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AMES LABORATORY

**Uranium in Soils Integrated Demonstration
Site Characterization
at
Fernald, Ohio
September 1992**

**Report of Uranium Concentrations in Soil
Determined by *In Situ* LA-ICP-AES**

Report Date
February 2, 1993

Ames Laboratory
Iowa State University
Ames, Iowa 50011

Prepared for
The U.S. Department of Energy
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Uranium in Soils Integrated Demonstration Site Characterization at Fernald, Ohio September 1992

Report of Uranium Concentrations in Soil Determined by *In Situ* LA-ICP-AES

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February 2, 1993

MASTER

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New Inorganic Field Screening Technique

Laser Ablation - Inductively Coupled Plasma - Atomic Emission Spectroscopy

LA-ICP-AES

**Report ---- Uranium in Soils Integrated Demonstration Site Characterization at Fernald, Ohio
September 1992**

The attached report has been prepared by the staff of the Technology Integration Program Office and the Analytical Chemistry Group at the Ames Laboratory. It reports on a very significant project activity undertaken during FY92 and which continues into FY93.

We know of no other "fieldable" system that can perform an in-situ, elemental analysis exhibiting the utility, speed, economy, and low detection thresholds of this system. Physical samples are not handled by the operator so the goal of improved safety is also achieved. With the first version of the system, we are able to conduct a screening for some 20 elements at a surface site in about 15 minutes and make the results available for immediate use. The list of elements being analyzed can be easily expanded or changed. A map of the contaminate concentrations can be prepared, in the field, from stored data.

A truck and trailer are moved onto a site and temporarily parked. The trailer contains all of the computational and analytical equipment, and all of the required utilities including a generator for electrical power. The mobile unit is self-contained and highly automated. Operating at the end of a 100' umbilical cable, the active ablation cell can be positioned to take a sample for analysis anywhere within an area of approximately 31000 ft² without requiring that the trailer be moved.

Detection thresholds vary by element but are generally in the 10's of ppm range. The threshold of the system for natural abundance uranium, for example, is on the order of 20 ppm -- approximately 14 pico Curies/gram (pCi/g).

Not only is the system highly sensitive and fast, it is also more economical than conventional sampling and laboratory analytical procedures for low level radioactive samples. It has been estimated that the current system could be fielded for about \$500 per sample in a commercial, for-profit production setting.

That's the entire estimated cost including labor, utilities, weather down time, maintenance, holidays, depreciation, vacations, typical corporate overheads, and profit.

But more good news is reflected in the fact that we know enough to design and build a second version that will be even better from the standpoint of cost, speed, reliability, repeatability, sensitivity, and accuracy. And we are working on a down-hole version to conduct sub-surface screenings. Plus, we plan to add GPS so that true map registered overlays can be prepared from data acquired during what we refer to as the "roaming around mode".

This system worked the first time out in the field and after only about 15 months of development -- engineering design, fabrication, and testing. We are already far enough up the learning curve to postulate that commercial operations could be undertaken within the next three to four years. Those who understand the typical time required to transition technical developments of this magnitude into commercial products and practice will recognize this as being a remarkably short development cycle.

Better, safer, faster, and cheaper has been achieved. Several DOE sites are currently looking at this system as are several other federal and state agencies. We are enthusiastic about this development and encourage your comments and inquiries. Please write to the address shown below or call the Technology Integration Program Office at (515) 294-6404. Our FAX is (515) 294-6963.

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The following two papers were presented at Spectrum '92, Boise, ID
August 23-27, 1992

- I Remote, Real-Time Analysis of Hazardous
 Wastes Through Laser Ablation-Inductively
 Coupled Plasma-Atomic Emission Spectrometry
- II Mobile Instrumentation Platform and Robotic
 Accessory for Real-Time Screening of Hazardous
 Waste

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Uranium in Soils Integrated Demonstration Site Characterization Fernald, Ohio September 1992

Summary

Laser ablation - inductively coupled plasma- atomic emission spectrometry (LA-ICP-AES) was used for in situ determination of uranium and thorium concentrations in soil at 80 sampling sites in the Sewage Treatment Plant area. This work was performed by the Environmental Technology Development Program of the Ames Laboratory using a completely self-contained mobile laboratory. This laboratory, the *mobile demonstration laboratory for environmental screening technologies (MDLEST)* and the *robotic sampling accessory (RSA)*, were designed and constructed by the Ames Laboratory during FY 1992. The instrumentation is capable of analyzing each sample for twenty operator-defined elements simultaneously.

Using the MDLEST/RSA, the uranium concentrations in the soil at the 80 sampling sites were found to range from < 20 parts-per-million (ppm) (< 13.5 pCi/g) to 303 ppm (205 pCi/g). The 95% confidence interval for these field determined values range from 80 to 110 ppm. Bore hole samples from two sites were analyzed. No measurable uranium concentration was detected below the one foot depth. Seven samples taken from sites within an area currently under remediation were analyzed and found to contain uranium concentrations ranging from 101 ppm (68.3 pCi/g) to 788 ppm (532 pCi/g).

Soil samples were taken from twelve of the 80 sampling sites in the field, using conventional sampling techniques. These samples were prepared by microwave digestion, using the wet chemistry capability in the MDLEST, and field analyzed using solution nebulization ICP-AES. The laboratory procedure followed for microwave digestion required the samples to be diluted by a factor of 100. This dilution resulted in uranium intensities too low to be accurately quantitated in the field. Optimization of the instrumentation and sample preparation will make this field capability useful in determining near real-time the soil matrix, and enable the performance of this quality assurance process in the field with greater sensitivity and accuracy.

In addition to the in situ field work conducted at Fernald, fifteen samples were selected from the 80 sampling sites and taken to the Ames Laboratory for subsequent analysis and comparison with the field results. Each sample was split and analyzed using two different methods to determine the uranium concentration. A laboratory LA-ICP-AES system was the first method used to determine the uranium concentration giving values ranging from < 20 ppm (13.5 pCi/g) to 101 ppm (68.3 pCi/g) with a 95% confidence interval of approximately 50 ppm. The second method used solution nebulization ICP-AES and the technique of standard addition of uranium to give uranium values ranging from 19 ppm (< 12.8 pCi/g) to 124 ppm (83.8 pCi/g) with a 95% confidence interval of approximately 10 ppm. The laboratory-determined values for eleven of the fifteen samples agree, within the uncertainty of the determined concentrations, with the field determined values. In general, the field determinations of uranium concentrations gave higher values, by a factor of two

to three, than the laboratory-determined values. These larger than expected uncertainties are in part due to the spectrometer, a conventional laboratory instrument, operating in a less than ideal mobile laboratory environment. With suitable modifications to the spectrometer, these uncertainties may be minimized.

The MDLEST/RSA was also used to determine the thorium concentrations in the soil at the 80 sampling sites. Thorium was detected in a number of samples, but since the concentrations were < 20 ppm which was below the field quantitation limit of the currently configured LA-ICP-AES system, these results are not reported. The fifteen samples that were returned to Ames were analyzed for thorium using solution nebulization ICP-mass spectrometry. The results showed thorium concentrations in excess of 1 ppm but less than 10 ppm.

The sampling point identification and spectrometer results are printed out in report format immediately after taking the data. A more complete record including these results along with instrumentation settings, pressures, temperatures, etc. are recorded in data files on an optical disk for archiving. Thus, we have created an electronic chain-of-custody for sample quality assurance.

While the MDLEST was developed over a very short time period, the system worked extremely well during field operations. A number of improvements are planned for the MDLEST/RSA that will increase the precision of the analytical determinations and enhance the field mobility of the system. Likewise, operational procedures will be optimized to increase the efficiency and reduce the size of the team needed to staff the MDLEST.

Mobile Demonstration Laboratory for Environmental Screening Technologies

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1. Air Handling System
2. Diesel Fuel Storage Tank
3. Diesel Generator
4. Cryogenic Gas Storage
5. Lavatory
6. Overhead Storage
7. Workspace
8. Sample Preparation Area
9. Electronics Equipment Rack
10. Spectrometer
11. System Control Center
12. Neslab Chiller
13. Laser Mount
14. Laser Power Supply

Robotic Sampling Accessory

Ames Laboratory
Technology Integration Program (TIP)

Introduction

Ames Laboratory, under its Environmental Technology Development and Technology Integration Programs, has developed a mobile demonstration laboratory for environmental screening technologies (MDLEST) and robotic sampling accessory (RSA) for in-situ, real-time screening of hazardous wastes. This project is part of the effort by the U.S. Department of Energy to characterize and remediate its wastes sites. The analytical characterization technology demonstrated was remote laser ablation-inductively coupled plasma-atomic emission spectrometry (LA-ICP-AES). The operation of the system was demonstrated as part of the Uranium in Soils Integrated Demonstration (USID) at the Fernald Environmental Management Project, Fernald, Ohio. The Integrated Demonstration at Fernald involved a number of analytical technologies designed to aid in the characterization and remediation of uranium contaminated soils at the Fernald Plant. The characterization technologies included high resolution gamma spectroscopy (Pacific Northwest Laboratories), wide area beta scintillation counting (Pacific Northwest Laboratories), long range alpha detection (Los Alamos National Laboratory), and laser ablation-inductively coupled plasma-atomic emission spectrometry (Ames Laboratory).

Real-time field analyses by LA-ICP-AES for the determination of uranium in soils was demonstrated at the Sewage Treatment Plant area at Fernald, September 18 through September 27, 1992. Uranium concentrations in the soil at a total of 80 locations (on a predetermined grid) in the Sewage Treatment Plant area were determined by analyzing the soil surface using the LA-ICP-AES technique. This is a report on the analytical data obtained during these tests.

Instrumentation

The MDLEST and RSA comprise a mobile laboratory that houses all the equipment necessary to analyze soil samples for various contaminants (e.g., uranium, thorium, lead, and chromium) by LA-ICP-AES. Laser radiation from a frequency doubled Continuum NY81-20 Nd:YAG laser (housed in the MDLEST) is transmitted through a 25 meter long fiber optic cable to a remote site that is to be sampled. The output from the optical fiber is focussed onto the surface of the soil that is to be analyzed; the interaction of the laser with the soil surface causes material to be ablated from the soil surface. The ablated material is transported out of the ablation cell by argon gas flowing through a 20 meter length of tubing to an RF Plasma Products ICP (on the RSA) which dissociates, atomizes, ionizes, and excites the ablated soil sample, producing atomic and ionic optical emission (light) characteristic of the constituents present in the soil sample. The emitted light is coupled into a 12 meter long fiber optic bundle that is routed back to the MDLEST to a Thermo Jarrell Ash (TJA) ICAP 61E spectrometer. The ICAP 61E is a twenty channel simultaneous spectrometer that enables the detection and quantitation of the emission signals for nineteen different elements. Two of the channels of the ICAP 61E are utilized for determining uranium. The light emitted by the ICP is also coupled through a nine meter long fiber optic bundle to an Acton Research 0.5 meter spectrometer located in the MDLEST. This spectrometer is equipped with an EG&G Princeton Applied Research intensified photodiode array that is used to detect ICP emission signals for a ~10 nanometer region of the spectrum. Figure 1 is a two nanometer segment of the LA-ICP-AES spectrum obtained for site sample M2 in the Sewage Treatment Plant area at Fernald (upper trace)

Figure 1. A two nanometer (nm) segment of the LA-ICP-AES spectrum. Upper trace, data from the Fernald Sewage Treatment Plant Area sample site M2. The lower trace, a Fernald soil blank sample that contains no uranium. The 409 nm uranium line is shown.

and for a Fernald soil blank sample that contains no uranium (lower trace). The uranium 409.0 nm emission line is labelled.

The mobile laboratory is self-sufficient, providing all the utilities (power, electric, gases, water) required to perform LA-ICP-AES analyses. The MDLEST houses the majority of the analytical instrumentation, including the laser, spectrometer, computers, power supplies, and electronics. The RSA is a platform for an ICP torch and a robotic arm that can be used to place an end effector at the soil site to be sampled. An operator inside the MDLEST controls the robotic arm to position the end effector at the site to be analyzed. The end effector contains the ablation cell, an xyz stage, optics, and the output end of the laser fiber optic cable. The ablation cell has a circular knife edge that penetrates into the soil around the site to be analyzed. A gas-tight seal is formed by applying water to the soil surface so that the ablated soil can be swept into the ICP for analysis. The z-axis of the xyz stage and the optics inside the end effector enable the laser fiber optic output to be focussed onto the surface of the soil. The x- and y-axes are used to raster the focussed laser beam in a spiral pattern over an area of approximately one inch square. The focussing and rastering systems are operated remotely, under computer control, from the MDLEST. The end effector can be detached from the robot arm and manually positioned on the sampling surface up to 50 feet from the MDLEST. This was the predominant mode of operation for the analyses performed at Fernald.

The field sample identification, instrumentation settings, and spectrometer results were recorded in files on a *write once read many* (WORM) optical disk. This procedure prevents any alteration of the data once it has been taken and written on the disk.

The experimental conditions used for the LA-ICP-AES analyses performed at Fernald are given in Table I. Appendices I and II provide more detailed information regarding the LA-ICP-AES technique and the operation and capabilities of the MDLEST and RSA, respectively.

Analysis of Soil Samples by LA-ICP-AES

The response of the LA-ICP-AES technique was calibrated for uranium (U) and thorium (Th) prior to analyzing any of the sites in the Sewage Treatment Plant area at Fernald. This was done by analyzing U- and Th-containing soil standard samples. The samples used for calibration consisted of a Fernald soil blank and two U- and Th-containing synthetic standards, 100 parts-per-million (ppm) U and Th and 1000 ppm U and Th in Fernald soil. The Fernald soil blank was a soil sample containing no uranium or thorium, obtained off-site in the vicinity of the Fernald Plant, which was dried in a drying oven and then sieved to pass through a number 25 sieve. The 100 and 1000 ppm U and Th in Fernald soil synthetic standards were prepared by adding the appropriate volumes of uranium and thorium spectrometric standard solutions (National Institute of Standards and Technology standard reference materials 3159 and 3164) to one hundred gram quantities of the sieved Fernald soil blank, weighed to the nearest milligram into polyethylene bottles. The resulting slurries were mixed and subsequently dried in a drying oven. The dried U- and Th-spiked soil samples were transferred to an agate mortar and pulverized to obtain a fine mixture. The samples were transferred into polyethylene bottles and thoroughly mixed prior to analysis by LA-ICP-AES.

Standards were analyzed by placing the sample into a one ounce polyethylene container that was put into the standards sampling station on the RSA. The end effector was subsequently

Table I. Experimental Conditions for Laser Ablation ICPAES

Fiber Optic Delivered Laser Radiation:

Laser	Continuum NY81-20 Nd:YAG
Wavelength	532 nm
Repetition rate	20 Hz
Pulse duration	7 nsec
Fiber optic cable length	25 meter
Fiber optic cable core diameter	600 μm

Ablation Conditions:

Fiber optic output energy (at sample)	3 mJ
Average output power	60 mW
Peak power density at sample	100 MW/cm ²
Laser spot diameter at sample	600 μm
Transport tubing	20 meter length, 3/16" I.D.

Inductively Coupled Plasma:

ICP system	RF Plasma Products ICP-16L
RF frequency	40 MHz
RF power	1.1 kW
Plasma gas flow rate	16 L/min
Auxiliary gas flow rate	0.8 L/min
Sample gas flow rate	1.0 L/min
Imaging	Emission coupled to ICAP 61E via a 12 meter fiber optic bundle

Spectrometer and Detection System:

Thermo Jarrell Ash model ICAP 61E
twenty channel simultaneous ICP spectrometer

<u>Element</u>	<u>Wavelength (nm)</u>	<u>Element</u>	<u>Wavelength (nm)</u>
Aluminum	308.215	Scandium	361.384
Barium	455.403	Silicon	288.158
Calcium	317.933	Sodium	588.995
Chromium	267.716	Strontium	421.552
Iron	259.940	Thorium	401.913
Lead	220.353	Titanium	337.280
Magnesium	383.826	Uranium	409.014
Manganese	293.930	Uranium	424.167
Phosphorus	213.618	Vanadium	292.402
Potassium	769.896	Zirconium	343.823

positioned on the standards sampling station prior to LA-ICP-AES analysis. Calibration curves for U and Th were obtained by analyzing the Fernald soil blank, the 100 ppm U and Th standard and the 1000 ppm U and Th standard samples. Optical emission signals for U and Th (the peak line intensity minus the intensity at a selected background correction point) were determined using the TJA ICAP 61E spectrometer. This net analyte emission intensity was ratioed to the emission intensity of a soil matrix element (silicon and iron were both used as internal reference elements) in order to generate intensity ratio vs. concentration calibration curves for U and Th. Uranium and thorium concentrations for the sites analyzed were determined from these calibration curves.

After the LA-ICP-AES technique was calibrated, site samples in the Sewage Treatment Plant area were analyzed. The process typically involved the following steps. The MDLEST and RSA were driven to a position within the Sewage Treatment Plant area such that up to four different sites could be analyzed by manually moving the end effector from one site to another. After the MDLEST and RSA were in position, the 100 ppm U and Th in Fernald soil standard was analyzed as a quality control sample. This quality control sample was used to normalize the calibration curve prior to analyzing the site samples. Prior to moving the MDLEST and RSA to the next set of sites to be analyzed, the 100 ppm U and Th in Fernald soil sample was analyzed again. The Fernald soil blank sample was analyzed by LA-ICP-AES after every ten site samples analyzed to determine if carry-over or memory effects were occurring. For approximately every tenth site analyzed, soil samples were collected by Fernald personnel for subsequent microwave digestion and ICP-AES analysis. This was done so that the results obtained by LA-ICP-AES could be compared to those obtained using conventional solution nebulization ICP-AES, an EPA accepted method of analysis for 26 elements.

Analysis of Soil Samples by Microwave Digestion ICP-AES

The MDLEST has an on-board capability for processing soil samples by microwave dissolution procedures. The dissolved soil samples are subsequently analyzed by solution nebulization ICP-AES using the TJA ICAP 61E spectrometer in the MDLEST. The procedure utilized during the Integrated Demonstration at Fernald is given below.

Soil samples were collected and prepared by Fernald personnel for approximately every tenth sample site that was analyzed by LA-ICP-AES. Roughly 35 grams of topsoil were removed from each sample site and prepared. Sample preparation involved removing organic debris manually, drying the sample overnight, and subsequently pulverizing the sample using a ball mill. No further manipulation of the samples was carried out prior to dissolution.

The method used for microwave assisted acid digestion was developed using CEM application note OS-14 and EPA Method 3051 as guidelines. Approximately one gram of the soil sample (weighed to the nearest milligram) was transferred to a digestion vessel; an acid mixture consisting of hydrofluoric acid, hydrochloric acid and nitric acid was added to the vessel. In addition, a known quantity of a scandium solution was added as an internal reference. The vessel was then sealed and placed inside a microwave oven (CEM model MDS 2100) and subjected to microwave radiation, which produces elevated temperatures and pressures inside the vessel to facilitate dissolution of the soil sample. The microwave assisted acid digestion consisted of a two step cycle. In the first step the temperature and pressure were brought up slowly to 40° C and 40 psi, respectively, and maintained for 20 minutes. This allowed for decomposition of organics in the sample, yet prevented

a rapid build-up of pressure in the sealed vessel and possible rupturing of the vessel. In the second step, the temperature and pressure were raised to 170° C and 120 psi and maintained for 30 minutes to allow digestion of the inorganics in the sample. After the digestion was completed, a saturated solution of boric acid was added to the resulting solution to complex "free" fluoride ions. The solution was then transferred to a polypropylene container and subsequently analyzed by conventional solution nebulization ICP-AES procedures (EPA Method 6010) using the TJA ICAP 61E spectrometer in the MDLEST.

Field Acquired Data and Results Preservation

The field sample identification, instrumentation settings, and spectrometer results were recorded in files on a *write once read many* (WORM) optical disk. This procedure prevents any alteration of the data once it has been taken and written on the disk. The WORM is organized into a series of directories and files as shown in Figure 2. Under each daily directory there is a log file that contains date and time stamps for each sample or each sample attempt that was made. The data for aborted samples also is included as far as the sampling had progressed before being aborted. Under the log file there are sample directories that contain a history file and three data files. The history file is a "trace" of the sampling program execution and, therefore, can be very long and difficult to understand. The purpose of the history file is to provide the capability to retrace the process, instrumentation settings, times, etc. of collecting the sample. The three TJA data files are generated from the spectrometer operating software supplied by the spectrometer manufacture. The .DBF data file is a dBASE format file that contains the spectrometer results for the point sampled and can be easily read by most database and spreadsheet programs, e.g. Fox Base, Lotus 1-2-3, and Quattro Pro. The other two TJA data files, .NDX and .SBF, contain report formatting information, not sample information. The log and history files are ASCII text files and can be read by any text editor or word processing software.

The data on the WORM can only be read using a Panasonic LF-7010 Optical Drive or equivalent. This is important since there is not a common agreed upon format for recording information on optical disks. There are two WORM optical disks with the Fernald site data, one disk is retained at the Ames Laboratory's Technology Integration Program office, and the second disk was delivered to the Uranium in Soils Integrated Demonstration (USID) director at the Fernald, Ohio site.

Results and Discussion

The LA-ICP-AES technique was performed on sites prepared as specified in the USID Demonstration Plan. The sampling areas were prepared by the removal of vegetation and rocks down to pebble size. The top layer of sod was removed. The sites were prepared on a 20 x 20 meter grid starting in the southwest corner of the sampling area near the Sewage Treatment Plant. These sites are referenced by sample names which consist of a letter which ascends from south to north (A-Y) and a number which ascends from west to east (1-11). The state coordinates for these sampling sites are provided in Table II. Not all sampling areas on this grid were made accessible to our team. Some of the points were located inside the Sewage Treatment Facility and some were

Figure 2. Optical disk file format.

Table II.

Sample Name	State coordinates	
	East	North
A 1	1382680.831	479735.913
A 2	1382741.091	479734.389
A 3	1382801.392	479732.875
A 7	1383042.072	479726.589
A 8	1383102.050	479724.888
A 9	1383162.356	479723.437
A 10	1383222.626	479722.403
A 11	1383283.098	479720.549
C 1	1382682.987	479796.054
C 2	1382742.771	479794.640
C 6	1382983.513	479788.299
C 7	1383043.719	479786.963
C 8	1383103.775	479785.249
C 9	1383164.197	479783.785
C 10	1383224.139	479782.162
C 11	1383284.429	479780.885
E 1	1382685.331	479856.364
E 2	1382745.056	479854.902
E 6	1382985.275	479848.614
E 7	1383045.190	479847.065
E 8	1383105.451	479845.577
E 9	1383165.714	479843.973
E 10	1383225.832	479842.512
E 11	1383286.069	479840.938
G 1	1382686.397	479916.836
G 2	1382745.768	479915.294
G 3	1382805.264	479913.379
I 1	1382687.444	479976.792
I 2	1382747.415	479975.757
I 3	1382807.911	479973.947
K 1	1382688.720	480037.381
K 2	1382749.050	480035.504
K 3	1382809.518	480033.915
M 1	1382690.313	480097.344
M 2	1382751.075	480095.973
M 3	1382810.974	480094.537
O 1	1382692.180	480157.068
Q 2	1382754.426	480216.197
Q 3	1382814.662	480214.750
S 1	1382695.388	480277.560
S 2	1382755.809	480276.161
S 3	1382816.118	480274.769
S 4	1382876.372	480272.856
S 5	1382936.402	480271.552
S 9	1383176.485	480265.338
S 10	1383237.318	480263.836
S 11	1383298.039	480262.197

Table II continued.

Sample Name	State coordinates	
	East	North
U 1	1382697.122	480337.812
U 2	1382757.413	480336.401
U 3	1382817.835	480334.830
U 4	1382878.144	480333.411
U 5	1382938.318	480331.754
U 6	1382998.433	480330.194
U 7	1383056.141	480327.969
U 8	1383118.785	480327.072
U 9	1383180.280	480325.322
U 10	1383240.366	480323.943
U 11	1383299.377	480322.254
W 1	1382698.396	480398.109
W 2	1382759.101	480396.376
W 3	1382819.129	480395.088
W 4	1382879.286	480393.072
W 5	1382939.588	480392.183
W 6	1382999.806	480390.546
W 7	1383060.117	480389.044
W 8	1383120.068	480387.741
W 9	1383180.528	480386.020
W 10	1383240.796	480384.537
W 11	1383300.997	480383.213
Y 1	1382700.204	480458.566
Y 2	1382760.875	480456.967
Y 3	1382821.399	480455.644
Y 4	1382880.843	480453.878
Y 5	1382941.098	480452.330
Y 6	1383001.231	480450.998
Y 7	1383061.396	480449.325
Y 8	1383121.551	480447.851
Y 9	1383181.837	480446.244
Y 10	1383242.006	480444.636
Y 11	1383302.245	480443.306
<hr/>		
x 0.0-0.5	1383098.153	480326.962
x 0.5-1.0		
x 1.5-2.0		
x 2.0-2.5		
z 0.0-0.5	1382809.849	480114.040
z 0.5-1.0		

fenced off in preparation for remediation. In addition, one point (Q1) was submerged in a swampy area and was therefore inaccessible to the MDLEST and RSA, making sampling impossible.

Determinations of the concentrations of U and Th in the soil at the sampling sites were performed by comparing intensities of two uranium emission lines (409.0 and 424.1 nm) and one thorium emission line (401.9 nm) to that of a silicon line (288.1 nm) using the TJA ICAP 61E spectrometer. The concentration of silicon in the soil was assumed to be constant across the sampling area and equal to that in the prepared calibration standard samples. Determinations were also performed by comparing the intensities of the two uranium lines and the thorium line to an iron emission line (259.9 nm). However, the relative amount of iron in the soil at the sites analyzed was found to vary by as much as a factor of two throughout the site. Therefore, only values obtained from the silicon calibrations are reported here.

Thorium

The levels of thorium determined at all of the sampling locations were below the LA-ICP-AES quantitation limit as determined in the field (<20 ppm). Spectral information obtained on-site through wavelength scans using the TJA spectrometer revealed the presence of Th in some samples, but no accurate quantitation was possible at these low levels. Subsequent analysis of solutions resulting from microwave digestion of some of the site samples by ICP-mass spectrometry at Ames Laboratory revealed concentrations in excess of 1 ppm Th but less than 10 ppm. Because of the low levels of thorium in the soil at the site, accurate quantitation in the field was not possible, so no values are reported here.

Uranium

During the course of the demonstration, values for the uranium concentration determined using the 424.1 nm line were observed to differ considerably from values determined using the 409.0 nm line. At times the values determined using the 424.1 line were considerably large and negative. Examination of a wavelength scan of this spectral region revealed an interference at the wavelength chosen as the background subtraction point. When this interfering element was present at relatively high concentrations, the calculated difference would lead to a negative value for the uranium concentration. Therefore, the uranium concentrations determined using the 424.1 line were unreliable, and the uranium concentrations reported are those determined using the 409.0 nm line.

The measured values for the uranium concentrations of the sites analyzed are reported in Table III. The uranium concentrations are reported in parts-per-million (ppm) by weight. For each site analyzed, three consecutive determinations were made of the uranium concentration. Therefore, the relative standard deviation (RSD) is reported in ppm as a measure of the precision of the determination. This value is also reported as a percent of the measured uranium concentration (% RSD in Table III). In addition to the error due to repeatability in the determination for an individual sample site, which is reflected in the RSD, there is additional error in accuracy due to uncertainties in the calibration procedure. This error (the uncertainty in the calibration) is the limiting factor in determining low concentrations and sets the limit of detection for uranium (as determined in the field) at approximately 20 ppm. The 95% confidence interval (95% c.i.) for the determined uranium

**Table III. Uranium Concentrations in Parts-Per-Million
From Uranium to Silicon Intensity Ratios**

Sample Name	Value (ppm)	RSD (ppm)	RSD%	95% c.i. (ppm)	Max. Value (ppm)	Calculated Activity (pCi/g)
A1	<20	---	---	---	---	<14
A2	<20	---	---	---	---	<14
A3	48	3	6	85	133	32.6
A7	74	6	7	86	160	49.7
A8	49	7	14	88	137	33.0
A9	<20	---	---	---	---	<14
A10	34	3	10	85	119	22.8
A11	25	4	15	86	111	17.0
C1	<20	---	---	---	---	<14
C2	21	10	50	92	113	13.9
C6	<20	---	---	---	---	<14
C7	<20	---	---	---	---	<14
C8	56	1	2	83	138	37.6
C9	31	3	9	85	116	21.2
C10	43	4	10	86	128	28.8
C11	72	21	29	102	173	48.4
E1	35	12	35	94	128	23.4
E2	31	7	22	89	119	20.7
E6	<20	---	---	---	---	<14
E7	21	0	1	82	104	14.5
E8	26	1	2	83	109	17.8
E9	54	2	3	83	137	36.5
E10	74	5	6	86	160	50.3
E11	58	11	20	93	150	38.9
G1	<20	---	---	---	---	<14
G2	45	2	5	84	129	30.4
G3	33	13	38	95	128	22.4
I1	83	5	6	86	169	56.3
I2	80	4	4	84	164	54.1
I3	34	1	3	83	117	23.1
K1	142	4	3	84	225	95.9
K2	157	1	1	80	236	105.7
K3	112	5	5	85	197	75.7
M1	76	3	3	83	159	51.1
M2	285	11	4	88	373	192.8
M3	97	18	19	99	196	65.4
O1	125	5	4	85	209	84.3
Q2	53	8	15	89	142	35.6
Q3	58	3	5	84	142	39.2
S1	78	3	3	83	161	52.9
S2	<20	---	---	---	---	<14
S3	103	4	4	84	187	69.5
S4	108	4	4	84	193	73.2
S5	112	5	4	85	196	75.3
S9	113	7	6	87	200	76.4
S10	142	27	19	107	248	95.8
S11	66	24	36	105	170	44.4

Table III. (continued)

Sample Name	Value (ppm)	RSD (ppm)	RSD%	95% c.i. (ppm)	Max. Value (ppm)	Calculated Activity (pCi/g)
U1	84	13	1	93	177	56.7
U2	100	5	5	85	185	67.6
U3	117	3	2	83	199	78.7
U4	133	8	6	87	220	89.6
U5	119	2	2	82	201	80.5
U6	133	4	3	83	216	89.6
U7	224	2	1	79	303	151.4
U8	314	12	4	89	403	212.2
U9	122	3	2	83	205	82.5
U10	88	5	5	85	174	59.8
U11	71	8	11	89	159	47.7
W1	130	4	3	83	214	88.1
W2	88	5	6	86	174	59.7
W3	104	5	5	85	189	70.3
W4	34	14	40	96	130	23.2
W5	217	13	6	91	308	146.4
W6	87	19	22	100	186	58.6
W7	87	6	7	87	174	58.8
W8	178	2	1	80	258	120.5
W9	127	9	7	88	215	85.5
W10	90	5	6	86	176	61.0
W11	180	4	2	83	263	121.7
Y1	113	3	3	83	196	76.0
Y2	165	9	6	88	253	111.6
Y3	60	4	7	85	145	40.3
Y4	68	10	14	90	158	46.0
Y5	72	14	19	95	166	48.5
Y6	147	5	3	84	231	99.5
Y7	303	11	4	88	392	205.0
Y8	228	11	5	88	317	154.3
Y9	218	4	2	82	300	147.4
Y10	116	2	2	82	198	78.6
Y11	267	7	3	84	352	180.6
asi 1	582	39	7	118	699	392.9
asi 2	546	5	1	84	630	369.0
asi 3	144	8	6	87	231	97.2
asi 4	101	4	4	84	185	67.9
asi 5	483	5	1	82	565	326.3
asi 6	788	1	0	85	873	532.6
asi 7	348	6	2	83	430	234.8
x 0.0-0.5	169	3	2	82	251	114.0
x 0.5-1.0	25	2	7	84	109	17.0
x 1.5-2.0	<20	---	---	---	---	<14
x 2.0-2.5	<20	---	---	---	---	<14
z 0.0-0.5	<20	---	---	---	---	<14
z 0.5-1.0	<20	---	---	---	---	<14

concentration is given in Table III. The sum of the 95% c.i. and the measured value gives an upper limit for the uranium concentration at each sample location, which is reported in Table III as the maximum value.

The Integrated Demonstration has requested that the uranium concentrations determined by LA-ICP-AES be reported in activity (pCi/g), rather than directly in the measured quantities in ppm, to facilitate comparison with other measurement techniques which rely on measuring activity. The stated threshold for remediation for the purposes of this demonstration has been set as 35 pCi/g for uranium. If the uranium present in the soil at the site contains a natural abundance of U^{238} , U^{235} and U^{234} isotopes (99.275, 0.72%, and 0.0054% respectively), this threshold level of activity corresponds to 51.6 ppm of uranium in the soil. The presence of uranium enriched in the U^{235} or U^{234} isotopes, which have a higher activity than U^{238} , would lead to a lower concentration threshold. Analysis of solutions resulting from microwave digestion of site samples M2 and W5 and the prepared calibration standards (the 100 and 1000 ppm U and Th in Fernald soil samples) by ICP-mass spectrometry at Ames Laboratory determined that these samples contained uranium having natural abundance uranium. Therefore, the activities reported in Table III have been calculated for the measured values of the uranium concentration on the basis of natural isotope abundance uranium.

Bore hole samples from three sites (designated x, y, and z) in the Sewage Treatment Plant area were taken for analysis by LA-ICP-AES. These samples were analyzed on the standards sampling station of the RSA. Due to some uncertainty as to the depth of the different segments of the y bore hole samples, these samples were not analyzed. The state coordinates for the x and z bore holes are reported in Table II. The uranium concentrations determined for these samples as a function of depth in feet are reported in Table III. No measurable uranium concentrations were detected below the 1 foot depth in bore hole x; no detectable levels of uranium were measured for the bore hole z samples.

The uranium concentrations determined are also presented graphically in Figures 3 and 4. In Figure 3, the sample site was divided into three areas. Each area was fit using the program SURFER (Golden Software Co.) to produce a surface from the grid. A topographical map of uranium concentration was then produced for each region. These regions were combined on this map. Also superimposed is a scale map of the Sewage Treatment Plant area obtained from the USID Demonstration Plan. Areas not included on the grid were not accessible for sampling. A number of samples taken by Fernald personnel from sites within the shaded area of the map were provided for analysis by LA-ICP-AES. These samples (asi 1-7) were analyzed on the standards sampling station of the RSA. The uranium concentrations for these sites are reported in Table III. However, no coordinates for these sites were provided, so these sites were not included in producing Figure 3. Figure 4 was produced using the uranium concentrations determined by LA-ICP-AES for the sites that were analyzed. All the inaccessible sites (those not analyzed) are arbitrarily set to the level of 100 ppm. A surface was fit to this grid using SURFER. Four different-angle views of the resulting surface are displayed in Figure 4.

These figures show that two major plumes of uranium concentrations exceeding the remediation level are present to the north and to the west of the Sewage Treatment Plant. Relatively high uranium concentrations (six to nine times greater than the remediation threshold) were measured at the edge of the sampling grid, indicating possible uranium contamination beyond the boundaries of the demonstration. The surfaces shown in Figures 3 and 4 are presented only as a graphical tool and should not be used to infer uranium concentrations at locations that were not sampled using the

Figure 3. A topographical map showing the uranium concentrations (ppm) determined using LA-ICP-AES for the Fernald site Sewage Treatment Plant area. Areas not included on the grid were not accessible for sampling.

NE to SW view

NW to SE view

SW to NE view

SE to NW view

Figure 4. A three-dimensional surface representation of the uranium concentrations (ppm) at the Fernald site Sewage Treatment Plant area shown from four different directional views. Inaccessible sites, those not sampled, are set to the level of 100 ppm.

LA-ICP-AES technique.

An additional source of error has been identified which undoubtedly affected the accuracy and reproducibility of the uranium concentration measurements made during this demonstration. The TJA ICAP 61E spectrometer is dependent upon strict control of the environmental conditions in which it is operated. Specifications for installation of this system require temperature control of the laboratory environment to ± 2 degrees Fahrenheit per hour. The humidity in the lab environment should not exceed 60% relative humidity. Conditions outside these ranges cause misalignment of the spectrometer and lead to analytical errors outside the specifications for the instrument. Conditions exceeding these limits were commonplace during this first site test of the MDLEST and need to be corrected to improve the analytical precision of the LA-ICP-AES analyses. The effects of these conditions were observed through experimental drift, causing the quality assurance determination of the 100 ppm U and Th in Fernald soil standard sample (performed at least after every fourth site sample analyzed) to drift significantly outside the range of acceptable error. The drift was typically less than 30 to 40% over an hour, but at one exceptional time was as high as 196%. The nature of this drift does not allow us to apply error analysis to determine the effects on any individual determination. As such, the problem must be dealt with in order to provide more reliable measurements in the future.

Discussions with the spectrometer vendor have revealed that errors of this sort are typical for mobile-laboratory environments. However, with careful attention to detail, several of these systems have been used in mobile-laboratory environments with fixed-laboratory quality results.

Microwave Digestion and Solution Nebulization ICP-AES in the Field

Soil samples for twelve of the sites analyzed by the LA-ICP-AES technique (sites A1, A2, A3, C1, C2, E9, K3, M2, U1, U7, W5, and W11) were subjected to microwave digestion and subsequent analysis by conventional solution nebulization ICP-AES analysis at Fernald. The ICP-AES detection limit for uranium in pure solution (an aqueous solution containing uranium only) is ~ 0.5 ppm; detection limits are typically higher for solutions having complex matrices (such as a dissolved soil sample). For the microwave digestion procedure used, one gram of soil was digested, and the solution was brought to a final volume of 100 mL. This results in an effective dilution factor of 100. Therefore only those sites that have uranium concentrations greater than ~ 100 ppm will be detectable using this method of analysis. Wavelength scans using the TJA ICAP 61E spectrometer indicated uranium emission for the samples analyzed; however, the intensities measured were too low to be accurately quantitated in the field using the present instrumentation. Methods of improving the detection limit (such as utilizing an ultrasonic nebulizer or improved sample preparation procedures) are required in order to accurately quantify uranium concentrations for samples that are at or below the remediation level at Fernald.

Comparison of Field LA-ICP-AES and Laboratory Determinations

Soil samples from fifteen of the 80 Sewage Treatment Plant area sites analyzed by LA-ICP-AES at Fernald were collected and prepared (removal of organic debris, overnight drying and subsequent pulverization using a ball mill) by Fernald personnel. These fifteen site samples were

analyzed at Ames Laboratory so that the uranium concentrations determined by LA-ICP-AES at Fernald (in the field) could be compared to values obtained in the lab.

Microwave digestion and subsequent solution nebulization ICP-AES analysis of the fifteen site samples were performed at Ames Laboratory. The instrumentation in the MDLEST was not used for these analyses. An ultrasonic nebulizer (CETAC Technologies model U-5000) was used for sample introduction into the ICP. The ICP was an RF Plasma Products ICP-16L system, similar to that used in the mobile laboratory for LA-ICP-AES. The ICP emitted light was focussed onto the entrance slit of a 0.5 meter spectrometer (Acton Research model VM-505) equipped with an intensified photodiode array (EG&G Princeton Applied Research model 1420) using a lens, rather than using a fiber optic bundle to couple the ICP emission to the spectrometer. The microwave digestion solution and standard additions of 1, 4, and 10 ppm uranium were analyzed for each of the fifteen site samples. The net intensity of the uranium 409.0 nm emission line was determined for the solution and its standard addition solutions. The uranium concentration in the solution was determined by linear regression using the standard addition curve. The results for the fifteen samples analyzed are presented in Table IV. The values are the averages of duplicate or triplicate microwave digestions and subsequent standard addition determinations of uranium by solution nebulization ICP-AES. The values listed have been converted from uranium concentration in solution to concentration (in ppm) for the solid soil samples. The 95% confidence interval (95% c.i.) for the uranium concentrations determined are also listed in Table IV. An example standard addition curve with its 95% c.i. limits is shown in Figure 5, for Fernald site sample M2. The intensity of the uranium 409.0 nm emission line for the microwave digestion solution for M2 (0 ppm U added) and for the 1, 4, and 10 ppm U standard additions to the microwave digestion solution for M2 are plotted. The uranium concentration for M2 is calculated from the intercept and slope of the standard addition curve.

The fifteen site samples were also analyzed by LA-ICP-AES at Ames Laboratory. Laser radiation from a frequency doubled, 10 Hertz repetition rate, Quantel International YG481 Nd:YAG laser was coupled into a ~20 meter long, 600 μ m core diameter fiber optic cable. The output from the fiber optic was focussed onto the surface of the soil sample, which was contained in an aluminum sample cup in the bottom of a glass ablation cell. A short length (~3 meters) of tygon tubing was used to connect the ablation cell to the ICP torch; argon gas flowing through the ablation cell transported the laser ablated soil material from the ablation cell to the ICP torch. The ICP and spectrometric system used are described in the previous paragraph. The response of the LA-ICP-AES system was calibrated for uranium by analyzing four soil standard samples - the Fernald soil blank and prepared 50, 100, and 500 ppm uranium in Fernald soil samples. A calibration curve of the net intensity of the uranium 409.0 nm emission line ratioed to the silicon 410.3 nm emission line versus uranium concentration was obtained for the four standard samples analyzed. Uranium concentrations for the fifteen Fernald site samples were determined from the measured LA-ICP-AES uranium to silicon ratios using the calibration curve obtained. The results are presented in Table IV. The values are the averages of triplicate LA-ICP-AES determinations. The 95% c.i. for the uranium concentrations determined are also given in Table IV. The calibration curve for the four standard samples analyzed, with its 95% c.i. limits, is shown in Figure 6.

The uranium concentrations determined for the fifteen site samples by LA-ICP-AES at Fernald (in the field) and the 95% c.i. values are also tabulated in Table IV, for comparison with the values determined at Ames Laboratory. Figure 7 is the uranium to silicon calibration curve obtained in the

Table IV. Technique Comparison for Uranium Concentrations in Fernald Soil Samples

Name	Microwave Dissolution, Determination by Std. Additions 0.5 m Acton		TJA LA-ICPAES (in situ)		0.5 m Acton LA-ICPAES at Ames Lab	
	Value (ppm)	95% c.i.†	Value (ppm)	95% c.i.‡	Value (ppm)	95% c.i.‡
a1	19.0	8.9	<20	---	45.7	48.7
a2	21.1	9.8	<20	---	<20	---
a3	43.7	10.0	48	85	37.1	48.9
c1	23.1	6.7	<20	---	24.2	49.3
c2	28.2	5.4	21	92	22.9	49.4
e9	37.4	8.3	54	83	<20	---
k2	72.9	6.5	156	80	34.9	49.0
k3	124.1	10.3	112	85	101.4	47.5
m2	105.8	11.6	285	88	70.6	48.1
q3	102.5	6.1	58	84	74.3	48.0
u1	63.2	13.4	84	93	36.5	48.9
u7	113.1	3.6	224	79	80.7	47.9
w5	62.2	6.0	217	91	29.1	49.2
w11	53.3	7.7	180	83	36.0	49.0
y1	54.6	10.8	112	83	20.9	49.4

† Confidence interval for predicted x from a known value of $y = 0$.

‡ Confidence interval for predicted x from a measured value of $y = \text{measured intensity}$.

Figure 5. Standard Addition Regression: Sample M2.

Figure 6. LA-ICP-AES Calibration at the Ames Laboratory.

Figure 7. LA-ICP-AES Calibration at Fernald.

field using the TJA ICAP 61E for the three standard samples (Fernald soil blank and the 100 and 1000 ppm U and Th in Fernald soil samples) analyzed. The 95% c.i. limits for the calibration curve are also plotted.

A number of facts are evident from the data presented in Table IV and Figures 5-7. The uncertainty in the field-determined LA-ICP-AES uranium concentrations is larger than the uncertainty in the laboratory-determined LA-ICP-AES values, which, in turn, is larger than the uncertainty in the microwave digestion and standard addition determination for uranium. This is not unexpected; field analytical determinations are generally less reliable than laboratory-determined values. For eleven of the fifteen site samples analyzed, the uranium concentrations determined in the field by LA-ICP-AES agree with the values determined in the lab (within the uncertainty of the determined concentrations). The four that do not agree are sites M2, U7, W5, and W11. The uranium concentrations determined by LA-ICP-AES in the field are typically higher than the values determined in the lab, overstated by a factor of two to three for some of the sites. For the two in-lab methods of analysis used, the uranium concentrations determined for the fifteen site samples agree (within the uncertainty of the determined values). The results for the microwave digestion and standard addition determination for uranium have less uncertainty than the laboratory LA-ICP-AES values and are therefore more reliable.

Conclusions

The mobile laboratory is a very important and useful tool that is needed if the DOE goals of *faster, better, cheaper, and safer* are to be met in cleaning up the environment. The MDLEST/RSA, incorporating the LA-ICP-AES technology for in situ field sampling and analysis, is a specialized tool for soil screening and characterization. We know of no other field deployable laboratory for in situ, near real-time, precise, quantitative and qualitative, elemental analysis capable of these low limits of detection. Although this laboratory was developed and constructed over a very short period of time, approximately fifteen months, it performed extremely well during field demonstration at the Fernald, Ohio site. A number of improvements are planned that will: increase the precision of the analytical determinations, enhance the field mobility, optimize the field operation, and reduce the size of the operating team.

Appendices

The following two papers were presented at Spectrum '92, Boise, ID
August 23-27, 1992

- I Remote, Real-Time Analysis of Hazardous Wastes Through Laser Ablation-Inductively Coupled Plasma-Atomic Emission Spectrometry
- II Mobile Instrumentation Platform and Robotic Accessory for Real-Time Screening of Hazardous Waste

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