process. Until recently, the only available technology to selectively monitor chlorinated organics was a gas chromatograph (GC) equipped with an electron capture detector or GC/MS. In addition to being expensive, gas chromatography requires grab samples and does not provide real time monitoring capability.

Accomplishments

The preliminary specifications and design features of the instrument have been established. Three modes of operation will be available: 1) Search mode, which is designed to provide up to ten quick, manually initiated analytical measurements for chlorinated vapors, 2) Evaluate Mode, which is a manually initiated mode of operation designed to provide up to four highly accurate analytical measurements, and 3) Monitor Mode, which provides automatic repetitive and highly accurate analytical measurements. Table 1 summarizes the operating specifications for the various modes of operation. Direct display and data logging capabilities are available. The instrument will weigh less than 15

pounds and have dimensions of 6.3 * 3.9 * 10.2 inches not including the handle. Components of the instrument are currently being fabricated, and a prototype is planned for field testing in June.

At a later date, specialized samplers will be tested. Custom samplers are being designed for multiple requirements including direct sampling for general operation, fixed dilution of high level vapors, preconcentration of low level vapors, and permeation sampling from condensed media.

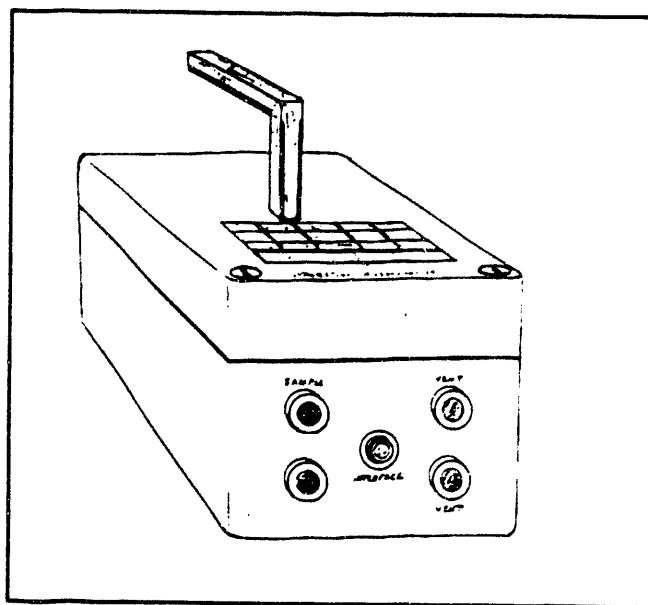


Figure 3. Line drawing of the advanced chlorinated vapor monitor currently being designed and fabricated. A test prototype of the basic instrument will be available by June.

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Table 1: Proposed operating specifications for the Chlorinated Vapor Monitor.

Parameter	Search Mode	Evaluate Mode	Monitor Mode
Range (ppm)	0.5 to 25	0.5 to 25	0.5 to 25
Detection Limit (ppm)	0.2	0.2	0.2
Cycle Time			
Measurement	1 min	5 min	5 min
Autozero	5 min	10 min	10 min
Accuracy (% of reading)			
>5 to 25 ppm	± 20%	± 10%	± 10%
>1 to 5 ppm	± 40%	± 15%	± 15%
>0.5 to 1 ppm	indication	± 20%	± 20%
>0.2 to 0.5 ppm	indication	indication	indication

Improvements in Inductively Coupled Plasma Mass Spectrometry

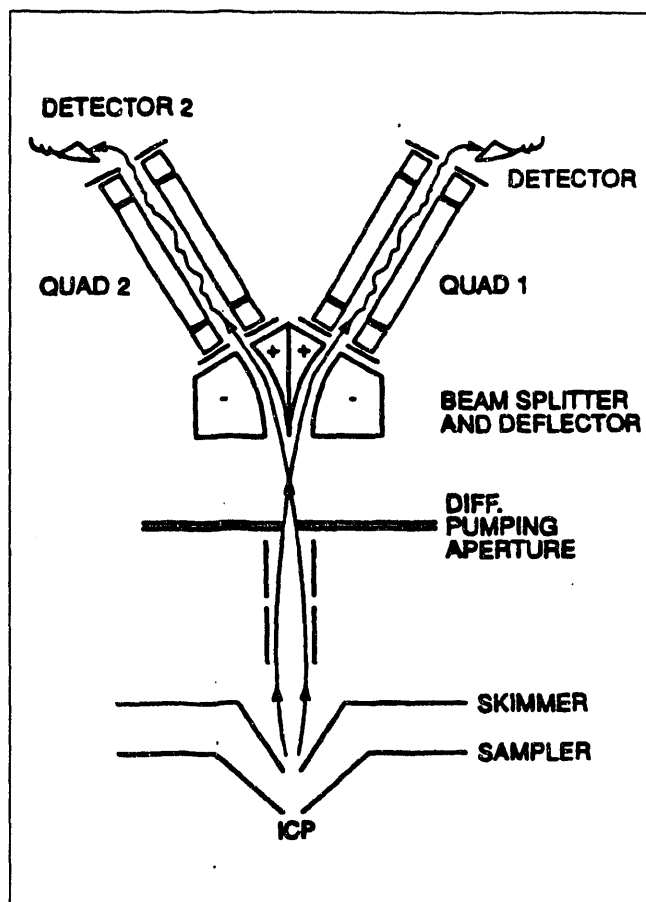
Task Description

Inductively coupled plasma - mass spectrometry (ICP-MS), which was invented by this project's principal investigator, is a very sensitive method for elemental and isotopic analysis. It is expected to have major applications for monitoring both stable and radioactive elements in waste remediation. However, the precision of ICP-MS is not particularly good (RSD of $\pm 5\%$ for elemental concentrations, $\pm 1\%$ for isotope ratios). The goal of this project is to improve precision and accuracy by at least one order of magnitude by constructing a new type of ICP-MS device. The ion beam from the ICP will be split into two parts. Each part will be directed into a separate mass analyzer set to monitor a different isotope. With this "double-beam" approach, isotopes will be detected simultaneously rather than sequentially as with present devices.

The anticipated benefits include: 1) improved accuracy for determining ultratrace elements; and 2) fast isotopic analysis at high precision and sensitivity with minimal sample preparation. Improvements in precision and accuracy for laser ablation ICP-MS will eliminate the costs, time delay and opportunities for contaminating the sample incurred by the usual need to dissolve the sample. These capabilities will improve the accuracy and speed with which stable or long-lived radioactive isotopes can be monitored, which is necessary for proper storage or disposal of toxic or harmful elements. In addition, this instrumentation should facilitate several new applications that lack suitable analytical methodology, including measurement of elemental speciation and tracer experiments with stable isotopes in biological and environmental systems.

A prototype instrument is presently being assembled for these studies. The instrument is being constructed with rugged, reliable components so that it can be readily transported to actual waste sites, if desired.

determination of long-lived radionuclides at ultratrace levels or for determining elemental speciation. Direct analysis of solids without prior dissolution is also an important capability. The use of laser ablation in conjunction with remote sampling technologies could make the assessment of 'hot' sites safer. This project will improve the capabilities of ICP-MS for all of these important tasks.



Schematic diagram of a double-beam ICP-MS device.

Technology Needs

Rapid, accurate measurement of elemental concentration and isotopic abundance is obviously necessary for waste remediation. In particular, there are no general methods for fast

Accomplishments

- Prototype instrument assembled and tested.
- Ions observed with single quadrupole oriented on-axis. The usual background ions and metal ions were detected.
- Laser ablation system installed.

Collaboration/Technology Transfer

Laboratories and industries involved with the handling and characterization of nuclear materials are the potential customers that will make best use of this new technology. The laser ablation techniques should be especially applicable in the analysis of soils at hazardous waste sites containing nuclear materials.

The work is being performed cooperatively with an instrument manufacturer (VG Elemental) so that the improvements realized can become available to analysts.

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Direct Measurement of ^{90}Sr in Surface Soils

Task Description

A real-time analyzer for measurement of ^{90}Sr in surface soils could provide a tremendous savings in nuclear waste clean-up efforts. The long-lived radionuclide (29 year half-life) is perhaps the fission product of greatest concern with regard to site remediation, but its analysis is the most difficult, time-consuming and expensive. The purpose of this project is to develop, calibrate, and field demonstrate a real-time ^{90}Sr analyzer for direct measurements in surface soils, on conveyor belts, or other locations. The technology will employ a sensor composed of multiple layers of organic fiber scintillators (each ≤ 1 mm thick) which will selectively observe the highly energetic beta particles of the ^{90}Y daughter (2.28 MeV maximum) in the presence of other long-lived fission and activation products and gamma decay radionuclides (see Figures 1 and 2). The lightweight, hand-portable sensor will employ coincidence and anti-coincidence counting techniques in order to provide the required selectivity for discrimination against other anthropogenic and natural radionuclides and cosmic-ray radiations.

Technology Needs

Current methods for the measurement of ^{90}Sr in soils require the collection of myriad samples followed by expensive, laborious, and time-consuming radiochemical separations and subsequent beta counting. Sample turnaround often takes weeks to months when an outside laboratory is contracted to perform the analyses. Consequently, to employ such techniques for large area site clean-up would be prohibitive from both a cost and time standpoint. The real-time beta sensor technology which is proposed has already proven effective for the rapid analysis of ^{238}U (via the 2.29 MeV beta from its equilibrium progeny, $^{234\text{m}}\text{Pa}$), and it will provide invaluable support toward the expeditious characterization of ^{90}Sr contamination of all DOE sites requiring remedial action.

Accomplishments

The existing prototype sensor was successfully demonstrated at the Fernald site (Cincinnati, OH) for the quantification of ^{238}U in surface soils over a range of ≤ 35 pCi/g to > 3000 pCi/g (preliminary investigations indicate that the current detector is capable of quantifying strontium activities as low as 20 pCi/g). This unit is approximately 1.5 m long, 20 cm wide and 8 cm tall, and incorporates a large (i.e., 1500-2000 cm^2), and light-tight beta-sensitive region that measures about 1 m long and extends to the full width of the unit. A 10 meter power supply/signal transmission cable connects the detector unit to a rack-mounted process module that triggers a bank of scalars, which then indicate the total number of counts in each layer as well as the number of associated inter-layer coincidences. The unit was set directly on the ground (grassy areas were cropped prior to surface characterization), and 100 second counts were obtained at each sample location. Relative errors were generally found to be around 10-15%, and somewhat higher as activity levels dropped below 35 pCi/g.

Current and future efforts will concentrate on those modifications which will be necessary for the reduction of background interferences (primarily due to gamma-rays and cosmic-induced particles), thereby enhancing sensor precision, decreasing total

Real-time Sr-90 Analyzer

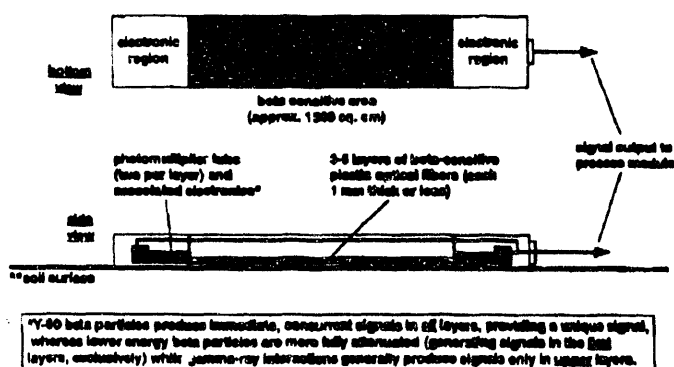


Figure 1. Schematic representation of the real-time ^{90}Sr analyzer. The sensor is set directly upon the ground (or slightly above) and observes the beta radiation emanating from strontium decay in the upper 3 - 5 mm of the contaminated soil. This unit may also be positioned above a conveyor belt for monitoring excavated or "washed" soils, or mounted to a roving vehicle for large-scale site characterization (see text for sensor dimensions).

This novel technology will be directly applicable to many existing nuclear waste and soil contamination problems throughout the industrial and DOE complex as a means of establishing remedial activities and verifying clean-up operations. As such, a considerable effort will be directed toward transferring this information to the scientific community and private sector via open literature publications and government- or industry-sponsored conference presentations. In terms of commercialization, PNL has applied for a technology patent for the beta sensor, and the Bicron Corporation (Newbury, OH) has been contracted as a possible candidate for mass production of the unit in the near future.



Figure 2. Detailed diagram of the interactions occurring within the sensor. The energetic ^{90}Sr - ^{90}Y beta particles produce coincident pulses in the bottom 3 - 4 layers to provide a unique signature, while lower energy betas generate signals in layers 1 and 2 only. Most gamma interactions lead to Compton electron pulses in layers excluding the first layer, and airborne radon daughters will trigger only the uppermost layers. Coincidence and anti-coincidence circuitry serve to mitigate such contributions, as well as those originating from cosmic-induced particles.

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Road Transportable Analytical Laboratory (RTAL) System

Task Description

The Department of Energy (DOE) Road Transportable Analytical Laboratory System builds upon ECO's successful development of road and air transportable laboratory systems and operational control centers for use in areas contaminated with chemical and biological warfare agents as well as conventional environmental contaminants. This family of Transportable Emergency Response Monitoring Modules (TERMM™) are used by the EPA, DOD, and other government agencies. These laboratories and operational control centers were designed for rapid-deployment during critical operations in seriously contaminated or remote areas which require sophisticated analytical capabilities without any external support. The TERMM™ modular laboratories and operational control centers provide the protection and support necessary to meet these stringent requirements.

The current effort addresses the unique requirements of the DOE for a road transportable analytical laboratory system capable of analyzing for a wide variety of radioactive and hazardous contaminants in soil, groundwater, and surface water. This laboratory system will be designed to provide the field and laboratory equipment necessary to detect and quantify radionuclides, organics, heavy metals and other inorganics, and high explosive materials. The RTAL system will consist of a set of individual laboratory modules which can be deployed independently or as an interconnected group to meet each DOE site's specific needs. System modules will include: (a) Field Analytical Instrument Laboratory; (b) Radiochemistry Laboratory, (c) Chemical Analysis Laboratory; (d) Biomonitoring Laboratory, (e) Robotic Sampler/Monitor Base Station; (f) Decontamination Trailer, (g) Operations Control Center, and, (h) Protected Living Quarters. Each module will provide full protection for operators against radioactivity and conventional environmental contaminants. Robotics, both in the laboratory and in the field, will be incorporated to maximize the efficiency of the human operators.

Technology Needs

The use of a road transportable, fully independent, highly reliable laboratory system will save the DOE significant time and money by providing rapid analytical guidance during site characterization and remediation monitoring activities, and by eliminating costly, paperwork-intensive and time-consuming shipment of samples to central laboratories. The integrated laboratory system will be able to provide a full range of accurate analyses within 16 hours. At the same time, the system will provide full protection for the operating personnel and sensitive analytical equipment against the hazards encountered at DOE sites.

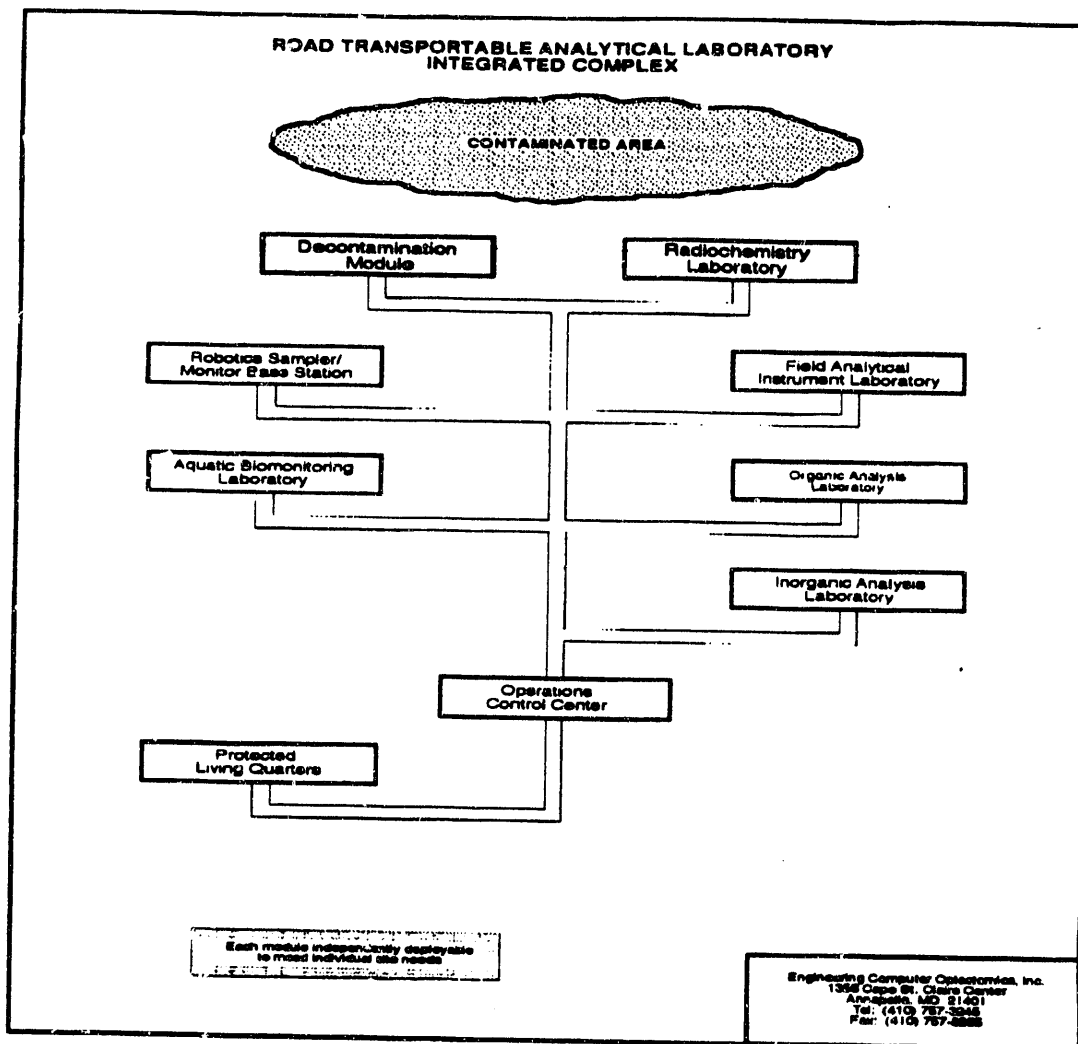
Accomplishments

The RTAL project was initiated in September 1992. Although still early in the project, the following accomplishments have been achieved:

- Completion of NEPA impacts analysis
- Completion of assessment of operational and construction requirements for the RTAL and its component modules
- Initiation of development of alternative designs for the integrated RTAL system and each component module

The development of alternative designs is progressing rapidly toward the development of a comprehensive design for the full RTAL system. This comprehensive design will incorporate the operational and construction requirements necessary to accomplish the RTAL's mission of accelerating and lowering the cost of characterization and remediation at contaminated sites across the DOE complex.

Road Transportable Analytical Laboratory (RTAL) System



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In Situ Secondary Ion Mass Spectrometry (SIMS) Analysis: Development and Evaluation of a Transportable Ion Trap SIMS Instrument for the Direct Analysis of Low Volatility Organic Compounds

Task Description

This program will develop a laboratory-based, transportable instrument that combines new SIMS technology developed at the INEL with an ion trap mass spectrometer. This new type of instrument will have the capability to analyze low volatility or non-volatile organic materials such as chelating agents, complexing agents and their metal complexes. It is anticipated that this instrument will be able to function on-site, require no sample preparation, produce no laboratory waste, and will be cost effective based on rapid analysis time, and moderate instrument cost.

Complexing agents and chelating agents contaminated with uranium (U) and plutonium (Pu) were disposed of together at DOE burial sites. U and Pu are difficult to detect because they have low alpha activity, and their only photon emission is weak X-rays; hence analyses are time consuming and expensive. The detection of complexing and chelating agents may be a useful indication of the possible presence of U and Pu, with substantially reduced cost and effort compared to the detection of Pu itself.

Newly-developed SIMS technology has been shown to be effective for the analysis of organophosphorus compounds adsorbed onto a variety of surfaces. The approach for the in situ SIMS program is to combine this technology with an ion trap (IT) mass analyzer for the analysis of materials such as low volatility organics and metal complexes that have been deposited onto the surfaces of samples such as soil, rocks, concrete, asphalt, plastic, and wood. The program will require analytical scoping studies, and ion optical modeling of a SIMS source with an IT. If the results of these studies are promising, then an IT SIMS will be constructed and tested in the laboratory. If these tests are positive, a transportable IT SIMS will be

constructed and tested.

Progress to date has shown that tributyl phosphate (TBP) can be detected on the surfaces of small rocks using an existing, laboratory-based, quadrupole SIMS instrument. TBP-sodium ions have been observed in other experiments, which provides encouragement that other metal ion species may be observable. Further, experiments demonstrated that SIMS is compatible with the instrument pressures used in an IT.

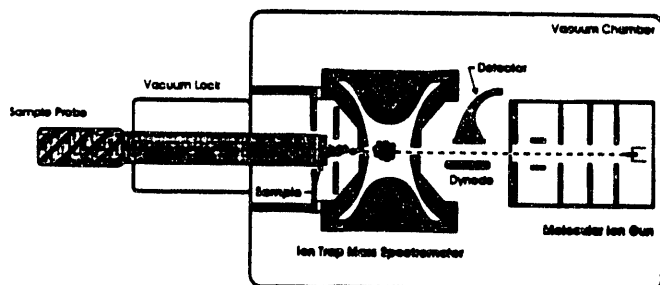
It is anticipated that the development of the IT technology will enable the analyses of samples that have heretofore been problematic, for analytes including low volatility organic complexing and chelating agents and their metal complexes. The end goal is to be able to perform these analyses rapidly, on-site, and without sample preparation or analytical waste generation. These attributes will facilitate activities such as definition of plume boundaries, and analysis of exhumed samples like rocks and waste containers.

Technology Needs

There is currently no rapid screening technique for the presence of plutonium. The lack of a screening technique is a problem because direct methods for plutonium analysis are difficult and expensive. A method that would detect tributyl phosphate (TBP) might ameliorate this problem, because TBP and plutonium were often buried together.

The need for SIMS characterization is broad, because there are numerous sites where combinations of alkyl phosphates, chelating agents, radionuclides and/or toxic metals were disposed of. Some well known examples of these sites are:

- 300 Area Ponds and Trenches at Hanford
- Radioactive Waste Management Complex Pad A and Test Reactor Area Disposal Well at the Idaho National Engineering Laboratory
- the S-2 Pond, Bear Creek Burial Ground, and Low-Level Burial Ground at Oak Ridge Y-12
- Area L (TA-54) at the Los Alamos National Laboratory
- TNX Seepage Basin at Savannah River



Schematic diagram of a quadrupole SIMS instrument

Accomplishments

SIMS technology has been used for the detection of 250 ng of TBP on the surface of basalt and sandstone. Excellent signal-to-noise was observed in these experiments and the minimum detectable limit for TBP on these minerals was estimated at 10 ng. Sample handling consisted of merely attaching a small sandstone or basalt rock to a target using double-sided tape.

SIMS was used to observe contamination of basalt by gas-phase TBP. If TBP and basalt were being handled at the same time in the laboratory (but the basalt was not spiked with TBP), then subsequent analyses of the basalt revealed TBP on the surface. These results are interpreted in terms of unusual affinity of basalt for TBP, and a sub-nanogram TBP detection limit for SIMS.

EDTA is difficult to detect because it strongly binds to mineral surfaces. However, EDTA detection on rock surfaces was demonstrated using SIMS. The success of this detection depends on the acidity of the EDTA solution that was applied to the rock. In those cases where the EDTA spiking solution was acidified, abundant ions originating from the center of the molecule could be observed: $m/z\ 61^+$ is a particularly diagnostic ion, because it corresponds to protonated ethylene diamine.



Photograph of a quadrupole SIMS

Collaboration/Technology Transfer

A manufacturer for fieldable instruments is being sought for technology transfer subsequent to demonstration of the ion trap SIMS. Potential sites for DT&E of the fieldable instrument are the Buried Waste Integrated Demonstration (Idaho), and the Arid Soils VOC Integrated Demonstration (Hanford). Collaboration has been initiated with both of these sites.

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Contaminants in Soil and Groundwater

Spectroelectrochemical Sensors for DOE Site Characterization

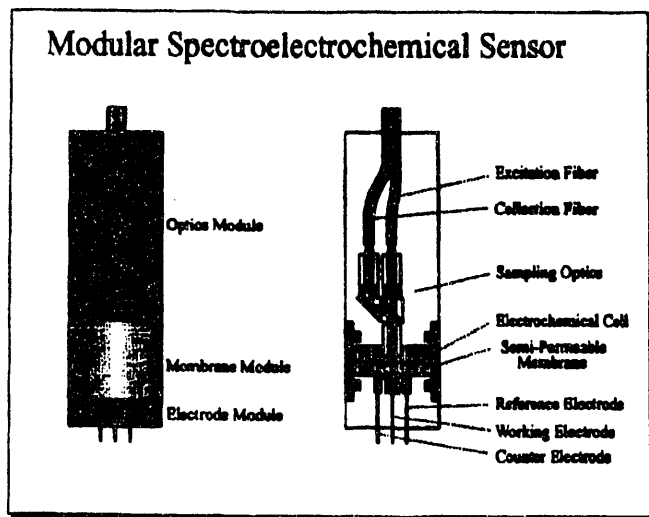
Task Description

This work will develop new, cost-saving, spectroelectrochemical technologies for the *in situ* detection of subsurface contaminants at DOE sites. The combination of electrochemistry with surface-enhanced Raman and fluorescence spectroscopy will provide a sensitive means to monitor chlorinated hydrocarbon solvents, light aromatics, and other ubiquitous contaminants in subsurface vapor and liquid phases. Modular fiber-optic spectroelectrochemical probes will be developed where the electrode in the probe will serve several important functions, including: generating reagents that react with select analytes to produce fluorescent or Raman-active products; adsorbing and concentrating reaction products from solution for greater sensitivity; and, providing a surface that enhances Raman spectroscopic signals up to a million-fold. The probes will be coupled with truly portable (< 25 lbs.) laser spectrometers capable of making Raman and fluorescence measure-

ments in real time and field tested on the Oak Ridge reservation. Demonstration of the new sensors is planned for the Savannah River site and other applicable locations.

Technology Needs

There is a need at Savannah River and most other DOE sites for *in situ* sensors that detect volatile organic contaminants in soil gas and groundwater. New instruments that can be used in the field and that respond rapidly can greatly reduce characterization costs in terms of both time and money and provide critical feedback information during remediation. This task addresses some of the most ubiquitous monitoring needs at DOE sites, including chlorinated hydrocarbon solvents and light aromatics which are used as solvents or found as fuel components.

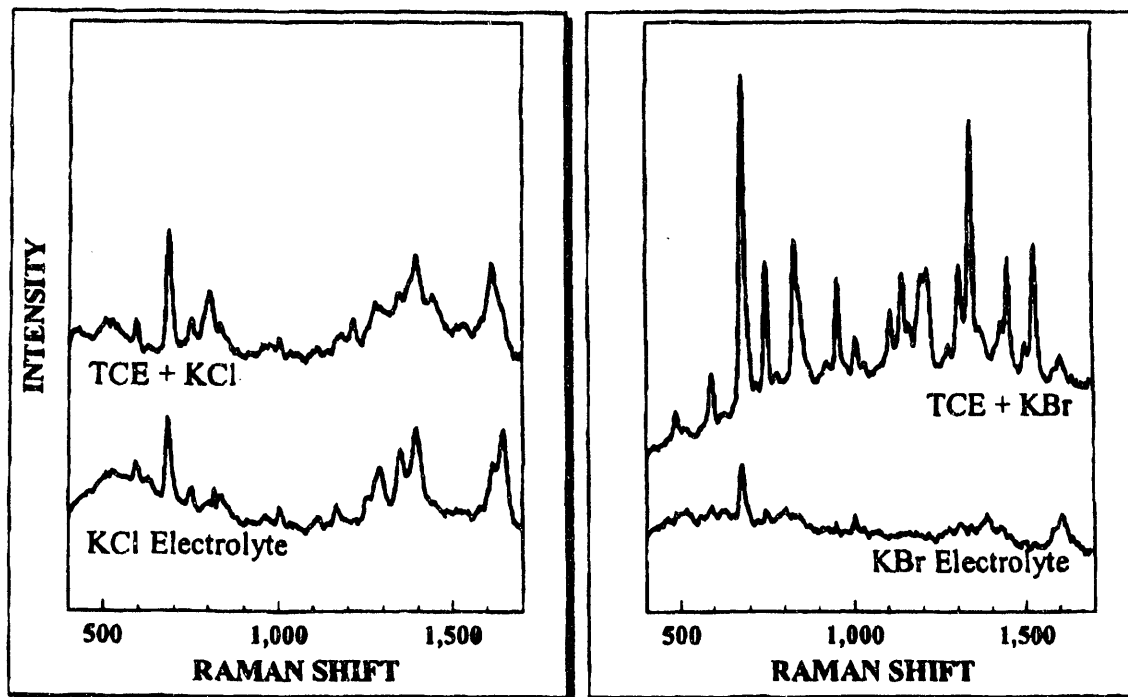


Schematic of modular spectroelectrochemical sensor. Using a common optics module (upper portion of sensor), coupled with analyte-specific semi-permeable membranes and electrodes, a custom sensor is easily assembled for detection of particular contaminants.

Accomplishments

A number of technical accomplishments have been recently achieved, including:

- development of advanced electrolyte compositions for enhanced detection of chlorinated hydrocarbons
- development of sensor technology for ppb-level detection of aqueous cyanide ion
- development of a modular sensor design allowing field configuration for specific target contaminants



Illustrations: Comparison of electrolyte effects on the spectroelectrochemical detection of aqueous TCE using a copper electrode. Left spectra, SERS response to TCE in KCl electrolyte (upper curve) vs. KCl electrolyte background. Major features observed in both spectra attributable to copper/chloride complexes. Right spectra, TCE in KBr electrolyte (upper curve) vs. KBr electrolyte background. Features in TCE spectrum well resolved from copper/bromide features. Note also significant enhancement of TCE response relative to chloride electrolyte.

Collaboration/Technology Transfer

The purpose of this task is to develop instruments that will be useful to DOE site workers involved in characterization and monitoring. Throughout this project, experimental results will be reported regularly to DOE and the public. In addition, emphasis will be placed on transferring developed instruments to the private sector as quickly as possible so that they become widely available.

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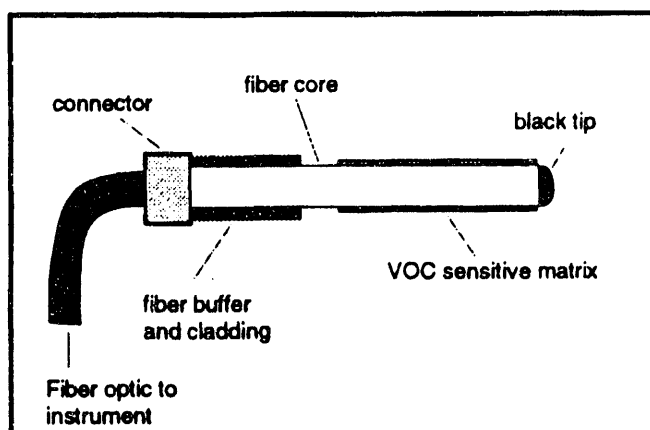
Improved Reversible CCl_4 Sensor for On-Line and Down-Well Measurements

Task Description

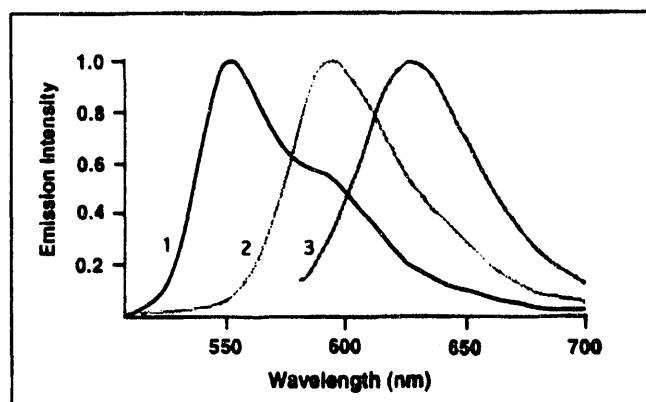
We are developing a reversible fiber optic-based sensor to detect carbon tetrachloride (CCl_4) and other volatile organic compounds (VOCs) that contaminate soils and groundwater. The sensor will be able to measure in real-time the concentration of general classes of vapor phase VOCs. The concept for the fiber optic VOC sensor involves the use of dyes whose optical properties change when they are exposed to vapors of volatile compounds of different chemical composition. The operation of the sensor is relatively simple. The VOC-sensitive dye-matrix is illuminated with a compact light source. Interaction of VOCs induces concentration-dependent changes in the emissive properties of the matrix which are monitored with a photodiode. The VOC-sensitive polymeric dye matrix can accommodate several basic configurations. This flexibility will enable the sensor to be incorporated into optical fibers for deployment down wells and bore holes to make vapor-phase measurements of VOCs in subsurface locations. It will also permit the sensor to be assembled into a portable handheld device that will measure above ground sites contaminated with VOCs. We are currently developing the VOC sensor to monitor carbon tetrachloride, but by changing the composition of the polymer-dye matrix we will be able to more selectively measure other compounds, such as aromatics in gasoline or the alkanes of jet fuel.

Technology Needs

The discovery and remediation of contaminated sites is very costly and new technology is necessary to mitigate clean-up and site monitoring costs. To achieve this goal, inexpensive analytical devices are needed that monitor VOCs in a variety of environments and process streams. Our innovative sensor is field portable, capable of measuring vapor phase VOCs on-line and down-well, and will provide cost efficient analytical field support.



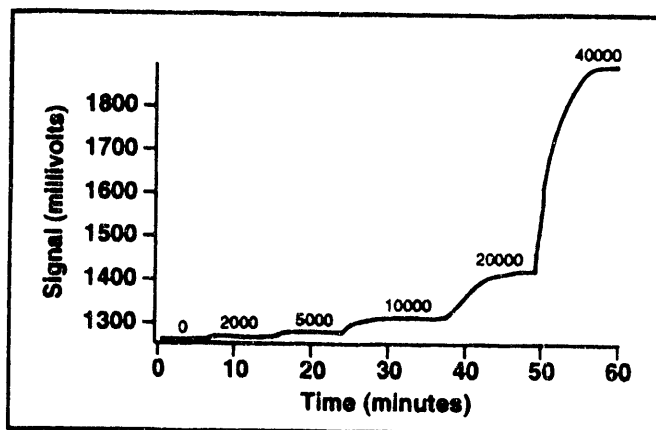
Schematic of VOC sensor, measuring $\sim 600\mu\text{m}$ in diameter and 3cm in length. Protective shield not shown.



Emission spectra measured with the VOC sensor upon exposure to vapors of (1) CCl_4 , (2) CHCl_3 , and (3) EtOH .

Accomplishments

To date we have characterized the spectral properties of several solvatochromatic VOC-sensing dyes and evaluated the interaction of VOCs with the dyes in polymer supports. Several polymer systems were evaluated on the basis of their ability to reversibly absorb and concentrate the VOC from the vapor phase. These polymers were also tested for their chemical inactivity towards the dyes. We measured the uptake of VOCs by the polymer and have quantified some important changes in the refractive index of the polymer as it absorbs VOC vapors. We have made vapor phase measurements of carbon tetrachloride with the reversible fiber optic sensor in flowing and static systems. The present sensor can detect this VOC at the 1000 ppm level. Our current effort is directed toward modifying the polymer matrix to achieve lower detection levels. We are also constructing a small monochromator system to increase the number of volatile organic compounds that can be detected. Lastly, we have constructed a vapor concentration device that will be miniaturized and included as part of a VOC detection system. This device is a thermoelectrically-cooled concentrator that will provide a means for analyzing very low levels of VOCs (low ppm levels) with a variety of different fiber optic-based technologies, ranging from simple chemical sensors to direct spectroscopic measurements with Raman or IR.



Response of VOC sensor to vapor phase CCl₄. Numbers represent the concentration of vapor phase CCl₄ (ppm).

Collaboration/Technology Transfer

This technology has been licensed by Purus and has received attention by several other companies and institutions.

For more information, please contact:

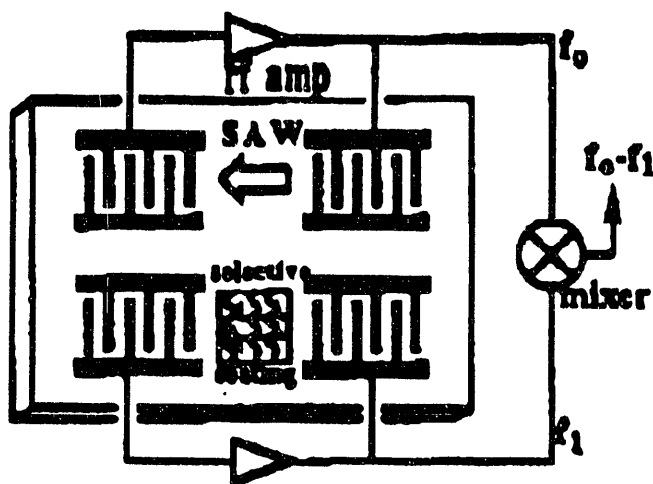
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Chlorinated and Aromatic Hydrocarbon Thin Film Chemical Sensors

Task Description

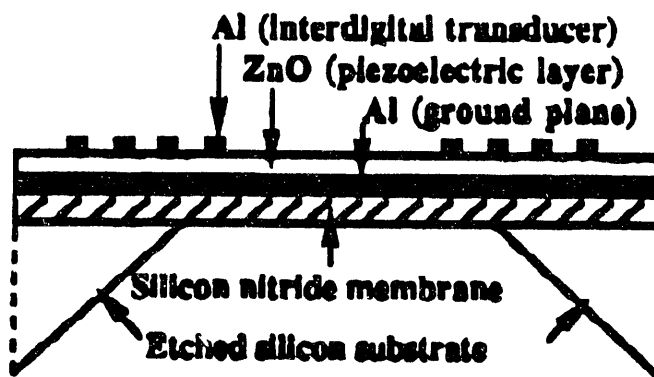
This proposed Surface Acoustic Wave (SAW) based chemical sensor R&D for chlorinated and aromatic hydrocarbons will greatly advance the current base-line SAW technology (which is inadequate for DOE needs); and may be a viable alternative to existing sensors, including the fiber optic conventional SAW based sensors. Proposed R&D will base a sensor on observable phase velocity change of a SAW or Lamb Wave (LW) device upon mass loading by a specific organic compound. A thin-film coating based on covalent attachment of a species selective reagent (to be determined) will provide the molecular specificity, reversibility, and sensitivity needed for both gas and aqueous phase measurements. When operational, the proposed SAW- or LW-based sensor will be reversible, inexpensive, and robust under realistic environmental conditions and will be capable of providing species identification. The operational sensor will also meet state and federal regulations (NEPA, RCRA, Clean Air Act, etc...) for continuous real-time monitoring of ambient air, ground and surface water, and contaminant plume conditions in subsurface water. Motorola Corp. is investing on a 50% cost sharing with the DOE to co-develop this technology (via CRADA) for commercial applications within industry.



SAW-based chemical sensor

Technology Needs

Sensors for continuous real-time monitoring of ambient air, ground and surface water, and contaminant plume migration will be needed for compliance with state and federal regulations (the Clean Air Act, the National Environmental Policy Act, and the Resource Conservation and Recovery Act, to mention a few). Moreover, the specific needs of the ER and WM programs require a new generation of sensors and advanced fieldable instrumentation. Present technology is simply inadequate to address the multitude of monitoring needs in site characterization, cleanup verification, post-closure monitoring, and process diagnostics and control. There is a specific high level need at a number of ID sites for real-time sensors that will accommodate both air and down-hole aqueous phase sampling of chlorinated hydrocarbons.



Edge view of Lamb Wave device

Accomplishments

During this initial funding cycle we will prepare and characterize monolayer thin films of differing cyclodextrins as the sensing layer on SAW substrate surfaces. We plan to have validation of the surface modification techniques and proof-of-principle demonstration of the resulting sensing capabilities for detecting chlorinated hydrocarbons and aromatics in air in

time to allow an informed decision regarding continued funding.

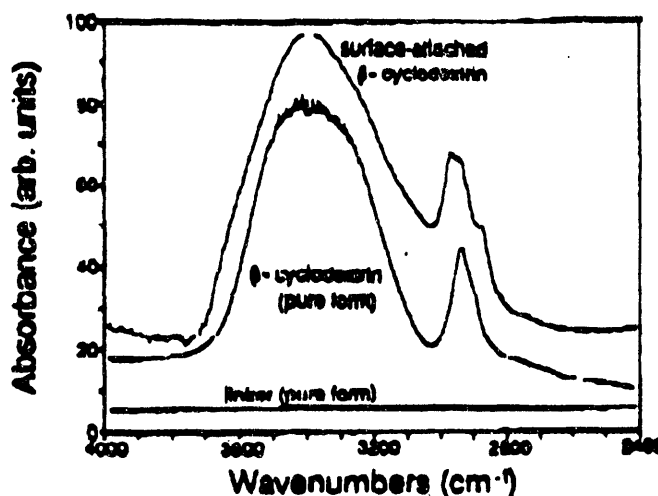
Toward this end we have made considerable progress in the six weeks since the start of the program February 1st. We have purchased and assembled hardware consisting of the SAW sensor rf-electronics, two frequency counters, and a personal computer with an IEEE interface, and have written software for basic data acquisition and real-time display of the sensor response. In parallel we have explored several of many possible routes to synthesizing and surface-attaching cyclodextrins. The first step requires attaching a linker layer that serves as the "glue" between the substrate and cyclodextrin reagent. We have been making extensive use of surface characterization techniques (optical absorption and FTIR-ATR spectroscopies, SIMS, XPS, and contact angle measurements) to gauge our success at each step. These methods give direct identification of the surface-attached species, and also provide information on the molecular orientation and film thickness. We have conclusively demonstrated successful attachment of several candidate linkers to the SAW substrate, and have very favorable indications of success in the subsequent and final step of attaching the cyclodextrin to the linker for the case of at least one — and probably two — of these linkers. Convincing evidence of success from FTIR spectroscopy for the case in doubt is clouded by the presence of spectral features that are common to both that particular linker and the cyclodextrin. Such ambiguities are commonly resolved by performing the same chemical synthesis and characterization with isotope-substituted cyclodextrin or other chemically similar cyclodextrin derivatives having FTIR spectral features distinct from those of the linker. While much work remains to be done we are very excited by the progress to date.

Collaboration/Technology Transfer

Motorola is interested in sponsoring and establishing a test facility at their Phoenix plant for waste stream monitoring and down-hole sensing in aqueous media. LANL will provide the sensor elements and technology transfer information to facilitate these tests which Motorola hopes to make applicable to the

semiconductor industry as a whole. A formal CRADA between Motorola and LANL is currently under development.

Several potential sites employed for the field testing and database development needed for entering into DOE's Methods Compendium are being considered. Discussions are currently held with Rocky Flats Plant and the Savannah River Plant ID.



FTIR spectra suggesting success in surface-attachment of β -cyclodextrin via a linker.

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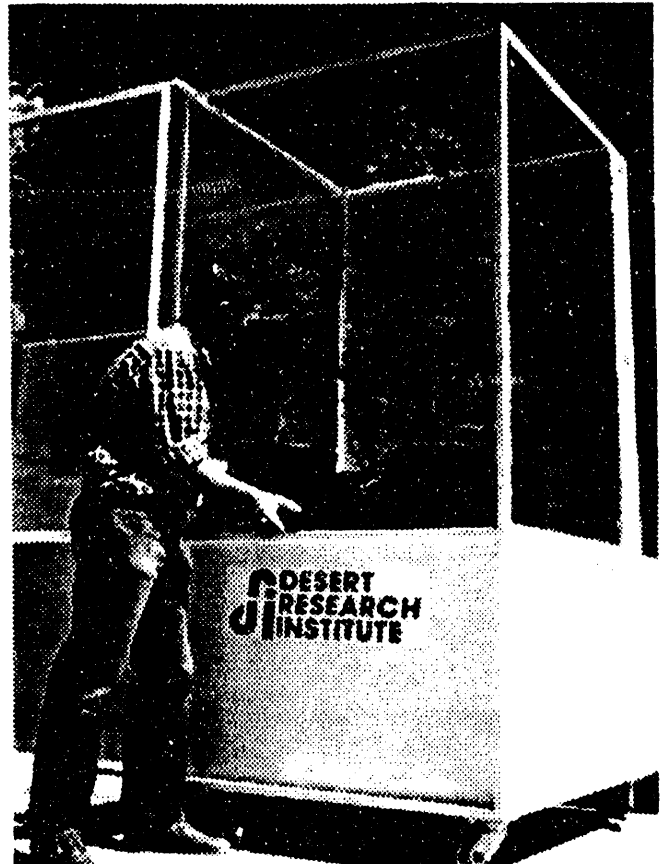
Remote Sensing Systems Development

Task Description

The focus of this project is the coordinated development and evaluation of existing sensor systems and new remote sensing technologies for environmental restoration and waste management applications. Emphasis will be placed on airborne remote sensing systems useful for surface and subsurface characterization studies. Airborne studies permit rapid, high resolution, non-intrusive thorough coverage of large areas. Potential applications exist at all DOE sites.

Existing remote sensing systems (both nuclear and non-nuclear) and their associated processing techniques will be evaluated and optimized to address site characterization and monitoring needs. Integration of remote sensing data with geographic information systems will also be developed. Remote sensing system commonalities, such as location equipment (e.g. Global Positioning System) and data recording devices and formats, will be examined.

In addition to optimization of existing technologies, several new technologies have been reviewed. Three have been assessed to be worth further development: hyperspectral imaging, subsurface characterization, and laser-induced fluorescence. These technologies will be refined and applied through airborne and ground field tests.



Environmentally controlled chambers known as ECOPODS designed for the study of the effects of contaminants on the spectral response of vegetation in support of waste identification on DOE sites.

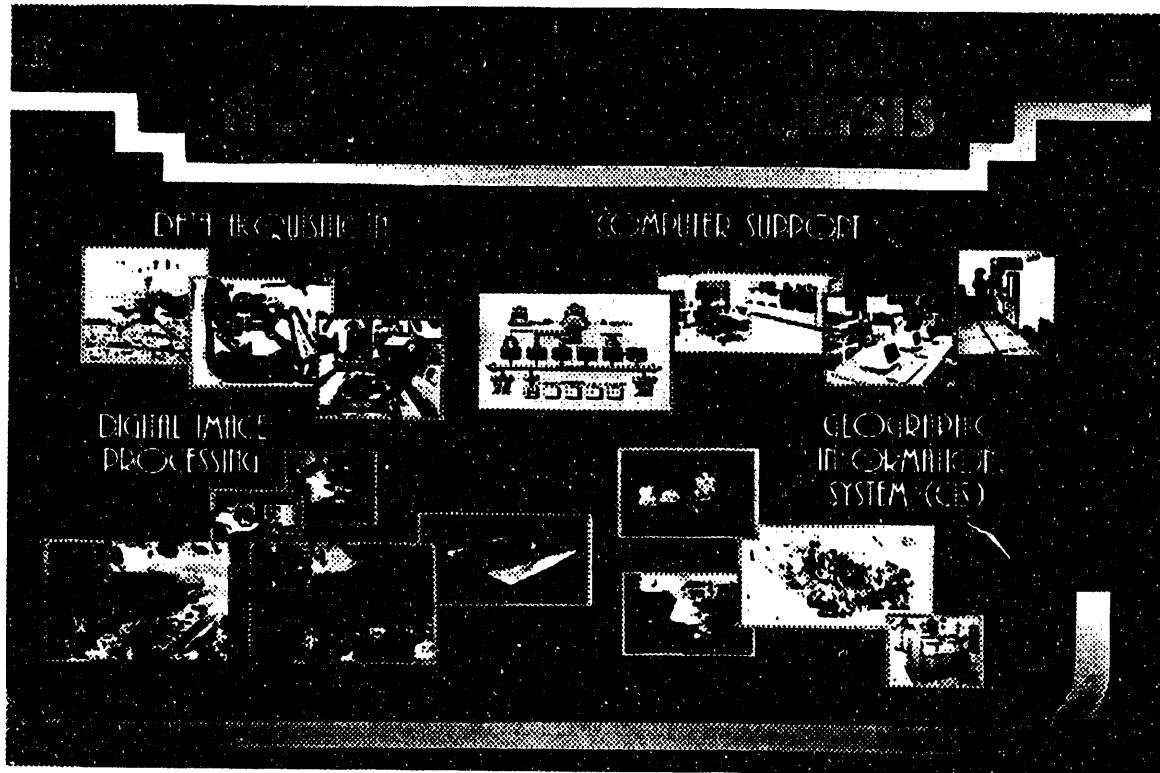
Technology Needs

There is a need for remote, in situ, and on-site local characterization of contaminants and resulting modification to the land surface. Airborne remote sensing systems can provide safe, spatially complete, high resolution, quantitative information on changes of the land surface resulting from ER/WM related activities. There is a clear need to disseminate information on existing DOE remote sensing capabilities as well as better coordinate future developmental projects.

Accomplishments

In April, 1992 the first annual Remote Sensing Liaison Working Group meeting was held at the EG&G/EM Remote Sensing Laboratory in Las Vegas. This meeting was successful in making the users more aware of remote sensing capabilities and applications as well as what the technology development program (EM-50) is doing.

Initial field trials of the Laser-Induced Fluorescence technology were successful. Depleted Uranium targets at Los Alamos National Laboratory and plumes associated with the Spill Test



Facility at the Nevada Test Site were identified. An airborne Multispectral Scanner survey allowed precise locations of buried waste sites at Los Alamos to be determined. Publication for the handbook of Airborne Subsurface Investigative Techniques is imminent. Data gathered through successful hyperspectral surveys of a toxic spill in the Sacramento River has demonstrated the usefulness of vegetation stress as a mapping tool for waste management and environmental cleanup.

Collaboration/Technology Transfer

The liaison working group will provide a linkage between OTD and ER/WM. It is expected that the working group will identify applications for existing technology and provide guidance for the development of new technology which are pertinent to current and future ER/WM needs. It is anticipated that the group will

meet once a year in addition to site specific meetings and informal communication as needed throughout the year. Collaboration with University of California at Santa Barbara, NASA Ames, JPL, and the US Geological Survey, as well as other DOE sites, is continuing to grow.

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Geophysical and Hydrogeologic Measurements

Zero-Tension Lysimeters: An Improved Design to Monitor Colloid-Facilitated Contaminant Transport in the Vadose Zone

Task Description

A lysimeter is a device permanently installed in the soil to sample soil water periodically. Zero-tension lysimeters (ZTLs) are especially appropriate for sampling water as it moves through saturated soil, although some unsaturated flow events may be sampled as well. Because no ceramic barrier or fiberglass wick is involved to maintain tension on the water (as is the case with other lysimeters), particles suspended in the water as well as dissolved species may be sampled with ZTLs. A ZTL design is proposed to capture samples of suspended colloids as they move in the vadose zone.

The improved ZTL design consists of a Teflon cylinder placed below an undisturbed core of soil that has been initially withdrawn with a hydraulically powered tube. The design has significant advantages over conventional designs with respect to simplicity and speed of installation. Because more ZTLs of the proposed design than of the conventional design can be installed to assess site variability, more accurate monitoring of contaminant transport before, during, and after remediation will be achieved. In addition, because of the improved spatial resolution in sampling and monitoring, if contaminants are mobilized, their source can be more easily identified than with piezometer-based monitoring methods.

The improved ZTLs will be installed at five sites contaminated with americium and plutonium at Rocky Flats Plant, Golden,

Colorado. They will also be installed near Ames, Iowa, where colloid-facilitated transport of heavy metals (Cu, Ni, Cd, and Zn) in municipal sewage sludge is monitored.

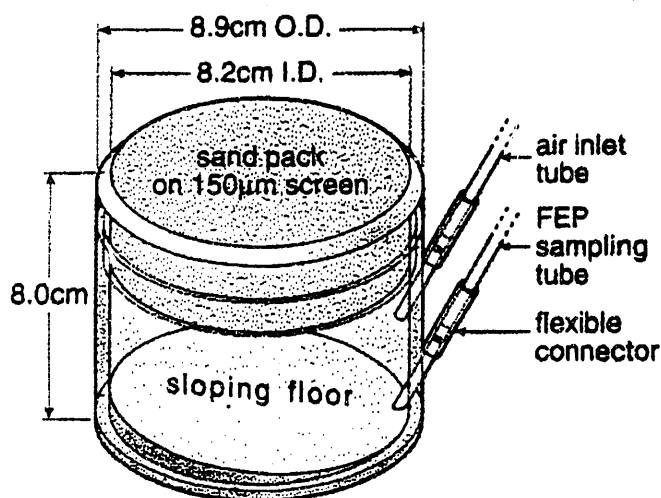


Figure 1. Basic design of the zero-tension lysimeter.

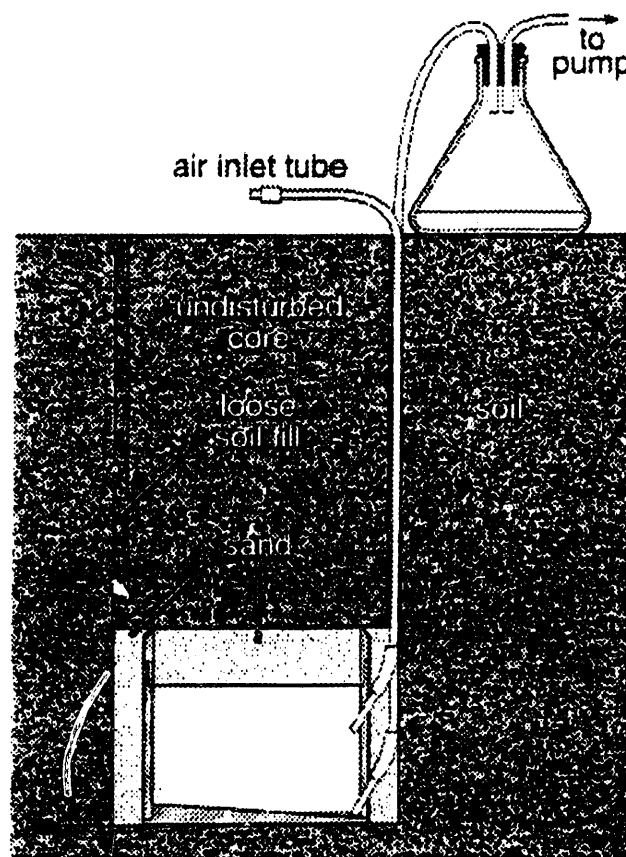


Figure 2. Sampling and maintenance of the zero-tension lysimeter.

Technology Needs

Zero-tension lysimeters are needed to monitor contaminant migration with mobile colloids. They are particularly applicable where a site is undergoing in-situ remediation treatment that leaves the soil physically undisturbed. They can also be used in

Zero-Tension Lysimeters: An Improved Design to Monitor Colloid-Facilitated Contaminant Transport in the Vadose Zone

other contexts where colloid-facilitated transport of contaminants is suspected or must be quantified in near-surface environments.

A zero-tension lysimeter that can be installed in much less time and with less site disturbance would allow more extensive and therefore more accurate monitoring of colloid transport in contaminated soils. In this proposal, a design that meets these needs is outlined.

eters to characterize the rates and mechanisms of actinide migration at the site. The improved ZTLs are also installed in soils near a municipal sewage treatment facility for monitoring migration of heavy metals. Successful field uses will result in wide acceptability at other sites for monitoring contaminant migration in soils.

Accomplishments

A prototype of the zero-tension lysimeter was installed in 24 research plots at a site receiving municipal sewage sludge in Iowa. The lysimeters effectively sampled colloid migration at 50 cm depth during both saturated and preferential flow events. Significant spatial variability of colloid and heavy metal migration occurred, even in plots receiving identical sludge and vegetation treatments. The ability to document such variability less expensively and more accurately than with conventional zero-tension lysimeters is seen as one of the important advantages of the present design. For installation at the Rocky Flats Plant, the design has been modified to improve the device's rigidity and to minimize potential for retention of contaminants.

Collaboration/Technology Transfer

The Principal Investigator works closely with an end user at EG&G/Rocky Flats in using the improved zero-tension lysim-

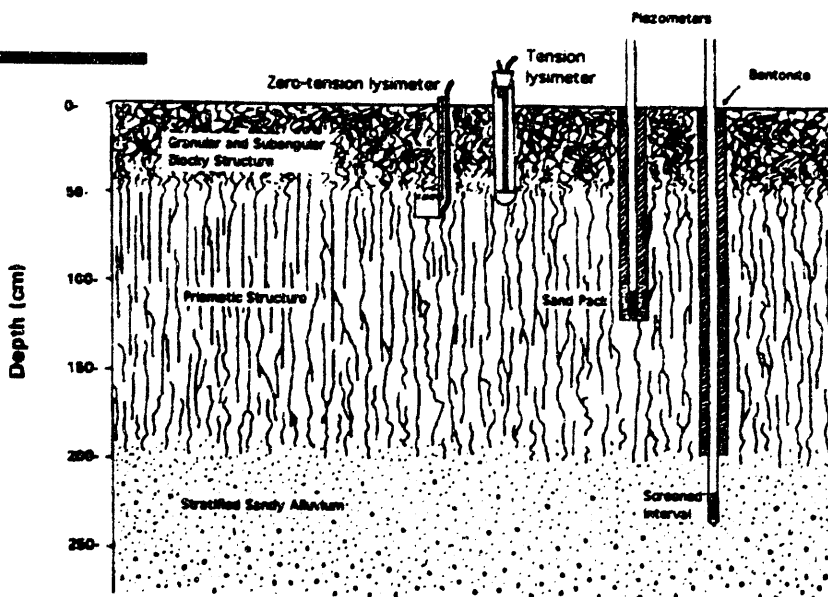


Figure 3. Typical installation of the zero-tension lysimeter will be in conjunction with other vadose-zone sampling and monitoring devices.

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Multi-Spectral Neutron Logging: A New Generation Pulsed-Neutron Induced Gamma-Ray Multi-Spectral Logging System for In-Situ Mapping of Contaminants

Task Description

The task is focused on neutron-induced, gamma-ray, multi-spectral borehole logging for in-situ mapping of contaminants. Resource Conservation and Recovery Act (RCRA) metals and chlorine are the elements of initial interest. The subtasks will be to modify an existing borehole logging sonde to make an innovative type of measurement, perform numerical calculations to re-design the sonde and interpret the data, and conduct experiments to physically demonstrate the hardware and verify numerical calculations.

Technology Needs

Under the DOE Environmental Restoration (ER) Program, techniques are needed which maximize the information obtained from boreholes and minimize the number of samples/analyses to reduce cost. Neutron-induced, multi-spectral borehole logging has the ability to map contaminants in-situ and is nuclide-specific. The technique can be used in boreholes as small as 4 inches in diameter and through steel casing. Measurement can be made repeatedly over any period of time to track changes in contaminant concentrations. No environmental contamination is caused by this measurement.

Accomplishments

- Work plans, budget and funding recently put in place
- Transfer of funds to USGS has been initiated
- Collaboration on modeling began in mid-February

Collaboration/Technology Transfer

This project will be performed jointly by the Los Alamos National Laboratory (LANL), the Grand Junction Projects Office (GJPO, operated by Chem-Nuclear Geotech), and the U.S. Geological Survey (USGS). The breakdown in responsibilities is as follows:

LANL: Numerical Modeling & Overall Coordination

GJPO: Neutron Generator & Logging System

USGS: Detection System

The LANL and GJPO organizations are funded separately and function under individual and separate Technical Task Plans. USGS funding is included in funding for GJPO.

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High-Resolution Subsurface Imaging and Neural Network Recognition: Non-Intrusive Buried Substance Location

Task Description

Ground Penetrating Radar (GPR) has been shown to be a powerful tool for environmental investigations, but in many situations the attenuation of radar energy is much too great for radar to be effective. In the southwestern United States, for example, the depth of penetration of radar energy is typically only one meter. In order to reliably obtain a usable depth of penetration for environmental investigations, it is necessary to use lower frequencies than are normally used in GPR investigations.

A high-frequency EM imaging system that overcomes the depth restrictions of ground penetrating radars will be developed for the frequency range 30 kHz to 30 MHz. The system will be an extension of an existing imaging system which has a frequency range of 30 Hz to 30 kHz. The high-frequency extension is necessary to provide high resolution over the range of possible depths that are of interest in this project.

The conventional method for interpreting EM data involves assuming simplified models of the earth, calculating theoretical EM fields for these models and comparing these to observed EM data. This procedure is typically slow and tedious. It usually involves a great deal of subjective guess work by the interpreter in order to choose realistic models. The procedure is often computer intensive. A more promising approach is neural network parameter estimation. The neural networks recognize

patterns in the data and estimate such quantities as the location, size and conductivity of the target in near real time. Unlike conventional interpretation schemes, neural networks retain their knowledge and continue to learn.

The high accuracy of the proposed imaging system, coupled with the fast, accurate interpretation by the neural network will provide a faster non-intrusive means of mapping contaminated sites with less ambiguity. The data collected will be interpreted in the field in near-real-time by neural network. The networks will output the identification and location of subsurface targets.

The proposed high-frequency imaging system could reduce occupational health risks by reducing the amount of time for conducting a geophysical survey and interpretation. The environmental risks will be reduced by pinpointing more accurately where objects are buried and what those objects may be. The more complete your knowledge is of the potential hazards, the more able you are to develop a remediation plan that will restore the environment. Faster and more accurate data acquisition and interpretation means both cost and time will be reduced for the remediation plan.

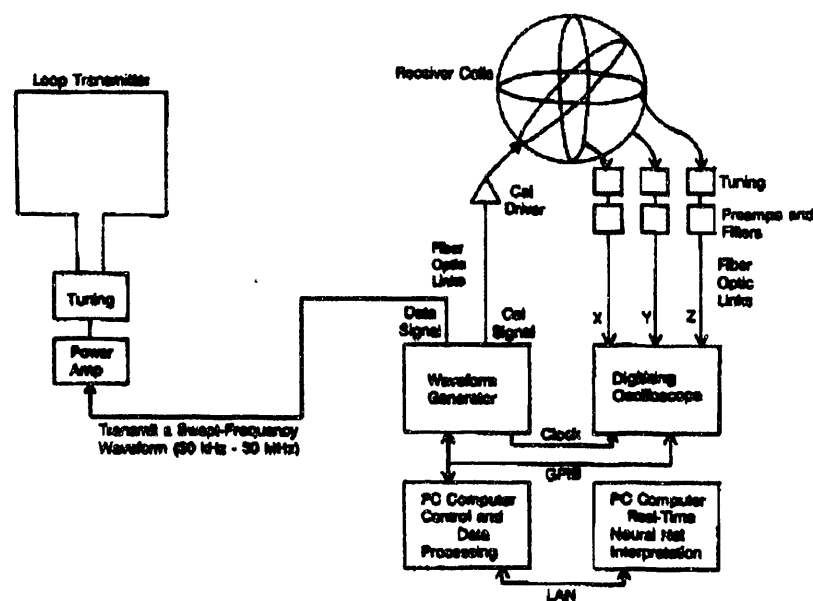


Figure 1. Block diagram of high-frequency ellipticity system.

High-Resolution Subsurface Imaging and Neural Network Recognition: Non-Intrusive Buried Substance Location

The project is expected to take eighteen months to complete with the first year spent building the imaging system and training the neural networks. The final six months will be spent testing the system in the field.

Technology Needs

Non-intrusive geophysical methods are needed for mapping subsurface structures, locating buried objects, and defining trench and pit boundaries. Performance enhancements which are expected to be accomplished by the proposed task will overcome the severe depth limitations of ground penetrating radar in conductive or clay-rich-soil and will provide higher resolution and accuracy than conventional DC resistivity or EM induction techniques.

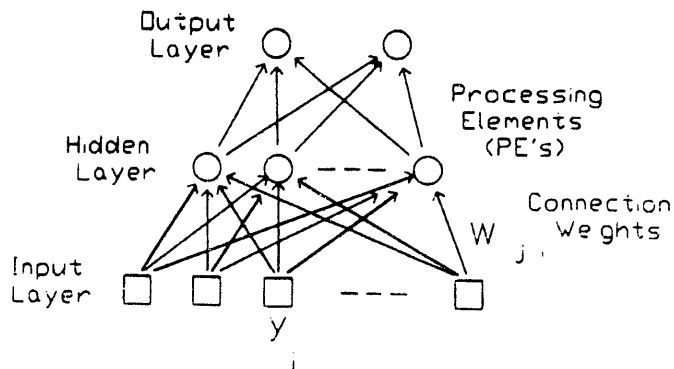


Figure 2. Simplified feed-forward neural network with one hidden layer. Input layer consists of observed ellipticities at various frequencies and distances. Output layer consists of target location coordinates.

Accomplishments

Initial version of software interface was developed on Dec. 15, 1992. Design of the receiver antenna was completed on Jan. 31, 1993.

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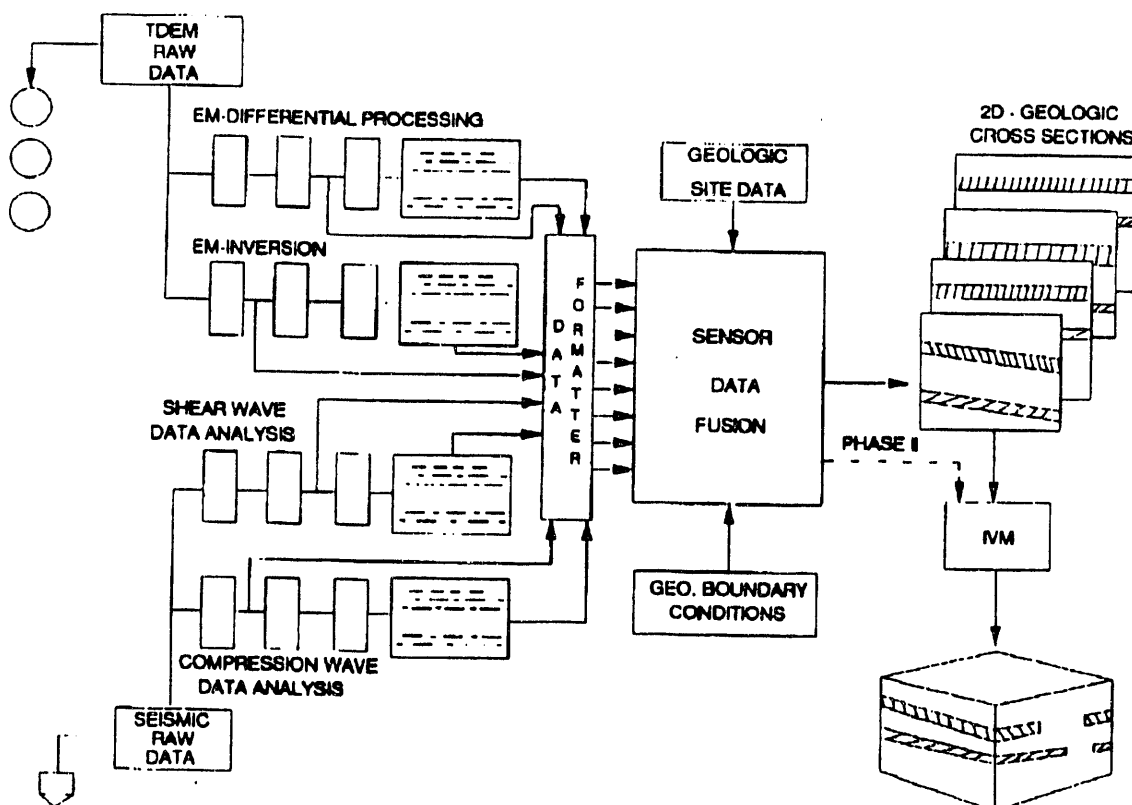
Geophysical Data Fusion for Subsurface Imaging

Task Description

A software package will be developed that will integrate geophysical data derived from multiple sensor technologies with geophysical understanding to obtain the best possible 3D subsurface image. A package will be developed to process multiple sensor data with sufficient automation to be accessible to engineers with minimal training.

The combining of data from non-invasive geophysical sensors, including time-domain electromagnetic induction (TDEM) and near surface seismic sensors, will be studied to determine the system effectiveness. To support decisions on the location of monitoring wells for contaminant plumes, it is desired to identify thin clay layers (1 to 3 feet thick) and geological discontinuities to depths of 300 feet. A high frequency seismic source will be evaluated to determine identification enhancement of thin strata.

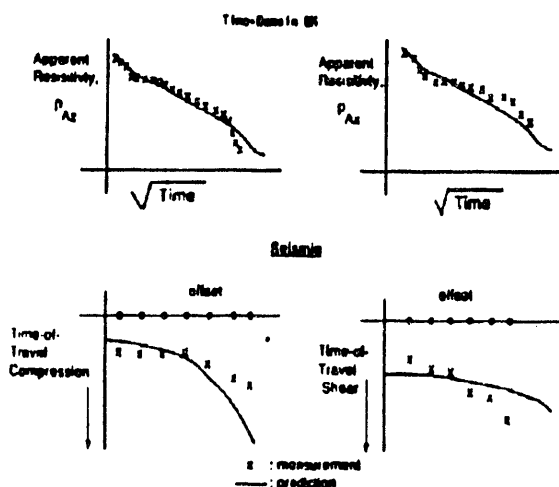
Shear wave surveys will be compared against conventional compression wave surveys to determine improvements in geologic detail. In addition, algorithms shall be developed for differential processing of TDEM. The conventional "out of loop" horizontal magnetic field TDEM process techniques shall be enhanced to increase sensitivity to geologic strata. This effort shall result in the generation of a three-dimensional (3D) display. The geophysical data fusion methodology will be demonstrated for an aquifer imaging problem at sites with hazardous waste contaminants. The proposed methodology will be enhanced by providing a full 3D capability, automating the approach, and optimizing the spatial computational flow. Advances in fusion and sensor technology will be incorporated in the software package. This software package will be portable enough so that it can run on a variety of work stations.



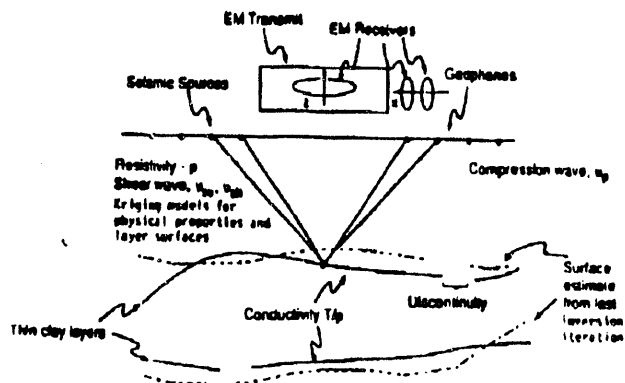
Fundamental Data Fusion Methodology

Aquifer Imaging Example Using Time-Domain Electromagnetic (EM) and Seismic Sensors

Inversion Shapes Image Until Data Consistent with Predictions



Multi-Sensor Imaging



Technology Needs

The measuring of highly stratified geology with thin, less than three feet thickness, and discontinuous layers intermixed with unconsolidated sediment at several hundred foot depth is currently not accomplished by any single or multiple geophysical surface sensor method. Extensive and time consuming logging of closely spaced exploration wells, and downhole geophysical detection such as cross-hole tomography are the only reliable alternatives for defining such complicated profiles. Even so, there is little guarantee that very thin clay layers have been adequately identified to permit meaningful groundwater flow migration predictions. This task will evaluate and develop an optimum suite of non-invasive geophysical sensors to address this need.

Accomplishments

Concepts on how to include the compression-wave and shear-wave seismic data, both reflective and refractive, along with vertical and horizontal TDEM in the fusion software have been developed. The initial plan is to use simple time-of-arrival data with apparent resistivities to test the fusion capabilities.

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Historical Imagery Applications for Environmental Restoration and Waste Management

Task Description

The feasibility of exploiting historical imagery to support ERWM activities has been analyzed with specific emphasis on the Fernald site for Discovery and Characterization.

This task has focused on site requirements, identification, acquisition, and evaluation of historical imagery, and definition of a system architecture which satisfies the functional, technical, and operational requirements of the site and regulators with flexibility to handle future imagery requirements during the Remediation and Surveillance and Maintenance Phases.

Future efforts will focus on analysis of the imagery and system implementation to meet current and future imagery analysis requirements.

Technology Needs

Site requirements for discovery and characterization which exploit imagery include Operations Reconstruction, Feature Detection and Recognition, Feature Identification and Mensuration; providing unique information and complementing other sources of information such as records and interviews. The process of analysis includes iterations between qualitative and quantitative analysis covering the entire site as well as individual operable units as specific issues evolve.

Historical imagery of DOE sites includes data from preconstruction through construction and operational phases. For the Fernald site, these are pre-1951, 1951-1954 (primary) and 1954-1988, respectively.

Imagery data taken during this period include ground, aerial, and space-based sensor information. Focus of this effort has been on aerial stereo mapping imagery in order to provide the quality required for the levels of analysis required. To exploit imagery in a cost-effective manner, data must be identified, acquired, and evaluated and processing and analysis tech-

niques and systems implemented which satisfy the various levels of site requirements while meeting regulator requirements and guidelines for meeting data quality objectives.

Accomplishments

The major objectives have been met with the final report, currently in draft, to be completed by 30 May 93.

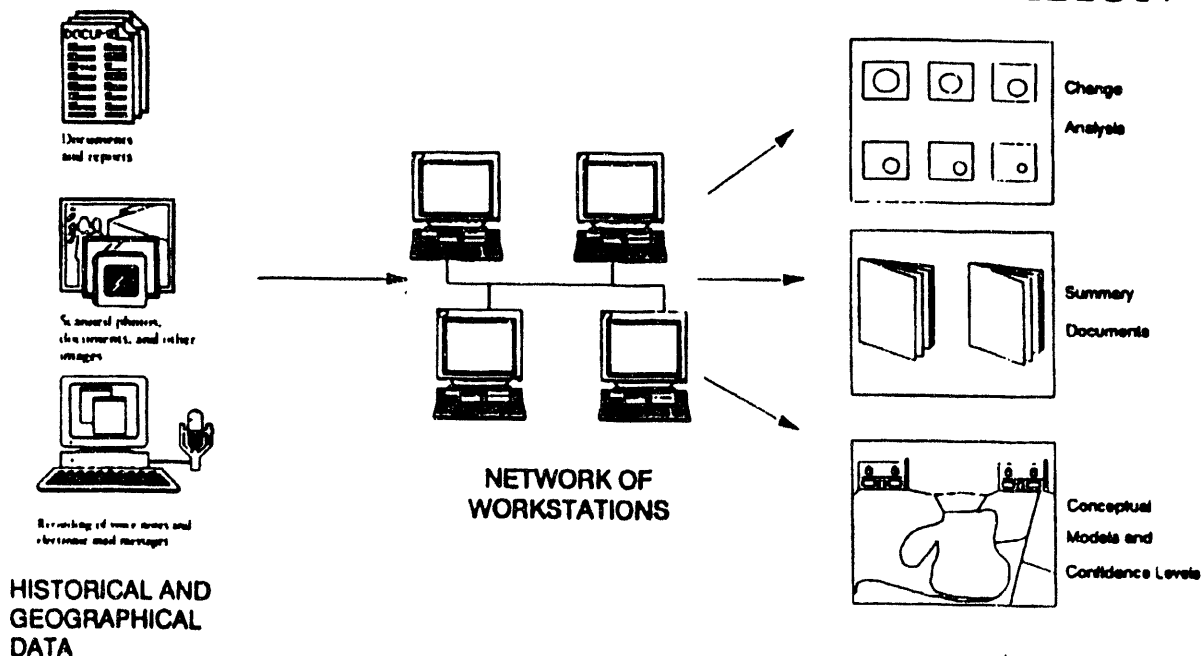
Site requirements have been defined and consolidated. Historical imagery, because of its synoptic spatial and temporal coverage, has been determined to support discovery and characterization requirements of all operable units. Consolidation of these requirements is therefore appropriate in order to define the most effective analysis plan.

Regulator requirements for the collection, analysis, and quality control of imagery have been identified. EPA recognizes the value and cost-effectiveness of remote sensing data, and historical imagery in particular, as an essential and cost-effective information source. Collection, processing, and analysis guidelines have been established for its exploitation.

Imagery has been identified, acquired, and characterized. Approximately 60 dates (over 200 images) of unclassified stereo mapping imagery, covering the period pre-1950 to 1988, have been acquired and characterized in terms of utility for ERWM applications. Sensors include panchromatic, color, and infrared with imagery in various scales, ranging from 1:3,600 to greater than 1:130,000. Virtually all of this data is useful for operations reconstruction, feature detection and recognition, and feature identification and mensuration.

System concepts and trades studies are being completed for systems design and implementation. Digital image processing systems are currently available with capabilities to convert, process, and enable analysis of the imagery cost-effectively for all levels of applications while satisfying regulator quality

HISTORICAL DATA FUSION WORKSTATION



Coleman Research Corporation (CRC) has been developing a workstation to retrieve, display and disseminate historical data of interest to DOE. This multimedia workstation currently has the capability to display and manipulate aerial photographs, maps, CAD drawings, oral interviews, text files and scanned images such as newspaper clippings. A user defined rule-based text retrieval capability is currently under development. The purpose of this retrieval system is to guide the user through the historical data in order to extract information that is relevant to a particular study.

(Accomplishments, continued)

control requirements. Image processing technology and systems are rapidly evolving in the commercial marketplace and, with open systems implementation, elements can be combined to provide a system solution which can produce the products required in a form compatible with site requirements, including the flexibility for handling future image processing requirements.

The results of this study, while focused on Fernald requirements, are believed to be applicable to other DOE sites where similar requirements and even larger amounts of available imagery are expected. Historical imagery, as part of a tiered approach to characterization, has been demonstrated to be a primary source of information for Preliminary Assessment/Site Investigation (PA/SI) and Remedial Investigation/Feasibility Study (RI/FS) and technology is rapidly evolving to enable analysis in a cost-effective manner.

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Data Fusion Workstation

Task Description

Currently thousands of waste sites exist at DOE facilities covering hundreds of square miles. The predominant characterization methods used are invasive techniques (bore-holes) which are inaccurate between bore-holes, risky, slow, and extremely costly. Additional sensor information from both remote and non-invasive sensors offer potential for dramatic improvement but no single sensor exists which address all characterization needs. An automated Fusion Workstation is being developed that offers the potential to mathematically fuse data from individual invasive, non-invasive, and remote sensors to produce a clearer picture with quantified confidence shown in Figure 1.

to show how the current state of practice can be automated into a single workstation and how existing sensor data can be fused to enhance the understanding of the overall problem as shown in Figure 2. The program has four goals for the development of the prototype workstation. These goals include:

- demonstrating the capabilities of data fusion within the environmental communities;
- producing a prototype workstation which provides tools in the actual data fusion process, manipulating, or interpreting raw data;
- gathering geophysical data at the Hanford Site which will be fused with existing bore-hole data;
- using a Geographic Information System (GIS) based man-machine interface to organize the data.

Technology Needs

Site characterization is currently performed manually by a principal scientist responsible for all aspects of a contaminated area. The principal scientist is supported by other personnel with expertise in individual fields of specialization (i.e., geophysicists, geochemists, geologists, sensor expertise, and model expertise). The goal of the Data Fusion Workstation project is

Accomplishments

A new theory for spatial non-causal random fields observed through nonlinear noisy data from multiple sensors was formulated for mathematical data fusion. The new data fusion theory is a transfer and extension of technology from novel Coleman Research contributions to space science, DoD, and intelligence applications.

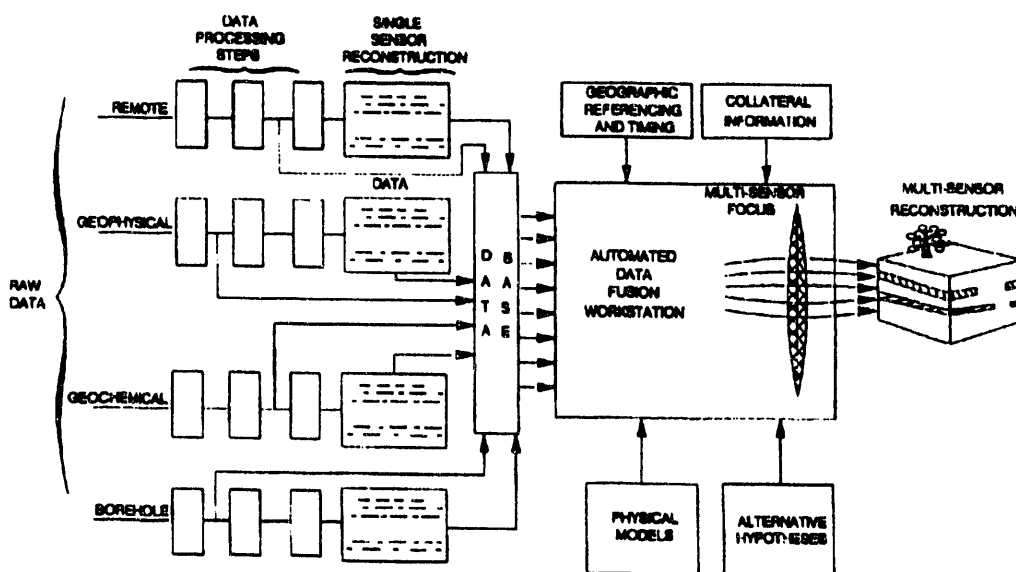


Figure 1. Integrated multi-sensor site characterization

Data Fusion Workstation

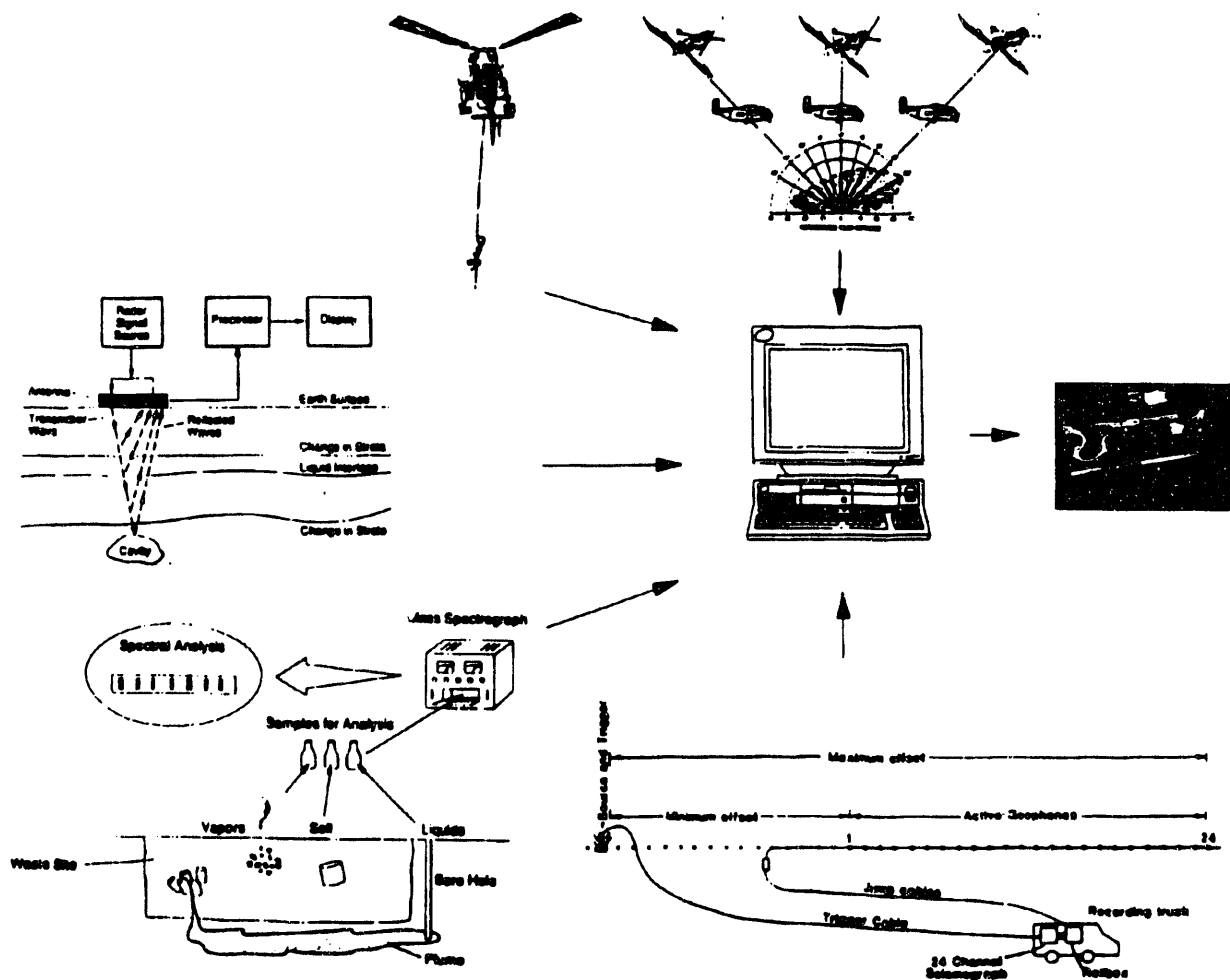


Figure 2. Data Fusion Workstation

(Accomplishments, continued)

The Hanford Site was selected for a data fusion demonstration in collaboration with the VOC/Arid Integrated Demonstration. A caliche layer that should be an impermeable barrier to contamination was chosen as the target for 3D imaging using seismic and electrical non-invasive geophysical techniques. Hanford data acquisition has recently been completed and data processing is under way.

Preliminary design of the deliverable prototype fusion workstation is completed and the detailed design is nearing completion. Commercial off-the-shelf hardware and software has been selected and is being acquired.

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Mixed Wastes in Drums, Burial Grounds and USTs

Associated Particle Imaging (API) Development

Task Description

The API imaging system is an active, non-intrusive, single-sided, fast-neutron based system that will examine the interiors of sealed containers, display the locations, shapes and sizes of their contents in three-dimensional image format, and identify their elemental compositions.

The general principles on which the API method is based are illustrated in Figure 1 (right). A Sealed Tube Neutron Generator (STNG), not shown in the figure, produces 14 MeV neutrons via the $t(d,n)\alpha$ reaction. The neutrons leave the collision site in a direction antiparallel to the α particles, as shown in the figure. The α particle hits a two dimensional position sensitive detector and starts the clock (labelled "TAC"). At the same time, the neutron heads off toward the region to be interrogated. If the neutron interacts with target material in the interrogation region, and if a gamma ray is produced and subsequently detected within a preset period of time, then the clock is stopped upon detection of the gamma ray. The α particle's location on the two dimensional detector gives the neutron direction of flight. The elapsed time between clock "start" and "stop" give information that tells how far the neutron travelled before hitting the target. These pieces of information ultimately give the x,y,z location of the neutron-target interaction. Furthermore, the energy of the gamma ray is characteristic of the material that was hit. Thus, the gamma spectrum allows one to identify the elemental composition of the material in the target region as a function of position.

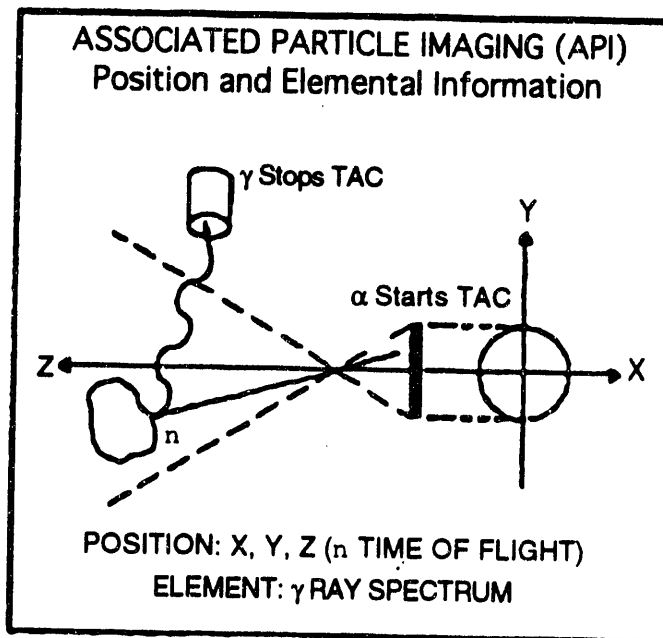


Figure 1

An example of API's 3D imaging capabilities is shown in Figure 2 (left). The color information is missing due to the constraint of a black and white text format. The data conglomeration on the left, labelled NaCl, is the salt image and is normally mostly green. The water image on the right is mostly blue. The boxes around the data aggregates were drawn by the user.

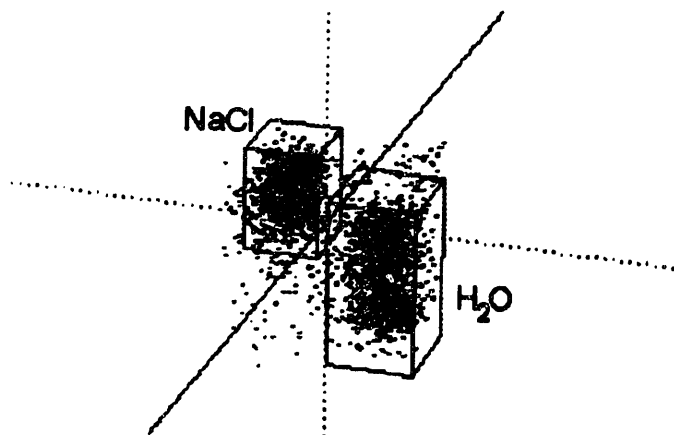


Figure 2

API's real power lies in its ability to gain simultaneous elemental and positional information. This is illustrated in Table 1 (see back). The two leftmost columns list the elemental information pertinent to salt (Cl), and water (O). The next two columns give information about each of these elements if only a "lump sum" of salt and water data is considered. However, if one is able to separate the salt from the water, as we have done in the boxes of Figure 2, one can significantly reduce the signal to noise ratio. This is evidenced by comparison of the third and fifth columns of the table. In terms of NDE measurements, this means that our confidence level is many times improved over what it would be without the ability to spatially isolate the data aggregates.

Technology Needs

API addresses the need for nondestructive evaluation of waste barrels and other closed containers by non-intrusive methods.

Accomplishments

The API system itself is very complex; the development of a system capable of producing the data shown in Figure 1 and Table 1 is an accomplishment in itself. API has progressed to the point, however, where such issues as improved signal to noise ratio (as in Table 1) are addressed. Work is beginning in the area of image enhancement as well.

Energy (MeV)	Element	Without Spatial Gate		With Spatial Gate	
		S/N Ratio	% Error	S/N Ratio	% Error
1.78	Cl	.15	17%	.43	9.3%
2.15	Cl	.12	16%	.58	5.1%
3.68	O	.15	6.9%	1.3	6.8%
6.13	O	.44	42%	.76	15%

Table 1: Spatial Gating Improves Signal to Noise Ratio.

Collaboration/Technology Transfer

There is as yet no transfer of API technology.

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Field Raman Spectrograph for Environmental Analysis

Task Description

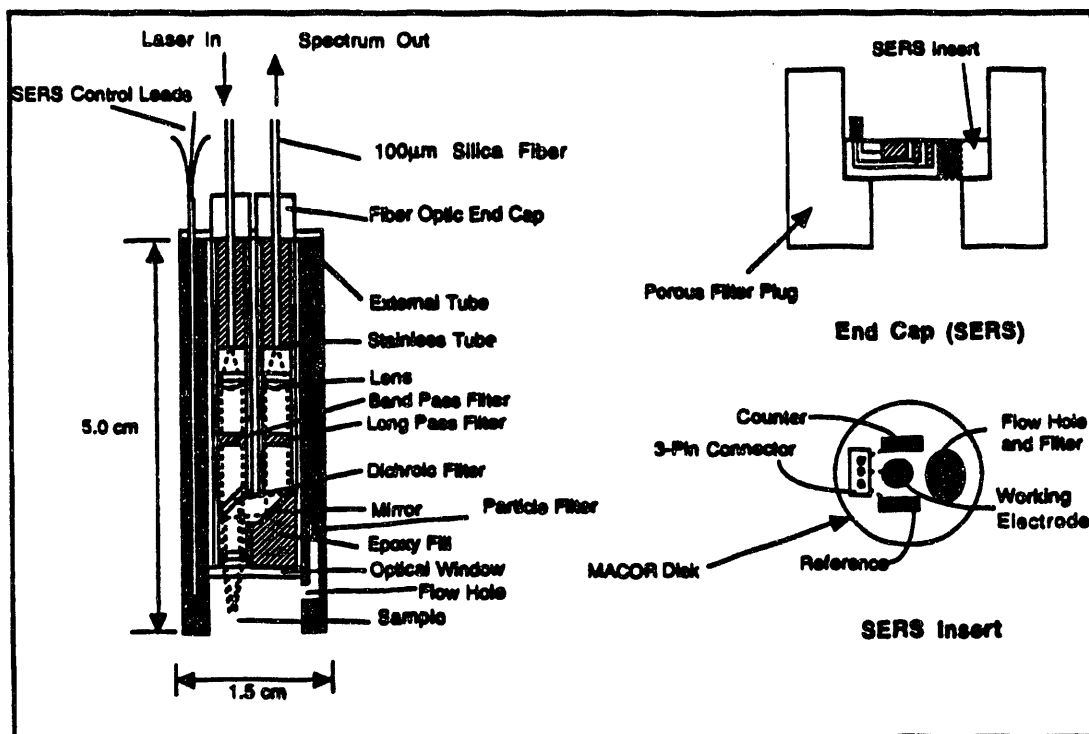
This is an instrumentation development project which combines fiber optics, Raman spectroscopy, echelle grating, tunable solid state lasers, micro-optical lenses and charge coupled devices (CCD). The field hardened, small (suitcase size) Raman Spectrograph will be used in field screening and monitoring of a wide variety of wastes, pollutants and corrosion products in storage tanks, soils, ground and surface waters at DOE sites slated for environmental restoration. The instrument will have the capability for remote operation which enables measurement to be conducted over >50 meters of optical fibers without interference from Raman scattering arising from the fiber itself.

The instrument uses monochromatic light from the tunable solid state laser which passes through fiber optics where a lens focuses the light on the sample. The light is scattered away from the sample, and some of the light is collected with another lens. The light is sent through fiber optics to the echelle grating where it is distributed on the CCD and the spectrum recorded. Correlation can be made with reference spectra to determine

specificity and concentration level. The wavenumbers of Raman bands provide the fingerprints of compounds, and the intensity of peaks provides an indication of the concentration.

The hermetically sealed spectrograph, lasers, detector and power supplies will be integrated into a small case which connects the sensing heads using fiber optic cable. The instrument, which contains no internal moving parts, is based on a unique design resulting in a ten fold reduction in size compared to conventional Raman spectrographs without loss of spectral resolution.

The Raman spectrograph system should be capable of characterizing critical pollutants at highly concentrated levels as well as at sub-ppm to ppb levels with enhanced Raman techniques such as surface enhanced Raman spectroscopy (SERS). Using the field portable instrument, Raman spectroscopy will be applied to site characterization problems through a subcontract with Oak Ridge and research collaborations with several national laboratories. Field tests will entail introducing Raman probes into waste storage tanks with both known and unknown contents, identifying the major chemical species present and mapping their distribution within the container. Additional site tests will involve introducing Raman probes into existing



Fiber optic, surface enhanced Raman probe used with the field Raman spectrograph for *in situ* characterization of subsurface contaminants.

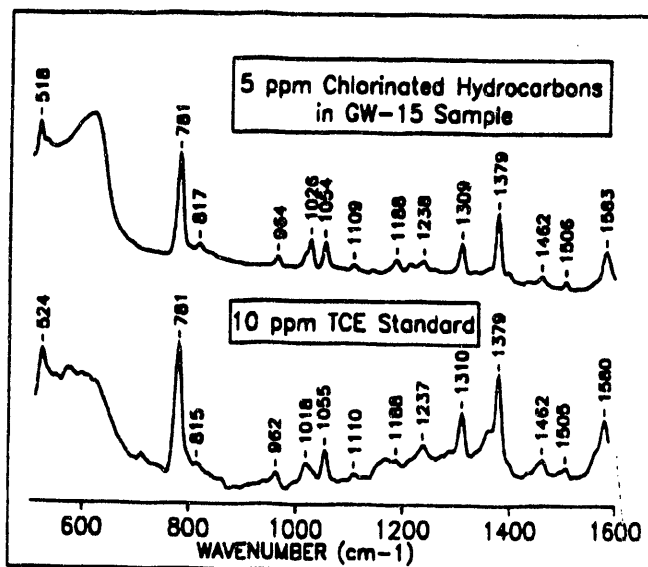
sampling wells and identifying groundwater contaminants such as chlorinated hydrocarbons and volatile organic soil gases *in situ*.

Technology Needs

The field hardened, portable Raman probe will address the needs for *in situ* mapping of the chemical contents in underground storage tanks. The measurements which can be conducted remotely over >50 meters of optical fibers provide safe protection of the personnel from exposure to the harsh, radioactive environment. Advantages of the technique would also include increased efficiency of environmental characterization and mapping, reduced costs associated with field sampling and *ex situ* laboratory analysis and more effective real time monitoring of remediation activities.

Accomplishments

The design and fabrication of a prototype field-hardened Raman system has continued. Several off-the-shelf near infrared lenses were evaluated and found to be adequate for use if the focal length of the CCD lens was reduced to 75mm. Calculations indicated that the resolution of the system was not affected. The 75 mm lens should also confer the added benefit of shorter data acquisition time due to a demagnification process of the fiber optic input. The use of off-the-shelf lenses will speed up final assembly and testing of the system.



Surface enhanced Raman spectrum of groundwater in Oak Ridge, TN contaminated with chlorinated hydrocarbon solvents such as trichloroethylene.

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Remediation, D&D and Waste Process Monitoring

Novel Mass Spectrometric Instrument for Gaseous and Particulate Characterization and Monitoring

Task Description

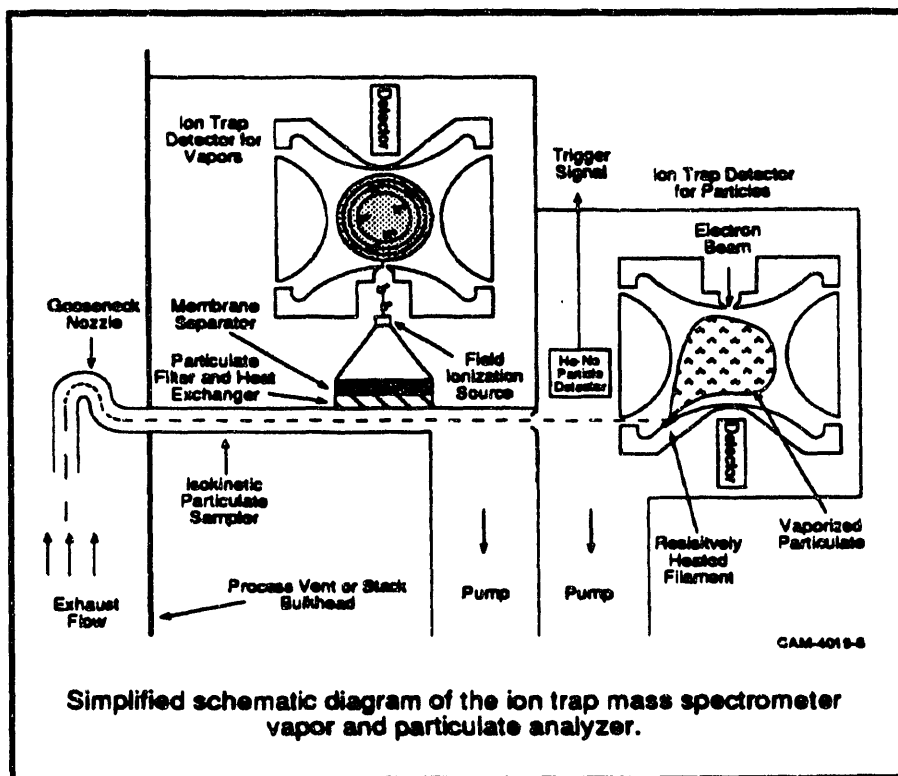
A unique and versatile instrument will be developed that will provide real-time, part-per-billion detection sensitivity for important pollutant species relevant to DOE waste cleanup activities. This instrument will have an isokinetic sampler for parallel detection of gases and particles by two ion trap mass spectrometers. Initially, the instrument will be developed for use in conjunction with the K-1435 Toxic Substances Control Act (TSCA) incinerator at the Oak Ridge National Laboratory K-25 site. Ultimately, the instrument will be designed to operate in the field at any cleanup site, located near the stack or process vent to provide the plant operations personnel with real-time information and alarm capabilities.

The instrument relies on both commercially available technology, including a simple laser scattering particle detector and an ion trap mass spectrometer, and unique technologies in the areas of field ionization mass spectrometry, high-temperature sampling, and membrane separation. The instrument is expected to be easily transportable with minimal utility requirements, making it ideal for deployment at a variety of DOE waste sites.

Moreover, the straightforward design and use of proven technologies will result in an instrument of high reliability and sensitivity that can be ready for actual field use within two to three years.

The instrument will be capable of detecting and identifying volatile organic compounds, polynuclear aromatic hydrocarbons, heavy metals, and transuranic species released during waste cleanup. It will be unique in its ability to detect and quantify in real-time these diverse pollutants in both vapor and particulate form.

The instrument to be developed under this program will consist of four major components: a sampler capable of operating over a wide range of temperatures; two small mass spectrometers, one optimized for organic analysis using a unique field ionization source and one optimized for particle characterization using thermal pyrolysis and electron-impact ionization; and a powerful computer for control and data acquisition.



Technology Needs

Real-time monitoring of off-gas stack emission for hazardous pollutants is needed to safeguard workers and the general public. The instrument being developed will detect and quantify volatile organic compounds, polynuclear aromatic hydrocarbons, heavy metals, and transuranic species in the gaseous and particulate phases. The instrument will also be used for air quality monitoring during waste cleanup.

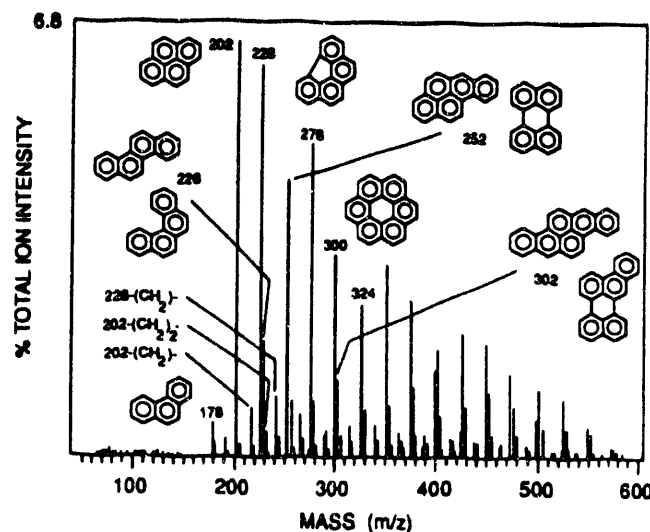
Accomplishments

The initial 18-month, Phase I research project was initiated in the Fall of 1992, with the actual technical effort beginning at the end of the year following satisfaction of all NEPA requirements. The goal of the first phase project is to demonstrate a laboratory prototype instrument capable of simultaneous vapor and particulate chemical analysis and characterization.

To date, the major vacuum components comprising the differential pumping system and sampler interface have been designed and are under construction. In parallel with the vacuum system work, both ion trap mass spectrometers are being modified. One ion trap will have a heated pyrolysis filament installed for particulate detection, while the other will incorporate an SRI-developed, microfabricated field ionization source optimized for organic vapor detection.

Preliminary tests have shown that the *in situ* volatilization of small, metallic particles within the ion trap volume leads to excellent detection sensitivity and reproducible isotopic ratios. Vapor detection by field ionization mass spectrometry has also been shown to provide rapid chemical characterization of very complex mixtures, including polycyclic aromatic hydrocarbons and other volatile organic compounds.

The project is presently on schedule to begin testing by August, 1993, using laboratory generated test atmospheres.



CAM-4018-7

Field ionization mass spectrum of soot from pyrolysis of butane showing the sequence of PCAH intermediates and their alkylated derivatives.

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Air Quality Site Boundary Monitoring for Volatile Organic Compounds by Preconcentration/Membrane Ion Trap Mass Spectrometry

Task Description

This proposal describes a methodology in which patented preconcentration technology developed at Los Alamos will be combined with a transportable gas chromatograph ion trap mass spectrometer (GC/ITD) for the field collection and detection of airborne VOCs. Membrane sampling technology, developed at Purdue University, will be used to introduce the preconcentrated VOCs to the GC/ITD. Each of these individual technologies has been proven successful. Sensitivities can be tailored to quite easily surpass any existing or realistically anticipated regulatory requirements for the lower limits of detection. This technology can provide both primary (fast screening) data and secondary (slower) legally defensible GC/MS data to verify air quality at and around DOE facilities and remediation sites. Sampling can be done in near-real time (minutes); additional samples can be easily collected for verification by independent off-line analysis.

for accidental releases during waste exhumation at the Buried Waste ID (B-1) at INEL.

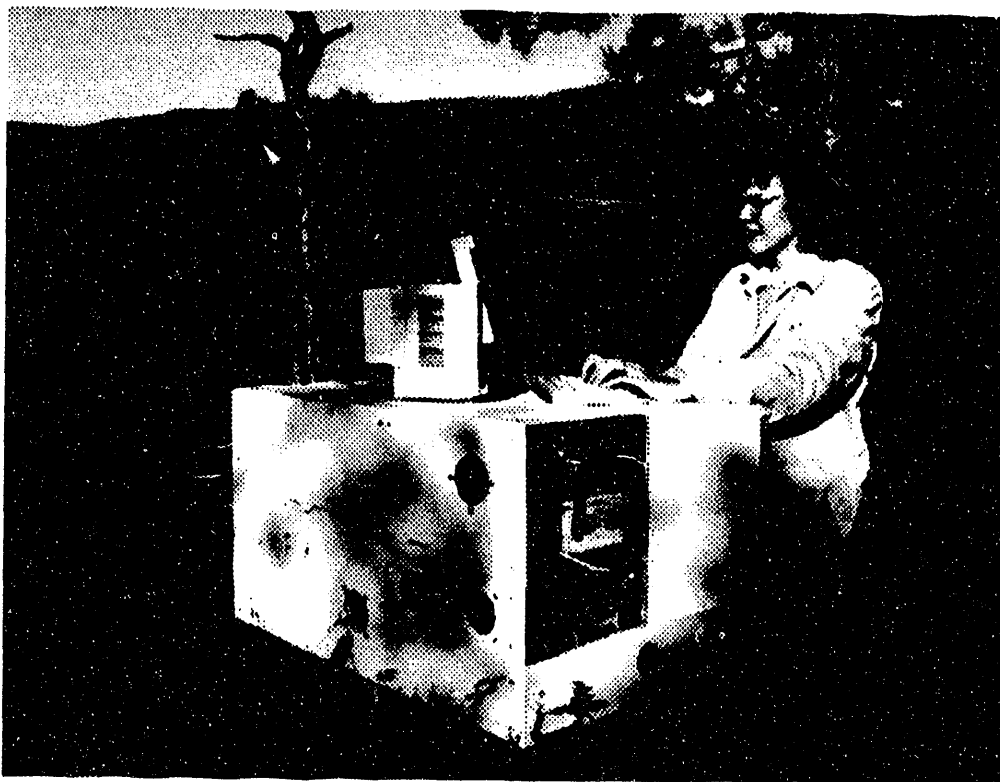
The proposed technology can support many other activities in the environmental arena: (1) Air monitoring at hazardous or mixed waste storage sites. (2) Air monitoring at DOE and private industrial areas to ensure worker safety and document exposure. (3) Detection of atmospheric tracers to provide data for global modeling efforts. Finally, the instrument could be used to monitor chemical emanations that are signatures of military operations, drug processing, and other illegal or aggressive activities.

Technology Needs

This technology is needed at every DOE facility to monitor air quality at site boundaries. There are many scenarios which require high sensitivity detection of atmospheric volatile organic compounds: (1) Routine monitoring to establish baseline air quality at site boundaries. (2) Response monitoring to characterize and document atmospheric distribution of volatile chemical solvents from accidental releases or spills. (3) Waste site monitoring to ensure worker and public safety during remediation activities. Specific Integrated Demonstrations (IDs) that could be supported by this technology include those where remediation of VOC contaminated soil is planned or underway (Savannah River A-1, Hanford A-4, and Sandia-Albuquerque A-9). The instrument could provide monitoring

Accomplishments

We have made several accomplishments in the few weeks that we have been working on this project. The Compact Micromist Air Sampler (CMAS) is set up for the sampling and concentration of VOCs in air. Preliminary experiments, where micromist samples are collected and analyzed off-line using the transportable GC/ITD, are very promising, demonstrating excellent detection limits and good specificity. We have evaluated several methods to reliably prepare samples containing high (part-per-million to part-per-billion) to low (part-per-trillion) amounts of VOCs in air for instrument calibration. Professor Cooks' group at Purdue University has made some exciting advances in the development of a coaxial pneumatically assisted membrane/jet separator combination. Detection limits for organics in water, which is the form of the sample provided by the CMAS, using this separator with ion trap detection are below the 1 ppb level, even at these early stages. A patent on this concept is being pursued at Purdue.



The Los Alamos transportable GC/ITD is being used for field analysis of VOCs in the environment. This instrument operates under a turn-key operating system to minimize operator interaction. It has been successfully demonstrated at the Rocky Flats Plant and was operated there by program managers and pipe-fitters. An updated and down-sized version of this instrument will be the detection system for the air quality monitoring technology.

Collaboration/Technology Transfer

This project is in collaboration with Professor R. Graham Cooks of Purdue University. The Principal Investigator is also discussing potential collaborative efforts with Dr. George Stafford of Finnigan MAT, the commercial manufacturer of the ion trap mass spectrometer. We are also discussing technology transfer with MIMS, a company founded to develop and market membrane separators for chemical analysis. On-site demonstration of the proposed instrumentation is planned to be held at national meetings in mass spectrometry to help publicize those accomplishments to the general public.

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Continuous Emission Monitor for Incineration

Task Description

This project addresses the need mandated by the Clean Air Act of 1990 to monitor air toxics. The technology is applicable to both stack and ambient air continuous monitoring. The objective is to develop a Fourier transform infrared (FTIR) spectrometer combined with a heated long-path cell as a continuous emission monitor for incinerators. The instrumentation will continuously monitor organic products of incomplete combustion (PICs). To provide the required on-stream analysis, FTIR technology must be developed.

The primary benefit of an on-stream monitor for incinerator effluent is the near real time determination of incinerator performance. On-stream analysis of the incinerator effluent would satisfy the requirements of the Clean Air Act of 1990 and address public concern about incinerator safety. Other benefits would include elimination of the need for a trial burn and the associated cost of demonstrating the destruction and removal efficiency of the incinerator. This would replace the more costly sampling and analysis utilized under current procedures.

The project consists of assembling and testing the performance of an FTIR system first with a laboratory incinerator, which can be operated under closely controlled conditions, and then on mixed waste incinerators. Figure 1 shows a picture of the laboratory incinerator and FTIR monitor.

In the first year of this program, FTIR technology was advanced in four areas: 1) testing cell materials for stability to hydrogen chloride, 2) characterization of flue gas PICs, 3) interpretation and evaluation of data quality, 4) development of automated analysis software. The above information was used for the initiation of construction of the prototype equipment for field use.

The second year will have two subtasks. The first subtask will consist of using the information gained from the first year's research to complete the construction and preparation of the prototype equipment for field use. All instrumentation will be constructed and tested in the laboratory before field use. The second subtask will consist of technology development and testing. There will be a QA data assessment which will evaluate our data with the proposed EPA requirements. The instrumentation will be field tested at the Oak Ridge TSCA incinerator. This data will be presented to EPA for method certification.

Technology Needs

Title 3 of the Clean Air Act requires monitoring of air toxics from incinerators, stacks and fugitive emissions. This technology is applicable for these requirements. This work will result in a certified EPA method for FTIR monitoring. This method can be extended to cover stack and ambient air monitoring. Continuous monitoring will directly address issues of environmental safety. The technology can be used to identify and quantify organic chemicals in air. There is significant public concern about incineration. A continuous emission monitor addresses those concerns.

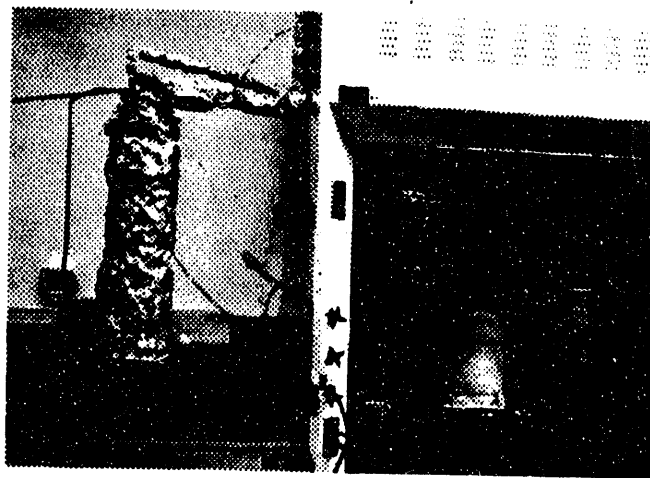


Figure 1. Laboratory incinerator and FTIR monitor

Accomplishments

A laboratory incinerator has been constructed and integrated to an FTIR system. The operating conditions can be deliberately and reproducibly changed to simulate off-set conditions of an

Continuous Emission Monitor for Incineration

incinerator. After an initial test burn of the single component: toluene, chlorobenzene, 1,1,1-trichloroethane and trichloroethylene, a mixture of the four with elemental ratio C:H:Cl=3:3.1:0.9 (simulated PCB) was incinerated at various conditions. Fig. 2 demonstrates the corresponding spectral changes detected successfully by the FTIR system. Figure 3 shows continuous emission monitoring results for 5 compounds during a controlled temperature reduction in the incinerator. The laboratory test demonstrates the capability of FTIR spectroscopy in monitoring incinerator emissions.

A transportable FTIR for field testing has been purchased after careful review. An optical system which interfaces to the FTIR long-path cell has been designed and is under assembly. The long-path cell has been ordered. The new FTIR system has higher sensitivity and resolution than the existing one and includes a visible beam to help alignment of the optical system. Hardware and modifications to the LabCalc Array Basic software have been completed for data acquisition in the field instrument.

Discussions with the Oak Ridge Gaseous Diffusion Plant to field test the FTIR continuous emission monitoring system at the K25 TSCA incinerator have taken place. A field test is tentatively scheduled for early summer, 1993.

Development of QA/QC protocols for EPA certification is ongoing. A calibration data set will be generated and used as a benchmark in the evaluation of varying data analysis algorithms. Data quality will be thoroughly demonstrated and validated for EPA certification.

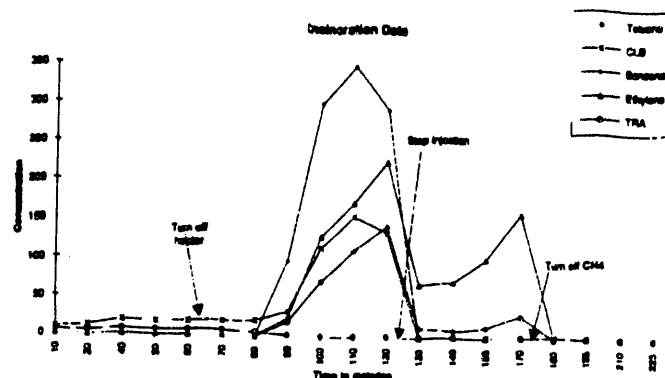


Figure 2. Plot of concentrations of five organic analytes during a burn.

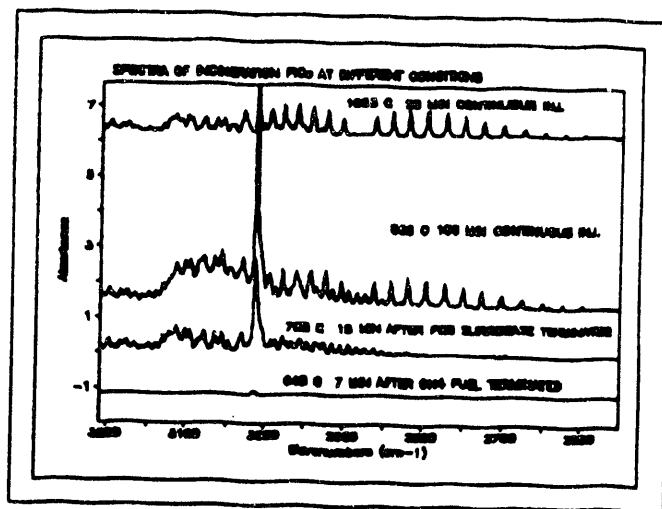


Figure 3. Spectra of incinerator emissions obtained before and during an up-set.

Collaboration/Technology Transfer

A CRADA is currently under development with Clean Air Engineering (CAE) and Hughes to advance the commercialization of the system after EPA certification. CAE will assist in developing stack sampling technology which is appropriate for this new technology.

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Infrared Analysis of Wastes: Novel Lab and On-Line Measurements by Photoacoustic and Transient Infrared Spectroscopies

Task Description

This project will develop a methodology for quantitative analysis of multi-atomic ions and molecular species in tank sludges, which are chemically active and hazardous under certain conditions, via Fourier Transform Infrared-Photoacoustic Spectroscopy (FTIR-PAS). Goals include a quick and easy method for producing quantitative FTIR-PAS spectra from small surrogate waste samples with a minimum of sample handling, and an analysis of FTIR-PAS spectra dependence on the complex composition of the waste materials. Once a methodology for producing quantitative spectra is determined, an FTIR-PAS system will be installed at Hanford and a training session will be held by Ames personnel.

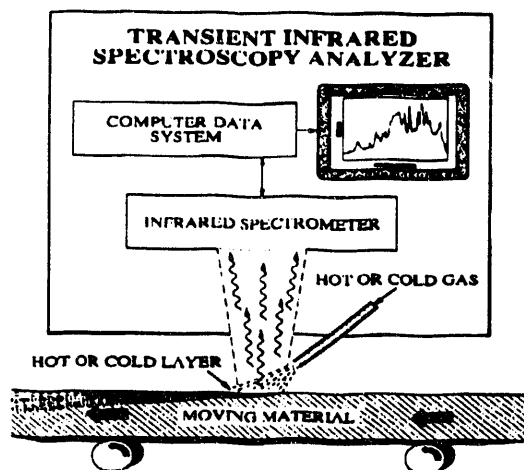


Figure 1. Schematic of a TIRS analyzer on a process line. A jet of gas heats or cools the surface of the moving process stream. This alters the infrared light emitted by the passing stream. This alteration is picked up and used by an infrared spectrometer in the TIRS unit to produce the infrared spectrum of the stream or to measure the desired chemical or molecular property of the stream.

Also to be developed is the application of Transient Infrared Spectroscopy (TIRS) as a monitor for polymer encapsulation of low-level waste (rad waste salt). Figure 1 shows a schematic of a TIRS unit on a process line. The monitoring will certify that the proper waste-to-polymer ratio is maintained during encapsulation and insure that excess processed waste volume is avoided. A laboratory-scale, proof-of-principle test on an extruder at Brookhaven National Lab has been successfully completed. A dedicated FTIR spectrometer for the monitor has been acquired and access to a lab-scale extruder has been secured for experiments to optimize the

TIRS approach for the salt/polymer waste monitoring application. The system will then be modified and expanded for a full pilot-scale waste processing system.

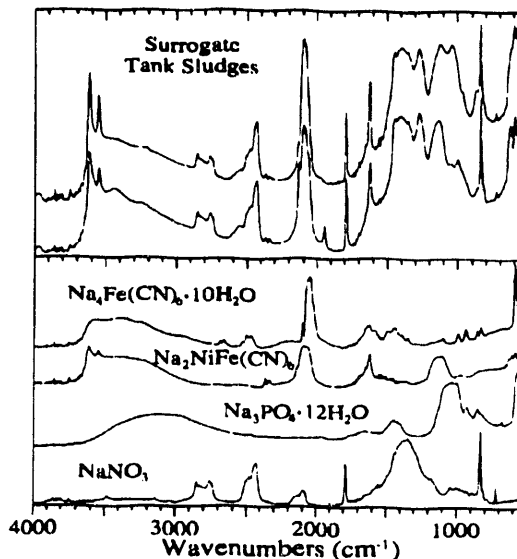


Figure 2. FTIR-PAS spectra of surrogate tank sludges (top) and of separate possible components of the sludges (bottom). There is an obvious match between sludge-spectra peaks and peaks arising from NaNO_3 (nitrate) and $\text{Na}_2\text{NiFe}(\text{CN})_6$ (ferrocyanide).

Technology Needs

The FTIR-PAS technique is uniquely capable of analyzing solids and sludges retrieved from the underground storage tanks. Virtually no sample preparation and hence a minimum of handling are required for their analyses with FTIR-PAS. The amount of material necessary for FTIR-PAS analysis is quite small, typically about 1 mg, which also reduces the hazards and complexities of the analysis. FTIR-PAS will provide a quantitative compositional analysis of the multi-atomic ions and molecular species in the sludges and solids. These species (ferrocyanide, nitrate, etc.) are of special interest because they pose the potential risk of explosive reactions.

Brookhaven National Laboratory, Westinghouse Hanford and Rocky Flats are developing the polymer encapsulation

Infrared Analysis of Wastes: Novel Lab and On-line Measurements by Photoacoustic and Transient Infrared Spectroscopies

process to immobilize radioactive salts and similar wastes to meet new RCRA requirements. The TIRS monitoring technique will improve the quality of the waste form produced by the encapsulation process and provide documentation of its composition. The real-time information provided by the monitoring system will allow the waste-process operators to maintain the proper salt-to-polymer ratio in the final waste form. This same information can act as documentation of the waste-form composition for certification purposes.

Accomplishments

FTIR-PAS has become a widely used technique for analyzing normally intractable solid samples. It often requires little or no sample preparation and only a small amount of sample. Successful qualitative analysis of milligram-sized samples of surrogate tank sludges has already been demonstrated. Figure 2 shows FTIR-PAS spectra of the surrogate sludges and of pure materials believed to be components in the sludges. Many peaks in the sludge spectra obviously match with peaks from the component spectra. Quantitative tests are now underway.

TIRS has been demonstrated as a real-time, on-line process monitor on the pilot or production lines of several manufacturers. The TIRS results from these demonstrations were as accurate in determining the property of interest as the manufacturers' usual off-line tests. A successful proof-of-principle test of TIRS as a waste-processing monitor has already been completed at Brookhaven National Lab, and optimization of the approach is underway at Ames Lab. Figure 3 shows the correlation between one set of TIRS measurements (infrared absorbances) and the known salt (sodium nitrate) loading in the stream. TIRS was chosen for an R&D 100 Award in 1992.

Collaboration/Technology Transfer

A training session at Hanford is planned to explicitly transfer the FTIR-PAS technology. Frequent communication is planned between Ames and Hanford to transfer expertise and receive feedback. The photoacoustic portion of FTIR-PAS

technology has been commercialized by a small company located near Ames Lab (MTEC Photoacoustics). The project staff has had substantial experience in studying the application of photoacoustics to the needs of many end users.

The application of TIRS technology to waste-process monitoring will be closely tied to the efforts at the user sites to develop the polymer encapsulation process. TIRS can be effectively used to monitor any molecule-based property (e.g., chemical composition, cure level of thermoset polymers and paints, oxidation level of and mineral content in coals) in process streams of solid or viscous-liquid material. The TIRS technology was invented by the project staff and has been patented by Iowa State University. It is available for licensing.

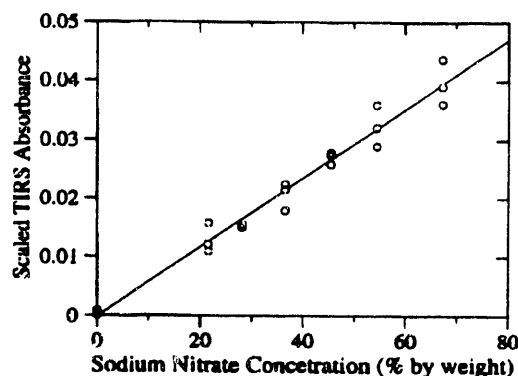


Figure 3. Correlation between the TIRS measurements (absorbance) and the known sodium nitrate loading in the molten salt/polymer process stream. The mean error of the measurements is 3.5 % by weight.

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Development of a Long-Term Post-Closure Radiation Monitor

Task Description

A low cost, multi-point radiation monitoring system will be developed for the long-term, continuous monitoring of radiation levels in the vadose zone of hazardous waste sites. The system will be based on gamma detection and will be capable of installation and monitoring to depths of up to 50 meters below ground level in existing wells and through the use of ground penetrometers. The system will be capable of monitoring large numbers of passive, permanently installed probes. On a component level, the monitor will employ commercially available scintillation material, optics, fiber optics and opto-electronics. A large number of passive probes with individual scintillators will be installed below ground to provide area coverage and these individual probes will be connected via fiber optic cable to above ground multiplexer/detector electronics. This combination of components will form a system that is not commercially available at the present time.

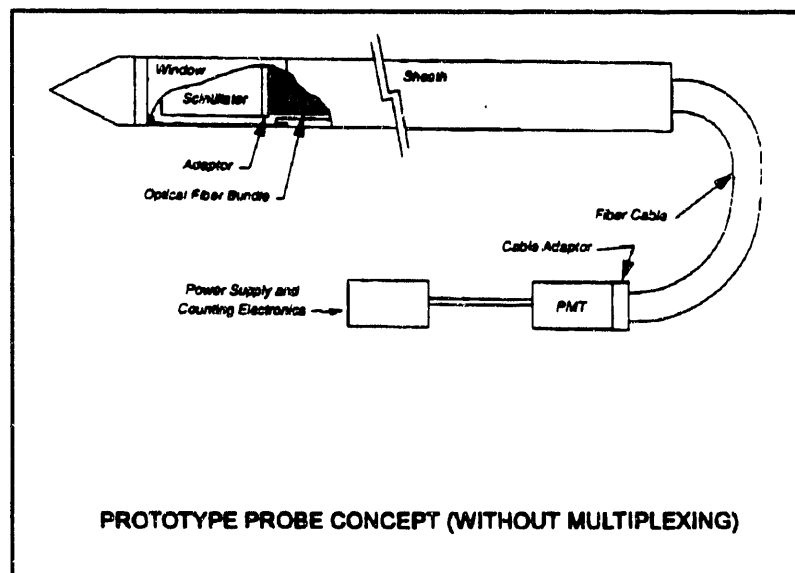
The development program will include the selection and testing of individual components and tests of a laboratory configuration. A single prototype probe will then be designed, fabricated and field tested in soil at a hazardous waste site. Finally, a prototype system consisting of several probes multiplexed to a single opto-electronics detector will be designed, fabricated and subjected to a year-long field test at a hazardous waste site.

Accomplishments

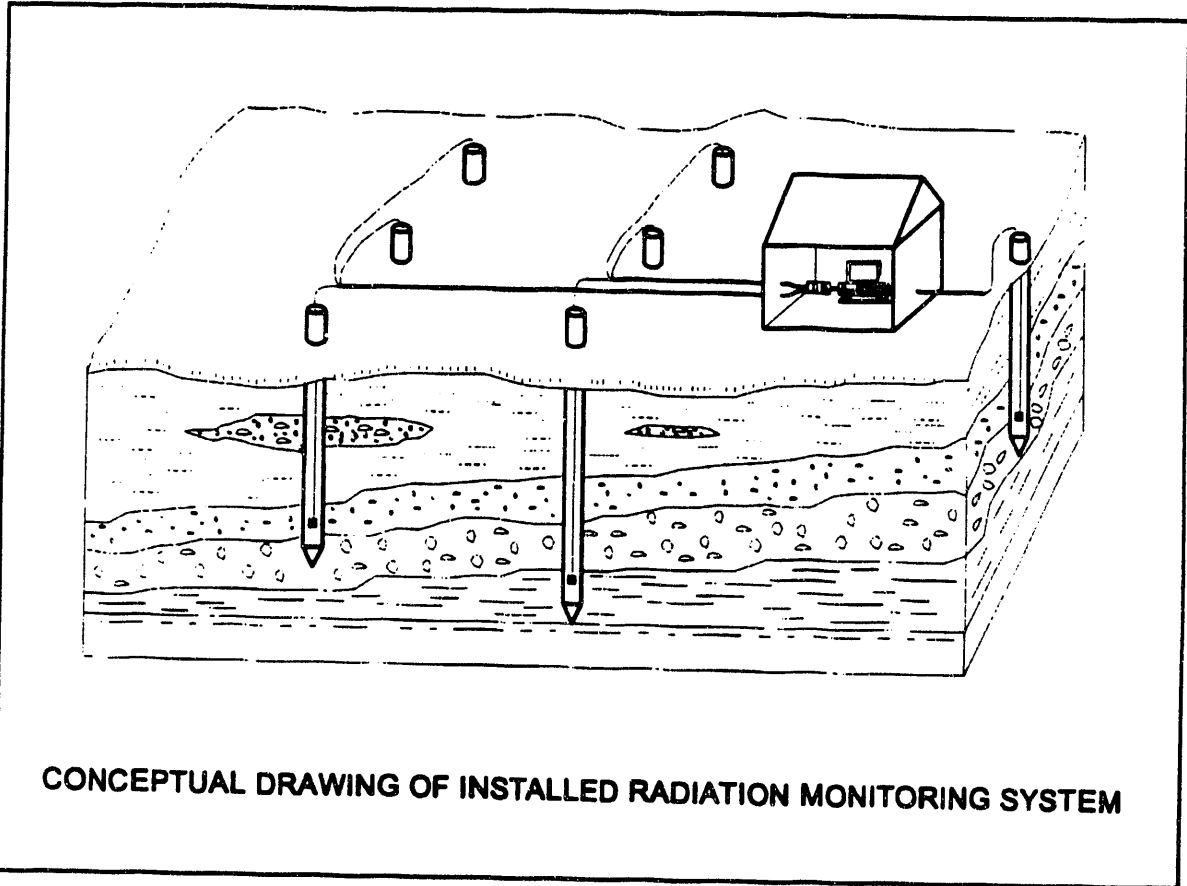
The first of three phases was initiated in October 1992. The initial project planning and contract submittals have been completed. An initial survey of available components has been completed and a parameter study is being performed to select initial design parameters for the probe geometry. Preliminary design concepts are being developed. Contacts have been made and working relationships identified with a scintillator supplier and a penetrometer supplier. The draft test plans for the laboratory testing which will take place in Phase 1 have been issued to the DOE for comments.

Technology Needs

In-situ, long-term, and real-time measurement techniques are needed for monitoring contaminant leakage and migration from underground storage tanks and post-closure landfills.



Development of a Long-Term Post-Closure Radiation Monitor



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ISV Field Data Collection at Arnold AFB and Parsons Superfund Site

Task Description

This task is designed to result in the collection of full-scale operational and performance data during FY-1993 and early FY-1994 in situ vitrification (ISV) operations conducted by Geosafe Corporation at the Parsons Superfund Site in Lansing, Michigan and the Arnold Air Force Base (AFB) in Manchester, Tennessee. In addition, field data will be collected from, and field data collection techniques verified on, an Operational Acceptance Test (OAT) planned by Geosafe Corporation in early FY-1993 as a precursor to the operations at Parsons and Arnold AFB. The field data collected via this task support the resolution of technical issues that are delaying the deployment of the ISV technology for contaminated soil applications within the DOE complex. This field data collection task supports closure of vapor release from unconfined soil, fate of VOCs and attainable depth issues. Closure of these ISV technical issues directly supports the planned demonstrations and RODs at the Hanford 100 B/C Area, Oak Ridge National Laboratory (ORNL) WAG 7 and Hanford 200 BP-1.

This task represents the first dedicated effort to collect full-scale ISV process data during remedial operations. Resulting data from this task will be reduced, interpreted and reported in a format suitable for use by regulators and end users of ISV. Geosafe Corporation, EPA Region IV, EPA Region V and Arnold AFB are supportive of this collaborative effort as this task will produce data that are of benefit to all parties concerned. The resulting data will assist DOE by resolving issues that are currently preventing the deployment of the ISV technology in the DOE complex.

Technology Needs

Contaminated soil within the DOE complex represents a significant waste stream that must be remediated. In general, the remediation of these contaminated soils in place, or in situ, is a preferred alternative in terms of economics and safety to the environment, workers and public. However, for soil contami-

nated with heavy metals, radionuclides and/or mixed waste, there exist a limited number of in situ technologies. Resolution of ISV technical issues for application to contaminated soil will increase the in situ remediation options available to DOE. This task supports the closure of these technical issues by obtaining the field data necessary for an increased understanding of the ISV process that will expand the applicability of the ISV technology to meet DOE remediation needs.

Accomplishments

The test plan that will guide field data collection activities during Geosafe operations was finalized during the first quarter of FY-1993. Included in the test plan is a description of instrumentation that will be used, placement of instrumentation, description of the data acquisition system, and frequency at which data will be collected.

A key component of the field data collection task is the data acquisition system. Configuration of the data acquisition system was completed on schedule at the end of January 1993. The Field Data Collection Data Acquisition System FDC.DAS V1.0 is the data acquisition system for the collection of field data during full-scale Geosafe operations. The system has been designed with built-in flexibility and can be used for all of Geosafe's operation with very little modification for each operation. The current version of the data acquisition system is capable of acquiring, logging and displaying over 150 instrument signals and can be expanded to accommodate additional signals.

Future efforts will focus on the placement of instrumentation, collection of field data, reduction of field data and reporting of results from field data collection activities from each of Geosafe's full-scale operations. The current schedule for full-scale field data collection operations is April 1993 for the Geosafe OAT, August 1993 for Parsons and October 1993 for Arnold AFB.

Collaboration/Technology Transfer

The identification of data needs, and the collection of field data to meet these needs, constitute the technology transfer activity for this task. Workshops held between members of the DOE ISV community and Geosafe Corporation identified the field data needed to resolve technical issues and identified instrumentation to collect this field data. Input from the workshops provided the basis for the field data collection test plan. Technology transfer activities for the field data collection phase of this task continues through the collaboration between this task, Geosafe Corporation and EPA. The final phase of this task will ensure that data information gathered is available through the dissemination of reports and presentations.

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Performance Specifications and Program Support

Technology Performance Specifications

Task Description

This project will establish and implement technology performance specifications for OTD R&D and DT&E projects. Technology performance specifications will be used to establish milestones, evaluate the status of ongoing projects and determine the success of completed projects. Preliminary performance specifications will be required in TTPs and will be evaluated as part of the TTP evaluation. In the first year, a pilot project will focus on chemical and radioanalytical measurement technologies for VOCs. The Principal Investigators (PIs) will prepare a preliminary list of specification topics and values for chemical and radioanalytic measurement technologies. The list will be derived from available sources and resources. Evaluation criteria will address whether the list is complete, whether the values are within regulatory requirements, whether the values are reasonable, and whether the entire system will be workable.

The reviewers comments will be used to revise the specifications, which will then be applied to the three OTD-supported technologies selected by the HQ Manager. The result will be summarized in a report to the HQ Managers, along with draft guidance to facilitate dissemination of specifications to the affected constituents (e.g., PIs, TSG's, etc.). In subsequent years, specifications will be prepared in other technical areas as directed by the HQ Manager.

the current cost of analysis or improvement in analyte detection over established measurement technologies.

We have prepared a list of relevant VOC measurement technologies that includes gas chromatography/mass spectrometry (GC/MS) and various sensors based upon electrochemical, optical, piezoelectric and biological sensing of VOCs. Each of the measurement technologies will be reviewed paying special attention to their application in field measurement campaigns, detection limits for analytes listed in EPA Method 8240 of SW-846, interferences and their speed of analysis, data recovery and data reduction. A draft report on GC/MS instrumentation and operation has been prepared.

Accomplishments

A Performance Specifications Model has been prepared. The first step of the performance specifications process is to state the need of the environmental restoration or waste management customer. The general approach of the model is to document the baseline technologies that currently exist or are nearing completion of their development and to compare the baseline to the customers' needs in order to determine unmet requirements. The unmet requirements form the basis for further technology development. Investigators must be quantitative in assessing their proposed technology's reduction in

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Defining Requirements for Evaluation and Testing of Characterization Technologies

Task Description

This task will establish performance guidelines for, and monitor the progress of, selected OTD geophysical and remote-sensing programs in characterization, monitoring, and sensor technologies. A detailed set of performance specifications will be written and used to review proposals for new projects and evaluate the progress of existing projects.

New project proposals will be examined for both technical and management content. Each experimental plan will be evaluated for overall technical merit by matching the proposed capabilities with the regulatory requirements to determine the probability of success in meeting those requirements. The sensors and their related instrumentation will be evaluated for performance expectation, while the data processing algorithms will be examined for technical merit and applicability. The proposed experimental plans, including schedules, milestones, cost estimates, and ultimate objectives in addition to the performance specifications, will be reviewed for reality and consistency. Similar proposals will also be screened to ensure complementarity and prevent duplication.

The status and progress of existing programs will also be evaluated for both technical and management content. Low-profile, on-site visits will be made, as appropriate, to selected laboratories to enhance the quality of technical assessments. Evaluation criteria for each of the programs will be established for the various measurement technologies to provide standards against which the performance of individual projects can be compared. These criteria will then be used to determine if the measured values meet the regulatory requirements, if the projected milestones have been reached, if the data are both reasonable and realistic, and if the system performed as proposed and anticipated.

The results of these evaluations will be transmitted to the Program Manager for appropriate action.

Technology Needs

The establishment of standards of quality (including performance specifications) for OTD programs will provide principal investigators, DOE customers, and DOE program reviewers with guidelines that can be used to evaluate existing and future OTD proposals for both quality and relevance.

Accomplishments

Note

No response to our request for updated information was received before the printing deadline for this brochure. The material presented here was extracted from the Technical Task Plan, TTP# NV-031002. Please address questions to the Principal Investigator, identified below.

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Non-Intrusive Characterization Program Support

Task Description

This project focused on reviewing nonintrusive techniques that might be used for environmental characterization at DOE waste sites. These reviews were to concentrate on geophysical ground-based and aircraft/satellite remote sensing techniques. Furthermore, techniques that might be brought to the "open" environment from the classified were to be considered. This was to be accomplished by holding a series of workshops and making recommendations based upon those workshops.

The project was to provide DOE a methodology for characterizing subsurface objects (e.g., drums and tanks), pit boundaries, geological features, and air/soil/groundwater contamination. Furthermore, the project was to provide support to the CMST-IP on these topics whenever appropriate.

Technology Needs

Before cleaning up the more than 3,000 identified waste sites at DOE facilities, there is logical and legal imperative first to characterize (describe) the sites as to their geography, type and degree of contamination, and their geology and hydrology. Sites are often characterized, in part, by drilling to gain information directly about the subsurface and its contents. Geophysics and airborne/satellites remote sensing can provide methods for non-intrusive investigation of the subsurface at lower cost and with less risk to the investigators than drilling.

If by developing certain noninvasive techniques incrementally so that drilling costs can be reduced, there is a real potential savings to be gained. In many of the recent calls for proposals, one of the highest priorities for further development is noninvasive techniques, so that cost and risk to personnel can be reduced.

Accomplishments

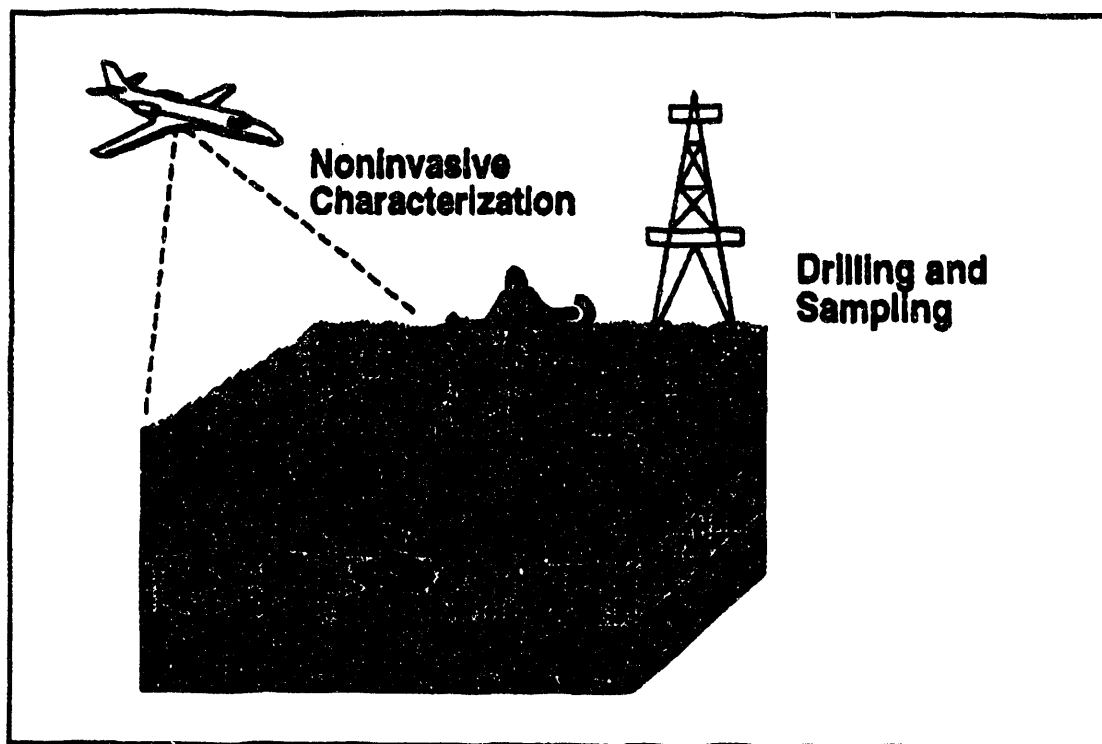
A workshop involving 58 participants was conducted in August of 1991. Participants were asked to discuss DOE's needs for waste site characterization, and the current status of noninvasive geophysical technology available to satisfy these needs. The initial discussion focused on electromagnetic, ground penetrating radar, seismic, gravity, and magnetic methods. However, an additional technology, "data fusion" arose out of these discussions and was added to the list. The ultimate ranking of these technologies (i.e., those that would provide the most benefit to the DOE at the most sites, and for the least cost—1 being best) was as follows:

1. data fusion,
2. electromagnetic,
3. ground penetrating radar,
4. seismic,
5. gravity, and
6. magnetic methods.

A report summarizing these results was subsequently written and is available.

A two-day workshop was convened in May of 1992 in Washington, D.C. to assess the value of using both classified and unclassified remote-sensing systems in meeting the DOE's waste site characterization/remediation needs. The meeting was attended by 62 members of the remote sensing community. It's recommendations were:

1. A dedicated group must be established to provide leadership in addressing the problems.
2. A common understanding of site-based needs and remote sensing capabilities must be further established.
3. Database management/fusion systems must be established to exploit existing data and incorporate new findings.
4. Permission for its use of the GPS Precision Position System on DOE site work should be requested by DOE through the ASDC31.
5. Sensible investment for mid- to long-term sensor system upgrade and/or development is recommended for high priority technology discussed in the workshop.
6. Near-term funding is necessary for demonstrations using multiple sources of information.
7. Common formatting of data is a very important consideration and should be incorporated on a DOE-wide or even U.S. government-wide basis.
8. Long term planning and multi-year funding should be developed.



-
9. Common language for the various steps of the site by site waste hazard assessment process should be developed and consistently utilized (e.g., discovery, waste characterization, waste remediation, waste monitoring, apart from site characterization).

This workshop has been summarized and is available to properly cleared personnel.

Support has been provided to the CMST-IP in the implementation and execution of these recommendations.

For more information, please contact:

Collaboration/Technology Transfer

The recommendations mentioned above have only been attained by the collaboration of many industrial, university, and governmental organizations and personnel. Please consult the reports and minutes from these workshops for a detailed list.

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Invited participants at
U. S. DOE
Office of Environmental Restoration
and Waste Management
Office of Technology Development
R&D/DT&E Mid-Year Review

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END

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