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Airborne Heavy Metal Monitor

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by

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EXECUTIVE SUMMARY

For the Phase I program, Physical Sciences Inc. (PSI) proposed to develop a simple, inexpensive, compact, real-time optical diagnostic for the measurement of metals in airborne aerosols and particulates. During the Phase I effort, key issues of sensitivity and calibration were to be explored and a prototype monitor for lead constructed and tested. The objectives of the proposed Phase I program were outlined in the proposal as follows:

- Build a simple, fieldable monitor for lead-laden aerosols based on PSI's experience with spark-induced breakdown spectroscopy
- Calibrate the monitor with lead-containing aerosols generated by PSI's in-house dry aerosol generator
- Test the unit offsite at a firing range.

PSI has successfully accomplished this statement of work. Highlights of our achievements are:

- Assembly of a fully functional lead monitor. The unit is composed of a spark power supply, spark and sample chamber, radiometer detection package, and computer for data acquisition and display.
- The system has been calibrated over the range 10 ppbw to 10 ppmw using a gravimetrically-determined dry aerosol generator (1 ppbw ~ 1.2 $\mu g/m^3$). The 3σ detection limit is 8 ppbw. Further optimization of the detection system could yield another order of magnitude in detection limit.
- The calibration has been shown to be independent of the chemical forms of lead tested. Tested compounds include lead nitrate, lead chloride and lead acetate.
- Our detection scheme has been proven to be free of potential spectral interferences from iron, silicon, aluminum, magnesium and potassium.
- The functionality of the lead monitor has been tested in-house by detecting the lead from handgun blanks containing lead styphnate primer.
- Simultaneous data from the monitor and lead collected on an internal filter (analyzed by an environmental laboratory) compare well.
- The lead monitor has been tested twice at a local, modern firing range. These tests have included measurements in the immediate vicinity of the shooting booths and downrange in the ventilation crawlspace before the lead-laden air enters the scrubbers. The data correlate well with shooting events and the observed concentrations are consistent with typical levels reported in the literature.

The program is well positioned for completion of technology development under Phase II. The Phase II program will take the current brassboard prototype and will develop a fully functional alpha prototype suitable for extensive in-house and field testing. Issues to be addressed under Phase II are: sensitivity improvement to achieve a 2 to 3 ppbw detection limit, simplified and unified architecture to reduce instrument size and number of components, and the acquisition and publication of data enabling NIOSH approval of the technology.

1. INTRODUCTION

Airborne heavy metals are known health hazards as typified by the following: Dr. Robert Kehoe is quoted in *Health Effects of Occupational Lead and Arsenic Exposure*, "the hazard of lead poisoning is not controlled by the physician, nor by prophylactic medical therapy, but substantially by controlling the environment to known safe levels."[1] This statement clearly indicates that the key to lead, and other metal exposure, is to control the ambient atmospheric concentration to which the worker is exposed. Proper environmental control, however, requires accurate, real time monitoring to establish that the entire workspace is at or below accepted lead standards. Unfortunately, the affected industries typically rely upon periodic extractive techniques in combination with personal dosimeters. Although these methods can provide average time histories or occasional time points, these are insufficient for real-time corrective action to be taken, particularly if the environment is complex with multiple sources. A real time monitor would enable work areas to be three dimensionally mapped, ventilation optimized, suspect areas investigated, and immediate corrective action to be taken in the event of a transient. The result will be an improved and safer environment for tens of thousands of workers. Indeed, a real-time sensor could be incorporated as part of a control system further enhancing the value of the sensor.

The health effects from lead exposure to children are well documented. They include weight loss, weakness and anemia for chronic exposure. Acute poisoning results in anorexia, vomiting, malaise, and possible brain damage. Exposure in children derives principally from ingestion of lead-based paint. Lead poisoning in adults is usually occupational due to inhalation of lead dust or fumes. The adult symptoms include renal failure, central nervous system disorders, infertility, and birth defects. The number of industries or activities in which lead exposure can occur in numerous industries and activities. In lead-acid battery manufacturing, lead dust is a known hazard; in building remediation and the construction industry, the removal of lead paint from structures such as buildings and bridges can generate high airborne lead concentrations; in both indoor and outdoor firing ranges lead is generated from bullet ablation and the lead contained in the primer and propellant; and in metallurgy (smelting and refining) where lead fumes may be encountered.

1.1 Occupational Exposure to Lead

Occupational lead poisoning is a long-standing and extensive health problem in industry. Lead has widespread industrial uses, and is a severe toxicant that affects the nervous system and many organs. In 1978 OSHA established the general industrial standard that sets the permissible airborne lead exposure level and includes biologic monitoring of lead-exposed workers.[2] A similar standard was extended to the construction industry in 1993.[3]

The OSHA health standards establish a permissible exposure level for airborne lead of 50 $\mu g/m^3$ and an action level of 30 $\mu g/m^3$. A variety of measures are required unless air monitoring ensures that lead exposures are below the action level. These measures include blood lead testing, use of respirators and protective clothing, hygiene facilities and employee training.

OSHA has estimated that nearly 1 million U.S. construction workers are exposed to lead in the workplace.[3] Roughly three-quarters of these workers are involved in commercial, institutional and residential remodeling projects, for which exposure levels are low-moderate. The most severe lead exposures occur to the 50,000 to 60,000 workers involved in the rehabilitation of highway and railroad bridges and large tanks.[3,4] Lead-based paints are extensively used on such structures due to their favorable rust and corrosion protection properties, and mechanical removal of previously applied paint can produce exposure levels of several thousands of $\mu g/m^3$. The total lead paint abatement market is reportedly \$400 billion.[5] Market research studies,[6] based on OSHA's preliminary estimates, report that the annual costs of compliance with the Lead in Construction Interim Final Standard are approximately \$400 million. Half of these costs, \$200 million annually, are associated with repainting and rehabilitation of bridges and large tanks. Exposure monitoring and related record keeping amount to \$120 million annually. Plumbing, welding, roofing and electrical conduits are other sources of lead exposure in building construction.

In addition to the construction industry,[7] significant occupational exposures to lead occur in a large number of commercial and industrial settings, reflecting the widespread industrial use of lead. These include primary and secondary lead production,[8] the manufacture of brass, bronze, copper and other non-ferrous metals, the electronics industry, lead acid battery manufacture, automobile radiator repair shops, and the manufacture of ink, paints, wall paper and cans.[9,10] Indoor gun firing ranges,[11-13] of which there are several thousand in the U.S., are another source of worker exposures to high concentrations of lead.

1.2 Current Lead Monitoring Techniques

Real-time measurement of airborne lead concentrations would enable immediate worker protection, reducing lead exposure to approximately one million U.S. workers. Current measurement techniques are based on filter collection followed by atomic absorption (AA) or inductively coupled plasma (ICP) analysis.[14-16] Generally the filters are sent to an independent laboratory and the results are returned within several days to a week or more.

The need for more immediate results are beginning to be addressed through the development of field portable instruments.[17] Our technology, based on spark-induced breakdown spectroscopy or SIBS, offers high sensitivity, field-portability, and fast response time (less than 1 minute) with no need for sample collection or workup.

1.3 Need for Real Time, Field Monitors

OSHA regulates lead exposure for private sector employees. The OSHA lead regulations 29 CFR 1910.1025 and 29 CFR 1926.62 require:

- If a worker works with lead, the employer must test the air for lead levels.
- If the lead level in the air is 30 $\mu g/m^3$ of air or greater, the employer must offer routine blood testing.

- An employee must be removed from all exposure to lead if the average blood level is 50 µg/dl or more on three tests. He or she cannot return to an environment where lead is present until the blood level falls to at least 40 µg/dl.

- The standard also establishes requirements for medical monitoring, respiratory protection, protective clothing, engineering controls and ventilation, work practice controls, hygiene facilities, and employee education.

Adherence to these regulations regarding the immediate work environment can be substantially strengthened with portable real-time monitoring. Additionally, secondary or adjacent areas can also be tested to determine the extent of fine particle and aerosol migration. The breadth of employer liability is increasing and could be minimized with more aggressive monitoring. Recently, two Massachusetts companies were indicted on assault and battery charges on accusations they exposed employees to high levels of lead, cadmium and other dangerous substances (Boston Globe, Nov 15 1996).[18] One of the workers complained to management about higher than normal blood-lead levels but was assured that since she worked in the office she had nothing to fear. Another worker complained of stomach pains, sore teeth, nervousness and occasional headaches. Despite decades of education, cases of acute and chronic lead poisoning still occur, as illustrated in this recent news article. Real time monitoring can help to eliminate such cases by reducing worker exposure, ensuring regulatory compliance, and minimizing employer liability.

SIBS instruments will offer substantial benefits to operators of plants and other facilities by providing an accurate real-time measurement of airborne concentrations of lead and other toxic metals. Real-time monitoring should reduce potential lead exposure to workers by quickly identifying situations where significant lead exposures pertain, thus enabling a rapid response to reduce airborne lead levels or utilize personal protective equipment (PPE) for worker protection. Plant and facility operators will receive substantial financial benefits as well, by avoiding analytical lab expenses and reducing biological monitoring requirements. With a real-time monitor to ensure that exposures are safely below the action level, the use of PPE and other worker hygiene practices can be reduced, leading to major gains in productivity. These benefits should be particularly attractive to the construction industry where lead exposures fluctuate widely from task to task and day to day. Indeed, PSI has obtained very enthusiastic responses from the lead abatement contractors and firing range operators we have contacted. Major benefits of the SIBS monitor will be provided to other industrial operators with lead exposure problems by quickly identifying occurrences of high exposure levels and reducing the use of protective equipment and excess air circulation when not needed.

2. EXPERIMENTAL

2.1 Technology Basis

The SIBS diagnostic is based upon well founded principles of atomic emission spectroscopy. The spark discharge creates a hot plasma that initially emits broadband from the UV to the IR regions of the spectrum. As the plasma cools, the broadband emission decays and the atomic emission lines from the lead can be easily observed. This process is summarized in Figure 1. Although several lead lines are available for analysis, we have found that the strong 405.7 nm line is optimum for our detector. The region around 405.7 nm is free from spectral interferences that arise from species generated in a firing range. We have verified this in extensive tests using species known to be present in the effluents of primer and bullets, e.g., Fe and K.


High Voltage Spark Initiates Discharge

Discharge Vaporizes, Ionizes Air and Aerosol Particles

As Plasma Cools Atomic Emission Occurs

Figure 1. Principles of the SIBS detection strategy.

We use a dual color radiometer to obtain high sensitivity for our lead monitor. One filter passes the lead line (405.7 nm) and a second filter passes a narrow band near 400 nm. The ratio of the emission intensities at these two wavelengths is used in the automated data analysis routines described below. In Figure 2 we show the lead emission spectrum obtained with an optical multichannel analyzer and have superimposed the two filter transmission curves.

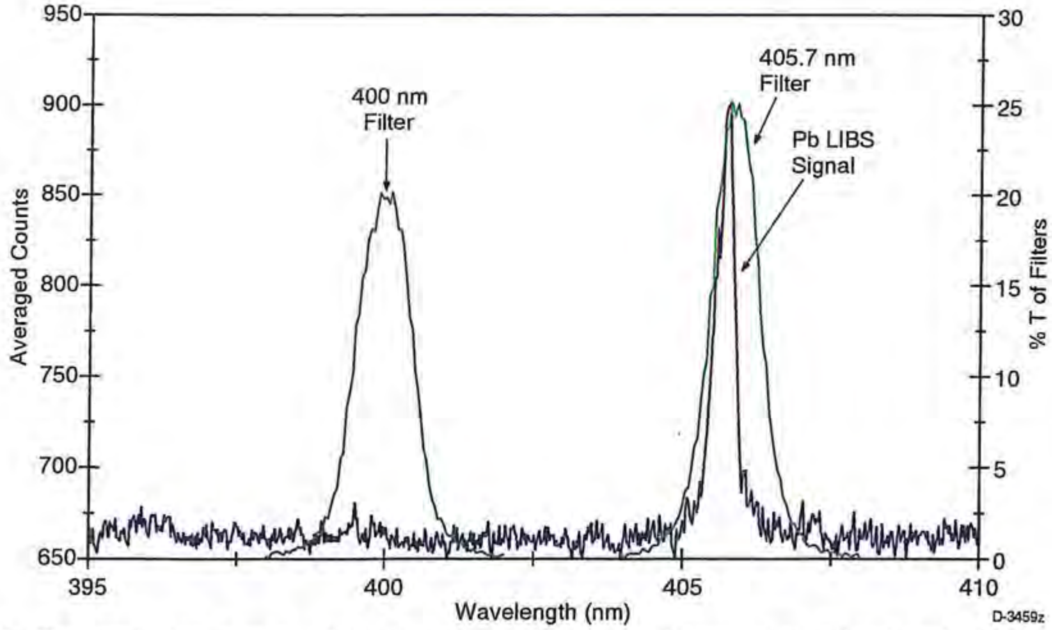


Figure 2. Transmission curves of the detection filters superimposed on a lead emission spectrum.

2.2 Calibration

Calibrations were conducted using an aerosol generator shown in Figure 3. The source relies on a modified Berglund-Liu aerosol generator to first produce a monodisperse liquid aerosol of water with suitable quantities of water soluble salts. The aerosol is made monodisperse by the imposition of a periodic instability on the thin stream of liquid (20 µm diam, which produces 2 to 10 µm site particles after evaporation) ejected from the generator. This instability, for certain combinations of liquid flow rate and instability frequency, forces the stream to break up into single-sized droplets. The instability is imposed on the stream using a piezoelectric crystal driven by a standard electronic function generator. The aqueous particles are then passed through a drying column equipped with a stainless steel porous tube wrapped in heating tape. Air drawn through the tube provides the drying heat and prevents significant particle loss on the interior walls of the device. Final particle concentration (number density) is determined by the passage efficiency through the generator (typically about 90% as measured at the porous tube exit), the air flow rate through the device, and the particle generation frequency (typically 100 kHz). Net concentration of the metal in air is varied by changing the metal salt concentration in the solution passed into the liquid aerosol generator.

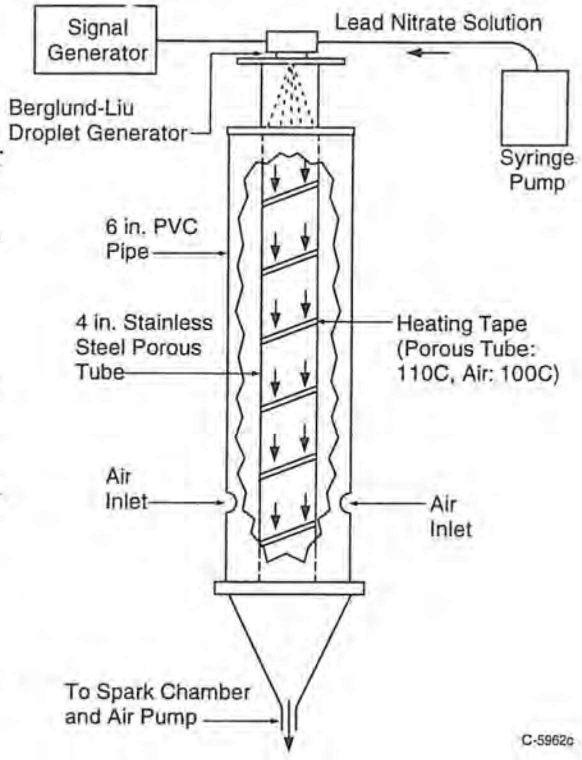


Figure 3. Apparatus for calibration of SIBS.

In addition to the Berglund-Liu droplet generator, we have also employed a commercial package from Sonotek. This device was chosen as it promised greater reliability then the homemade Berglund-Liu system which had been purchased from a local University. This latter system was accurate but was prone to clogging and we could no longer obtain spare parts. The Sonotek nebulizer produces a particle stream having a narrow size distribution peaking at 18 µm with little kinetic energy. The Sonotek system was gravimetrically verified to deliver 80 to 95% of the particles to the filter.

As is called out in the statement of work, we calibrated the lead monitor at ten points over the range of background to nearly 8 ppmw (parts per million by weight), equivalent to ~9.6 mg/m^3. This calibration was done by feeding diluted ICP $Pb(NO_3)_2$ solutions through a Sonotek ultrasonic atomizer with a median droplet diameter of 18 µm. These particles were allowed to fall through a heated column to dry off the solvent (water), leaving dry particles. The concentration of analyte in the dry aerosol is a function of the frequency at which the atomizer is driven, solution feed rate, rate of air flow through the column and solution lead concentration. For the purposes of this calibration, aerosol concentration was varied by changing the lead concentration in the feed solution.

2.3 Phase I Prototype System

We assembled a fully functional lead monitor for the Phase I program. A schematic of the monitor is shown in Figure 4 and a photograph in Figure 5. The sampling system is shown on an expanded scale to highlight some of the critical components. Details of each of the important subsystems are presented in the following sections.

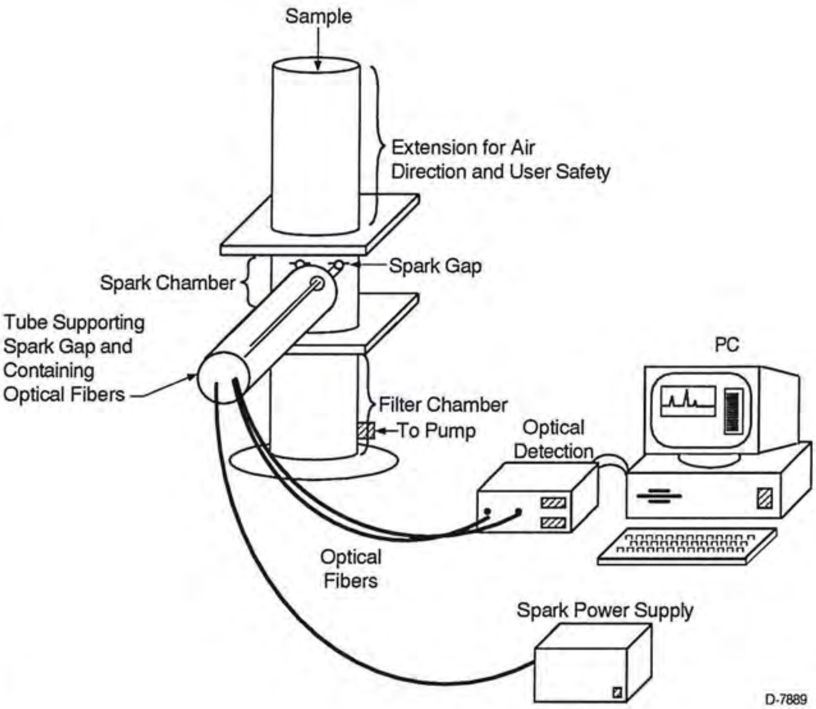


Figure 4. Schematic of PSI lead monitor (not to scale).

2.3.1 Spark Chamber and Sampling System

The probe is a tube that houses optical fibers and high voltage leads and supports the electrodes at the centerline. A photo of the unit is shown in Figure 6. The probe is entirely enclosed, with a quartz window on the front plate to collect the emission from the discharge. The probe is 2 ft long, 3 in. in diameter and weighs less than 5 lb.

The electrodes are two rods at a fixed gap. The electrodes are set perpendicular to the flow of gas though the gap such that the flow through the gap is unrestricted. The pulsed discharge at the gap results in a large volume filling spark (several cubic millimeters) of nearly spherical shape.

Figure 5. PSI metal monitor. Plexiglas sampling chamber is on right side of table.

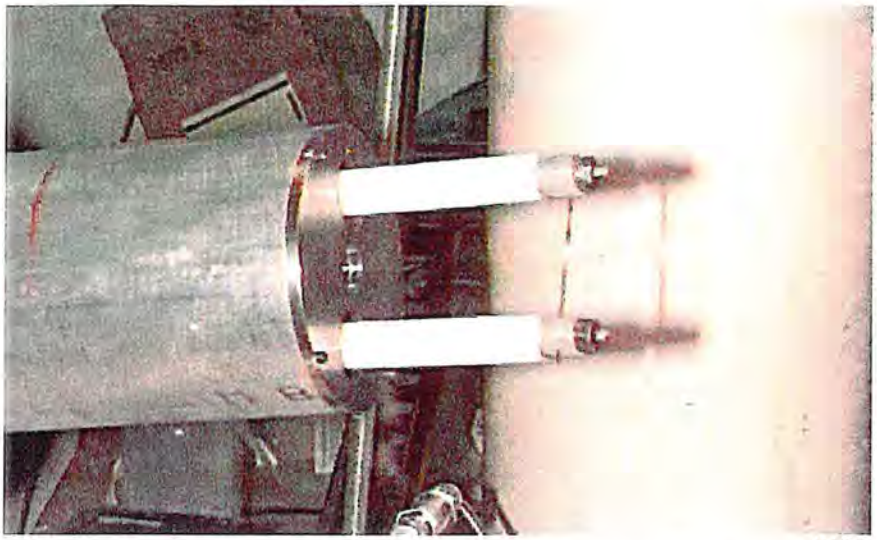

V-1451

Figure 6. Photograph of spark probe showing front face.

The SIBS probe in its current configuration is shown in Figure 7. The design was principally derived for the unit to interface to an elevated stack gas environment. This drove the choice of metallic, dimensions (length) and other temperature compatible components.

The schematic shows the electrical cables, the bundle of six fiber optics, the window, and the rear connectors. Although specifically designed for a CEM environment under PSI IRAD funding, this probe is also compatible with an industrial hygiene environment. The only adaptation necessary was to interface it to the plexiglass sampling chamber and properly support the weight at the rear end. The latter was accomplished with a plexiglass stand.

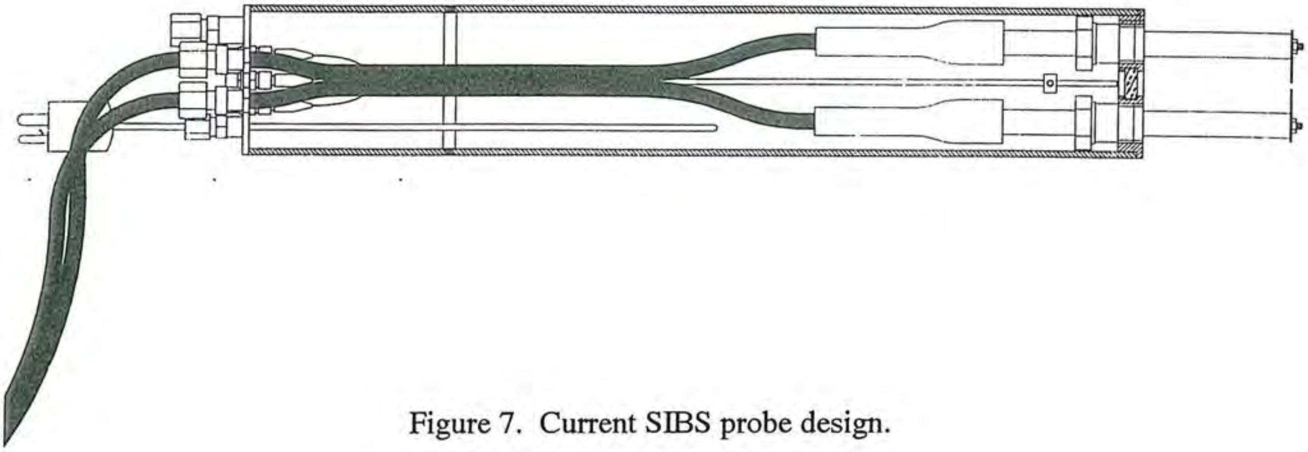


Figure 7. Current SIBS probe design.

2.3.2 Spark Power Supply

The power supply is housed in a box with dimensions of 13 x 15 x 16 in., and weighs approximately 25 lb. This unit requires 120 V/60 Hz line voltage. The timing of the power supply is controlled by a function generator, which can drive the power supply from about 0.1 Hz to over 10 Hz. We typically operate at 0.5 to 1 Hz. An electrical schematic is shown in Figure 8.

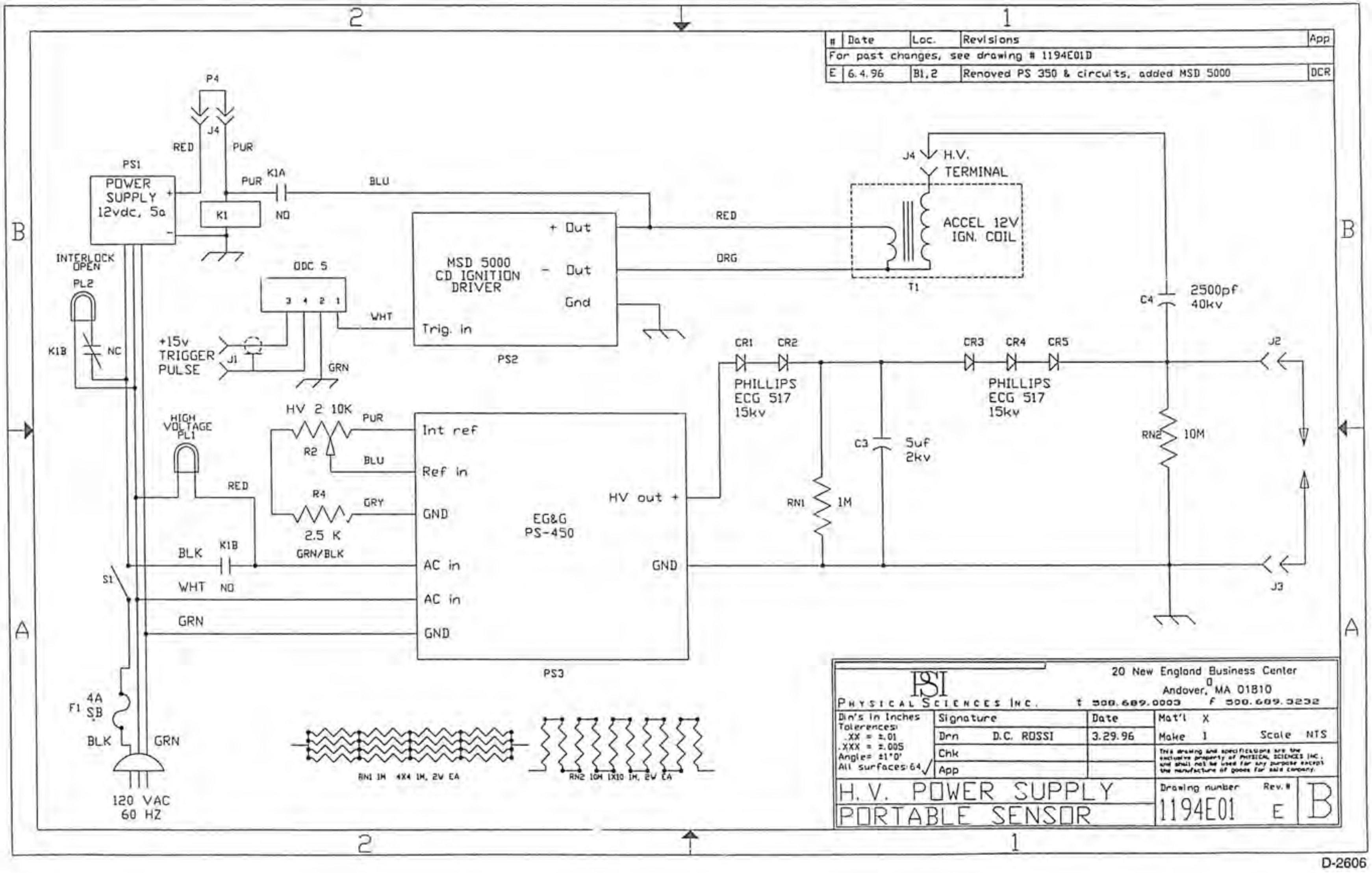


Figure 8. Schematic of Spark Power Supply

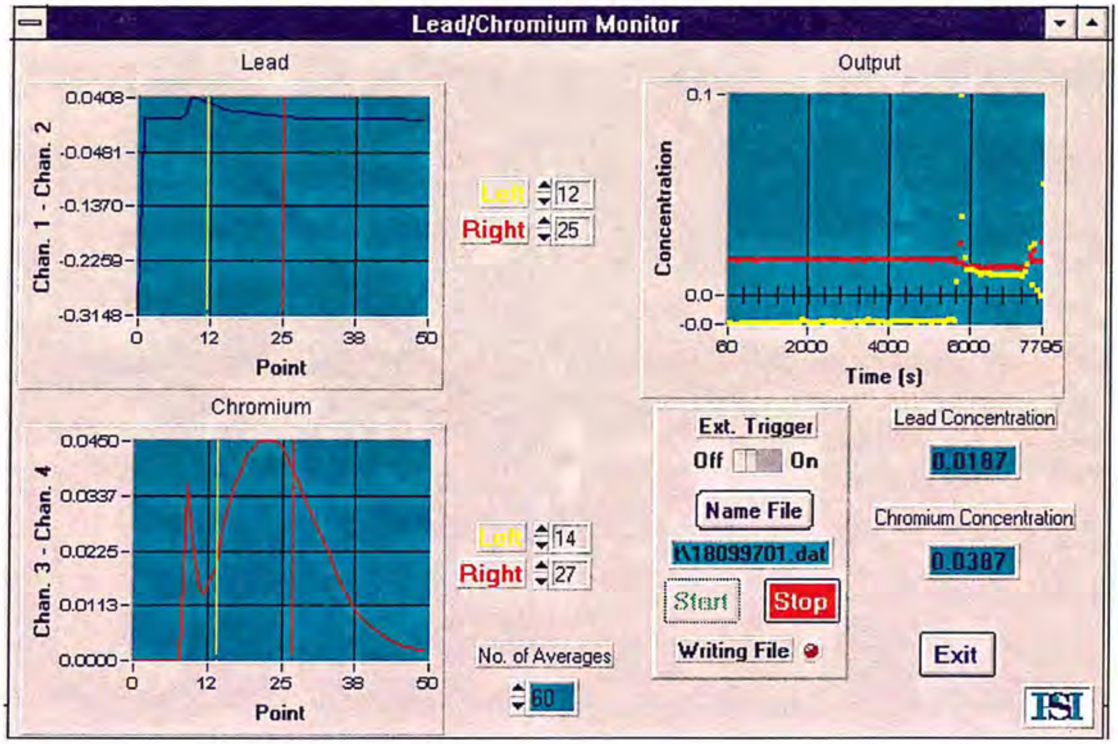
The unit operates by discharging the capacitor bank across the electrode gap. The capacitors are charged by the EG&G power supply which is capable of performing at 100 J/s. An initiator spark is provided from an automotive coil (40kV) which creates a low resistance path for the capacitors. A turnpot on the face of the power supply adjusts the power level provided by the capacitor system. We typically operate in the 1-5 J range, with 5 J the most common setting.

2.3.3 Radiometer Detection

The emission from the metals in the spark is passed to the radiometer with optical fibers, and detection of the persistent atomic features is accomplished by narrow-bandwidth interference filters and miniature photomultiplier tubes. The strategy behind the radiometer is to accumulate the signal associated with the atomic line with one filter and to subtract any background signal with a nearby filter that has no atomic feature inside its bandwidth. This unit requires only 120 V/60 Hz line current.

2.3.4 Data Acquisition and Display

The analog signals output by the radiometer are input into a computer with a 1.2 MHz A/D data acquisition board. The temporal traces are evaluated by PSI-developed routines employing LabWindows CVI® software. The on-line and off-line temporal traces are subtracted and integrated. An example of the screen performing this function for lead and chromium is shown in Figure 9.



Lead: Left 12, Right 25

Chromium: Left 14, Right 27

No. of Averages 60

Ext. Trigger Off On

I:\18099701.dat

Writing File

Lead Concentration 0.0187

Chromium Concentration 0.0387

Figure 9. Data temporal control and acquisition screen.

The left-hand window shows the temporal response of the radiometer. The difference between the 405.7 and 400 nm channels is shown. The initial high intensity spike is due to the intense, early time, broad band emission that is generated during the SIBS spark. The cursors are used to perform the temporal integration used to calculate the lead concentration shown in the right-hand screen. The bottom left hand screen is for chromium. The right-hand screen shows a "strip chart recording" of the lead concentration as a function of time.

2.4 Parts List

Table 1 summarizes the complete parts list for the lead monitor used for this program, excluding assembly costs.

2.5 Routine Operation and Maintenance

When the instrument is being calibrated the sampling system and probe are directly attached to the calibration manifold. When sampling air in the laboratory or in the field the sampling system is detached and allowed to sample ambient air. The sampling pump draws 1.3 cfm and the sampling system is equipped with a filter to trap particulate for later laboratory analysis and comparison to the monitor data.

We do not yet have a field calibration system. When the unit is sent into the field, we rely upon laboratory calibrations which are performed immediately before shipment. Through experience we have determined that the calibrations will hold to ±5% so long as the electrode alignment is maintained (both electrode gap of 6 mm plus alignment with respect to the optical fibers). To ensure this to be the case, we have developed a field electrode alignment technique. A low power red diode laser is connected via optical fiber to one of the six positions on the rear of the probe. The light passing though the optical fiber is then projected onto a pre-drawn template showing the optimum positions of the electrodes. If the electrodes deviate from this alignment, they are adjusted accordingly.

Re-adjustment of the electrodes is not common and is only needed after transportation or dis-assembly causes their physical mis-alignment. We have operated the system for periods exceeding 100 hours without having to re-align the electrodes.

The eventual commercialization of our technology will require the development of a field portable calibration system. We are in the process of considering the nature and extent of such a system. This need is one of the reasons we have adopted a commercially-available nebulizer for our own calibration system.

We do have a simple method we use in the field to verify functionality of the instrument. Using dilute ICP standards we have prepared a small aerosol spray bottle containing Cr^{3+}. We chose chromium because our system is inherently more sensitive to this element and we chose this valence because of its substantially reduced toxicity compared to Cr^{6+}. To test the system we simply spray the solution near the sample inlet creating a cloud of 100-200 ug/m^3 Cr^{3+}. This is not a field calibration but it is a useful check of instrument functionality.

Table 1. Parts List for Prototype Lead Monitor

Subsystem	Part Identification	Manuf./Supplier	Part #	Number	Item Cost	Total Cost
Radiometer/Data Acquisition						
Radiometer	HC120-06 PMT, 40 kohm, 500 kHz	Hamamatsu	HC120-06	2	$517.82	$1,035.64
	Instrument Enclosure (Version IP 40)	Rose Enclosures	07.40.0530-00	1	$125.74	$125.74
	Interference Filters (1 nm FWHM 400, 405 nm)	Andover Corp.	N/A	2	$598.00	$1,196.00
	73JA103-ND 2W, 10 Turn Pot, 10 kohm	Digikey Corp.	73JA103-ND	2	$14.64	$29.28
	3412KL-ND 20 Turn Dial w/ Lock	Digikey Corp.	3412KL-ND	2	$11.55	$23.10
	Panel DVM	Digikey Corp.	CDPM302-ND	2	$44.52	$89.04
	PW9946-ND +/- 15V/0.4 A Power Supply	Digikey Corp.	PW9946-ND	2	$38.61	$77.22
	T309-ND Wall Transformers	Digikey Corp.	T309-ND	2	$10.00	$20.00
	CP-6-ND Panel Mount Jacks	Digikey Corp.	CP-6-ND	3	$1.83	$5.49
	8729 Combination Fuse On/Off Switch	H/Eaton/Allied	548-1054	2	$2.38	$4.76
	Filter holders	PSI/Custom	N/A	2	$86.76	$178.52
	Wires	Local	N/A	1	$10.00	$10.00
Fiber Optics	Fiber cables, 10 meters, double ended SMA	Thorlabs		2	$150.00	$300.00
	Collimators	Thorlabs	F230SMA-A	2	$128.00	$256.00
Data Acq.	HTMIO16E-1 Data Acquisition Card	National Instruments	777142-01	1	$1,795.00	$1,795.00
	SCB-68; PN/776844-01 Breakout Box	National Instruments	776844-01	1	$295.00	$295.00
	SH6868 Cable; PN/182419-01	National Instruments	182419-01	1	$125.00	$125.00
	Pentium Computer			1	$900.00	$900.00
Cost						**$6,465.79**
Sampling/Spark Excitation System						
Sampling	HV spark plug wire & connectors	Weeden Racing	-	1	$43.00	$43.00
	Swagelok fittings	Cambridge Valve	various	5	$26.80	$134.00
	Igniter	Crown Engineering	IP-15	2	$78.40	$156.80
	SMA bulkhead fits.	Fiber Instrument Sales	861210000	2	$2.40	$4.80
	7/8" vacuum coupling	Industrial Equip. & Sales	S-87-KM	2	$45.00	$90.00
	Rhenium electrode wire	Aldrich	35,713-8	1	$137.75	$137.75
	5/8" Windows (excitation probe)	CVI Laser Corp.	PW-0712-UV	1	$140.00	$140.00
	Pump - 1.3 cfm	Fisher		1	$355.00	$355.00
	Filters	Millipore/Lab Safety	MAWP037-AM	1	$7.50	$7.50

Table 1. Parts List for Prototype Lead Monitor (Continued)

Subsystem	Part Identification	Manuf./Supplier	Part #	Number	Item Cost	Total Cost
HV Power	Strobe Power Supply	EG&G	PS-500 AC	1	$680.00	$680.00
	12VDC Power Supply	Sola/Allied	SLS-12-051	1	$84.50	$84.50
	Blocking Capacitor	Sprague/Newark	715P	1	$3.50	$3.50
	Ignition Coil	MSD	Blaster-2	1	$50.10	$50.10
	Diode - 15kV	NTE Electronics	517	8	$5.39	$43.12
	Connectors	Superior/Newark	PP50 GR	15	$22.00	$330.00
	Connectors	Superior/Newark	RS50 GR	15	$22.00	$330.00
	High-voltage cable 18 ga., 40 KV	Rowe/Newark	R800-4018-9	0.02	$192.09	$3.84
	Interlock relay	Potter and Brumfield	KRPA-11DG-12	1	$19.71	$19.71
	Energy Storage Capacitors 0.5 uF @3kV	PC/Newark	OF30-505	10	$74.93	$749.30
	Power Switch (fused) with retainer clip	Eaton-Hein./Newark	PS1	1	$4.22	$4.22
	Switch Bezel	Eaton-Hein./Newark	PS1--BZ	1	$1.03	$1.03
	Switch lamp	Eaton-Hein./Newark	#009-18250	2	$4.10	$8.20
	Potentiometer	Bourns/Newark	3540S	1	$19.92	$19.92
	Resistors (6MOhm, 2W, 750V)	Allen-Bradley/Newark	RC 42	18	$1.22	$21.96
	Rack-mount Chassis	Bud/Newark	CH-14404	1	$76.94	$76.94
	Chassis Cover	Bud/Newark	CH-14432	1	$28.17	$28.17
	Power Input Filter	Corcom/Newark	6VM4	1	$19.16	$19.16
	Trigger Relay	Crydom/Allied	ODC-15	1	$15.17	$15.17
	Delay Generator	BKPrecision/Newark	Type 4040	1	$499.00	$499.00
Cost						**$4,056.69**
Lead Monitor Total Hardware Cost						**$10,522.48**

Reliable, reproducible sparking (no missed sparks) is generally obtained in ordinary room air and in humidified air at elevated temperature. We have observed that the unit will miss occasional sparks in dry air. The reason is the increased breakdown threshold of the gap under such conditions. We are considering simple engineering solutions for this, such as pre-humidification of the sampled air or increasing the voltage of the initiator spark to overcome the increased capacitance.

The unit can easily be operated by a single person, or when started, in unattended mode. When we send the present instrument into the field, however, we generally send two people. The principal reason is that the system is a "brass-board" with several separate components. Two people can readily unpack the instrument and get it fully functional in under an hour. The next development phase, an alpha prototype, will emphasize a unified architecture to reduce total instrument size and number of components such that a single person can take the instrument into the field.

Very little routine maintenance of the instrument is needed. When exposed to high concentrations of analyte, the plexiglass body must be removed and cleaned and the electrodes must be cleaned. The procedure for cleaning the electrodes is to swab them sequentially with dilute nitric, water, then methanol. If extremely high concentrations of analyte have been encountered, it is sometimes necessary to gently sand the electrodes with a fine grit sandpaper to remove residual analyte material. The typical time for this procedure is about 10 minutes.

2.6 Data Acquisition and Analysis

The LabWindows software has been written to allow the data to be saved in time tagged strip chart fashion. We typically operate the system at 1Hz spark rate with 5 shot averaging set in the software. The program therefore saves the integrated lead voltage on this time scale with an associated data and time.

To process the data we import the saved data file into a speadsheet and apply an empirically-determined background correction with the calibration constant to obtain data in real concentration units. We also typically average the 5 second data for 1 minute to match the calibration conditions. The spreadsheet is then used to plot the data.

We have equipped the LabWindows program with the capability to plot and save data in real concentration units (ppbw or ug/m^3) by automatic application of the background correction and calibration. For the Phase I program, we have uniformly saved and processed raw data.

3. RESULTS AND DISCUSSION

3.1 Calibration Results

As is called out in the statement of work, we calibrated the lead monitor at ten points over the range of background to nearly 8 ppmw (parts per million by weight), equivalent to ~9.6 mg/m^3. This calibration was done by feeding diluted ICP $Pb(NO_3)_2$ solutions through a Sonotek ultrasonic atomizer with a median droplet diameter of 18 μm. These particles were allowed to fall through a heated column to dry off the solvent (water), leaving dry particles. The concentration of analyte in the dry aerosol is a function of the frequency at which the atomizer is driven, solution feed rate, rate of air flow through the column and solution lead concentration. For the purposes of this calibration, aerosol concentration was varied by changing the lead concentration in the feed solution.

Raw calibration data are shown in strip-chart fashion in two magnifications in Figure 10. The points for the concentration versus signal curve were obtained by subtracting the background signal and averaging the data acquired after the system had stabilized.

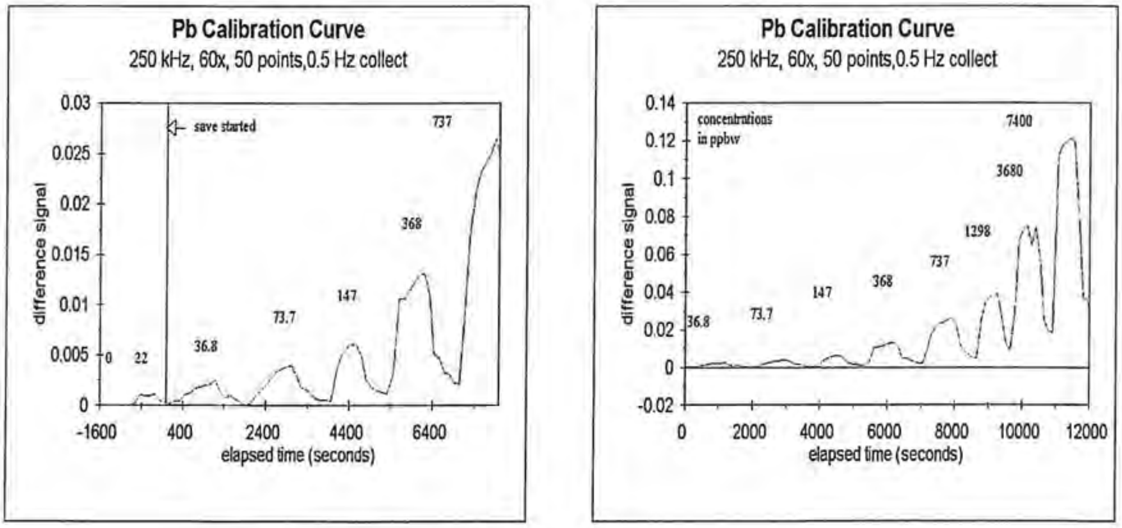


Figure 10. Two magnifications of raw data for the Pb calibration curve.

The calibration curve is shown in Figure 11. Along with the data, a linear regression of the first seven points is displayed. The comparison between the actual data (squares) and this line shows good correlation. Not shown on this plot are three points at higher concentration (1.3, 3.7, and 7.4 ppmw). These points deviate from the slope shown in Figure 8. Together, these three points appear to define a slope of about half of that obtained from the low concentration data. Changing sensitivity to larger particle sizes is a possible explanation for this behavior. We are planning a definitive examination of this phenomenon in the Phase II program.

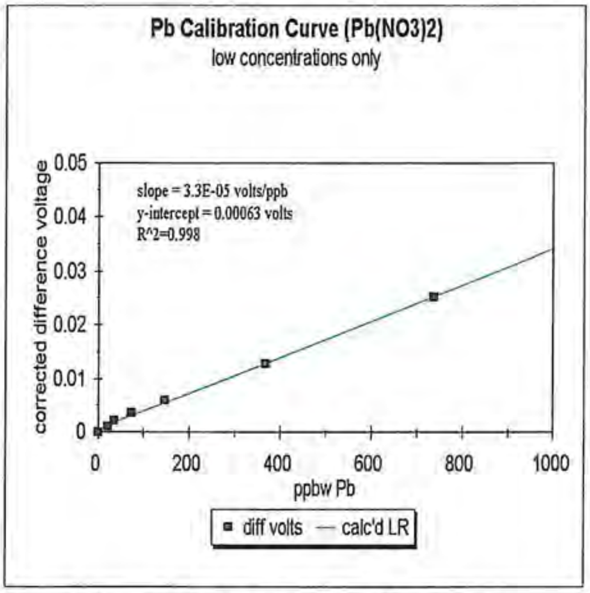


Figure 11. Lead calibration curve.

However, prior data and calibration indicate that the spark is capable of processing particles in excess of 10 µm corresponding to 100 ppm concentrations.

Another possible explanation of this effect may be the efficiency of dry particle delivery through the drying column changing with dry aerosol size (i.e., it could be a calibration effect). We have gravimetrically analyzed the output of the droplet generator and heating column by removing the spark chamber and replacing it with a filter assembly to catch the particles for weighing. This gravimetric analysis shows excellent recovery at low concentrations (95% at 100 µg/m^3 Pb) but has in some cases been seen to fall off as the particles become larger (80% has been measured at 7.4 mg/m^3). We plan to quantitatively evaluate and resolve this issue early in the Phase II program.

The two methods of determining the limit of detection (LOD) within the NIOSH[19] report on method evaluation are $3\sigma_b$ (3 times the standard deviation of multiple background measurements) and the statistical analysis measuring deviation of the individual calibration points from a linear regression of the points near the expected LOD. A $3\sigma_b$ LOD of our background data represents a limit of detectability of 8 ppbw (9.6 µg/m^3). The statistical workup approach requires more points in the region of the detection limit than were acquired in the Phase I effort. We will follow the more exacting formalism in the Phase II program. This will clearly be necessary for our method to be approved by NIOSH.

During the calibration phase of our project, we also examined the possible effects of speciation on the signal obtained. To make this determination, we generated aerosols of identical concentrations (368 ppbw) of lead chloride, nitrate and acetate. We admitted these aerosols one after another into the spark chamber, allowing the signal to return to near baseline between additions. These data showed no distinction between the three types of lead species.

3.2 Interference Measurements

The most likely interference in SIBS is spectral overlap. This overlap could occur from other elements in the sample or from the electrodes themselves. The 405.7 nm Pb atomic line and 400 nm off-line wavelengths were initially chosen because they were suitably free from electrode lines. To verify these lines are free from interference we prepared solutions of iron, aluminum, silica, calcium, and magnesium such that ppmw concentrations would result in the aerosol generator. Spectrally resolved data were then taken with a Princeton Instruments optical multichannel analyzer/monochromator combination. The results indicated there to be no spectral interferences from these elements in these wavelength regions at the bandwidth (1 nm FWHM) of the radiometer filters. Also, in preliminary experiments funded by PSI IRAD we have demonstrated the ability to detect lead in soil using SIBS without interferences from naturally-occurring humic soil components. This demonstration is an important first step towards adapting SIBS to condensed media for commercial application.

3.3 Lab Test Results

Before the instrument was taken into the field we verified it was capable of detecting lead in a firing range environment by testing it in the laboratory. Blank shells containing lead styphnate primer, only, were fired near the instrument inlet. The data indicate that our instrument will respond to lead aerosols produced from gun firing. It also responds promptly and continues to sense the lead as the aerosol cloud disperses. The observed concentrations approached 3 ppmw.

We also used this methodology to acquire data to compare the monitor results to the lead collected on the filter located immediately downstream of the spark. An example of these data are shown in Figure 12. A total of 10 shots were fired from a Smith and Wesson 357 Magnum near the instrument inlet while the monitor was obtaining real time lead concentration data. After the test, the filter was removed and sent to an independent laboratory for analysis of the lead content. The results for the data are 140 μg of lead on the filter versus 144 predicted by the monitor, as shown in Figure 9. A second comparison produced 69 μg of lead on the filter versus 100 μg predicted. The lead mass from this was obtained by integrating the data and convolving the air sampling rate (1.3 cfm).

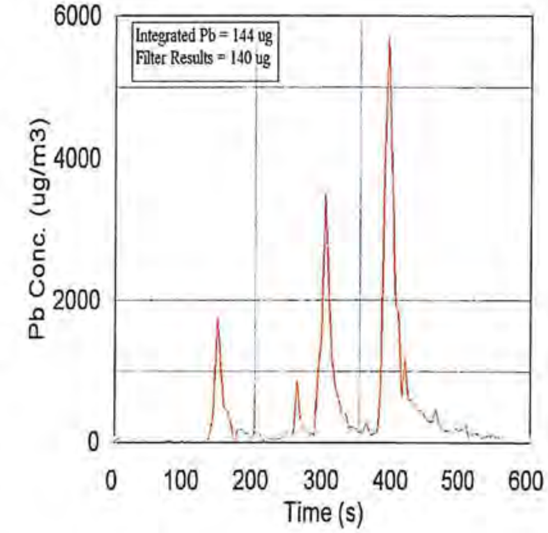


Figure 12. Filter comparison.

The Phase II program will perform a more comprehensive comparison. These preliminary data indicate, however, that the monitor is accurately responding to ambient lead aerosols.

3.4 Firing Range Tests

We performed two field tests of our instrument at a nearby firing range. The range is a state-of-the-art facility with clean air introduced behind the shooting booths. The air flow entrains the lead and carries it away from the shooters downrange through a ventilation system and into a set of filters.

For the first test we placed the lead monitor immediately in front of one of the firing booths (total of 20 booths). Active handgun firing was occurring 10 ft to our right. For these tests 15 booths (shooter locations) were active.

Our quantitative data from this test are shown in Figures 13 and 14. Figure 13 shows the data on a linear scale and Figure 14 presents the same data on a semi-log scale. The semi-log scale allows for a better assessment of our detection limit which is approximately 10 ppbw. These data also illustrate the real time nature of our monitor. The vast majority of the data show the measured lead concentration to be at or near our detection limit of 10 ppbw, well below the OSHA regulated level of 40 ppbw. Only six events were recorded showing the lead concentration exceeding 100 ppbw. The majority of these, however, were when firing occurred directly over the sample inlet. The first of these occurred near 3900 seconds when four revolver rounds were fired. The ammunition was of conventional type. The second event is near 5900 seconds when two full magazines from a semiautomatic pistol were fired overhead. Finally, in the time 7000 to 7300 seconds two different weapons were fired.

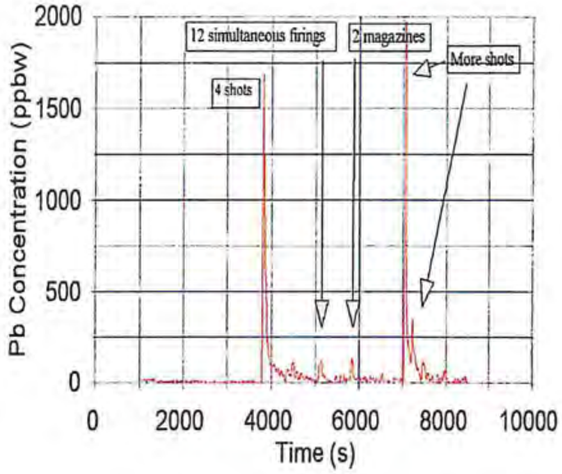


Figure 13. Linear plot of firing range data.

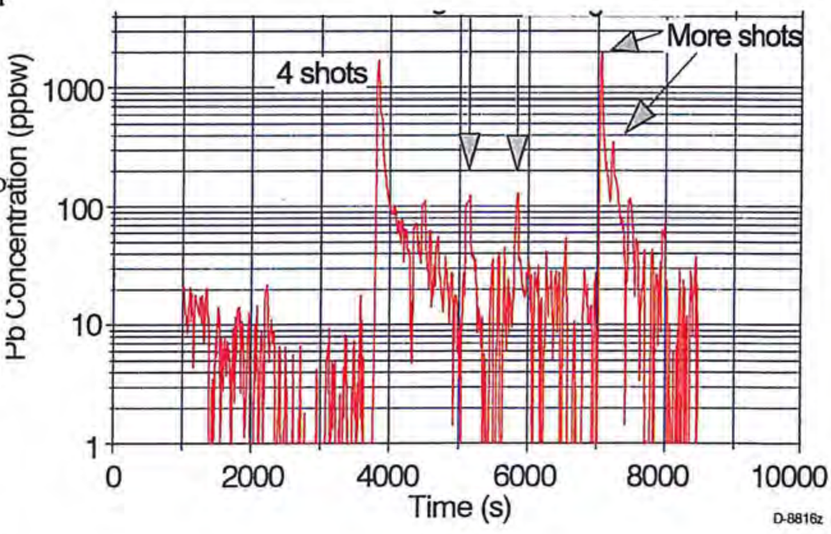


Figure 14. Semi-log plot of firing range data.

In the time period 6000 to 7000 seconds we tested ammunition specifically manufactured to be lead free. No significant lead readings were obtained when the ammunition was discharged directly over our sample inlet verifying the claims of the manufacturers. Of the remaining events, we confirm that at 5100 seconds approximately 12 individuals simultaneously fired two or more clips consecutively and that we could smell the smoke at our location.

From our understanding of firing ranges, this facility is extraordinarily clean and free of lead contamination. It was specifically designed for this purpose and our data confirm it. Other firing ranges, particularly those of older construction and in private use, often have ambient airborne lead levels approaching 2 ppmw.

We performed a second test at this range. For this entry, the lead monitor was placed down-range in the ventilation crawlspace immediately before the contaminated air passed through the filters. The data are shown in Figure 15 and a schematic of the range is shown in Figure 16. We have excluded all the data before 4000 seconds because this was before any firing was occurring and we saw no measurable lead.

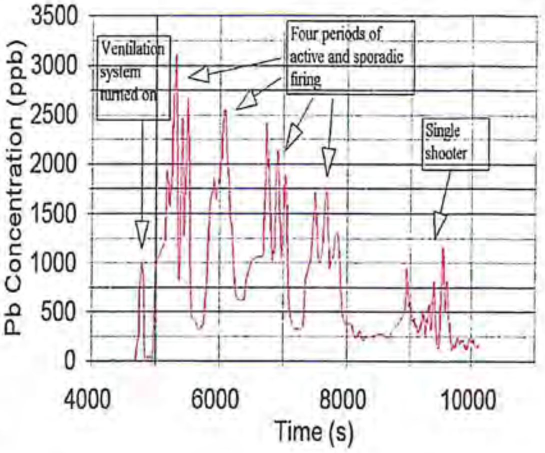


Figure 15. Crawlspace lead data.

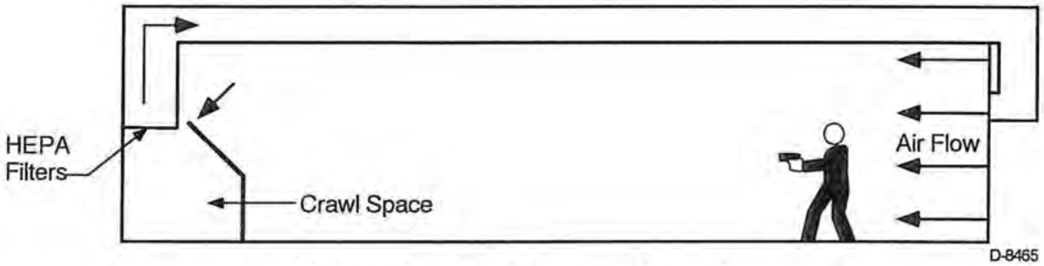


Figure 16. Firing range schematic.

The spike in lead just before 5000 seconds was caused by the ventilation system being turned on. No firing took place but the turbulent airflow stirred up lead particulate in the ventilation room producing a signal.

When active firing took place the concentration reached as high as 3000 ppbw (3 ppmw) in the ventilation room. This is consistent with our understanding that firing ranges can achieve concentrations of this order. This range, of course, reaches this level only in the ventilation crawl space just before the filters.

The data show four separate active periods of firing, each with spikes corresponding to different levels of activity. During the test we were able to correlate each of the enhanced lead concentration spikes with periods of firing. During the inactive periods the concentration always decreased.

Finally, at the end of the test we could see the lead produced from a single shooter.

3.5 Other Metals and Other Testing of the SIBS Technology

Principally under PSI IRAD funding, the PSI SIBS technology has been extended to other metals and has been tested at other facilities. During the summer of 1997 the spark probe was adapted to mount into an 8 in. schedule 10 duct to monitor lead and chromium at the rotary kiln incinerator simulator (RKIS) at Research Triangle Park, NC. This was a test co-sponsored by both EPA and DoE to identify continuous emissions monitors for RCRA metals monitoring under the proposed amendments to the 1990 Clean Air Act. Our technology successfully measured lead and chromium under the test conditions.

The instrument has recently assisted in the measurement of fugitive chromium emissions from a hard chrome plating facility in Albany, GA. This test was sponsored by USCERL and was performed in April 1998. The testing was performed side-by-side with the standard NIOSH/OSHA filter sampling method. The goal of this test was to enable the plating facility to optimize its air handling system with no danger from increased fugitive chromium emissions.

The PSI monitor performed well for this test. It was mounted in a cage, hoisted on a crane, and used to scan for chromium concentrations above the plating bath surface (typically 17 in. above the surface). Twelve different locations were tested at eight different air handling rates. The results show excellent correlation of fugitive chrome emissions as a function of ventilation rate. The PSI monitor measured fugitive emissions ranging from our detection limit (10 $\mu g/m^3$) to nearly 1000 $\mu g/m^3$. The areas of high concentration, "hot spots", correlated well with suspect areas identified with a standard smoke test. Finally, we have compared the monitor data with total chrome measurements obtained on filters. Four of five of the results agree to within ±20%.

4. CONCLUSIONS

The Phase I program successfully demonstrated the feasibility of the spark-induced breakdown spectroscopy (SIBS) technology as an ambient monitor for lead-containing aerosols and particulates. Highlights of our achievements include: assembly of a fully functional lead monitor; calibration of the system over the range 10 ppbw to 10 ppmw using a gravimetrically-determined dry aerosol generator; demonstrated 3σ detection limit of 8 ppbw; identical sensitivity to various forms of lead; freedom from spectral interferences; and successful lab tests and two tests at a firing range. We achieved all goals of the Phase I program.

The existing Phase I prototype is basically a "brass-board" device, intended for extensive laboratory characterization of the SIBS technology and sufficiently portable for field tests under well-controlled conditions. This instrumentation consists of several separate boxes. Only the spark electrode assembly is custom-made; the power supply is an off-the-shelf commercial unit and data processing and storage is performed on a stand-alone Pentium PC.

The primary objective of the proposed Phase II program is the construction and extensive testing of "alpha" prototypes of SIBS instruments. Two alpha prototypes are required to provide essentially full instrument functionality in the intended industrial product operating environment. The alpha prototype is not, however, based on a manufacturable design - it still employs selected off-the-shelf components. The alpha prototype is packaged in a single unit (or perhaps two components for certain applications, as discussed below), although substantially larger than the ultimate commercial design. Alpha units will be tested extensively in commercial environments, including lead abatement construction sites, industrial plants and indoor firing ranges. The objectives of these tests are 1) to obtain sufficient operating experience in practical situations to develop a manufacturable design, and 2) to obtain validation data to satisfy the needs of NIOSH/OSHA regulators. The first objective requires that the prototypes be tested in various sites for periods one to several weeks, under operation by site personnel with PSI personnel on location for instrument support and to document the test conditions. To meet the second objective, the alpha prototypes will include the ability to obtain standard filter samples under the prescribed NIOSH procedures, to establish the validity of SIBS data in the field.

In the proposed optional program, Physical Sciences Inc. (PSI) will extend the SIBS Instrument development to the beta design level. The beta prototype will incorporate changes to respond to experience obtained in testing of alpha units and will be a manufacturable design, with substantial inputs from our commerialization partner. Off-the-shelf components will be replaced with custom designed components where significant cost savings can be achieved. For example, data processing will be performed on dedicated specially-designed boards, rather than commercially-available Pentium processors, and the commercial power supply will be replaced with a custom design at substantial costs savings. This level of design will hopefully enable us to meet our goal of a commercial selling price in the $3000 to $5000 range. The beta unit will have a considerably smaller package than the alpha prototypes, and will probably not include the separate filter sample collection capability. The optional program will acquire and publish the data necessary for NIOSH recognition.

The following are the specific aims of the basic and optional programs:

Basic Program

- Design second generation (alpha) prototype
- Build two alpha prototypes
- Performance testing
- Field testing
- Performance evaluation

Optional Program

- Beta unit design
- Construct single beta unit
- Beta unit performance evaluation

Estimates of worker exposure to lead in the US workforce range up to 900,000.[3] The PSI lead monitor will enable better worker protection, reducing exposure thus reducing health impairment. The PSI lead monitor will augment the current NIOSH/OSHA air sampling protocols. Sensitive, real time monitoring provided by the PSI lead monitor will enable accurate identification and mapping of point sources, tracking of contaminant migration, immediate notification of concentration levels exceeding OSHA PEL and action levels, and improved evaluation of variable or periodic sources and exposure.

PSI has a commercialization partner, Mine Safety Appliances (MSA), which is interested in developing this technology as an industrial hygiene monitor. The industries or activities in which the PSI lead monitor will be used include: firing ranges (for existing ranges and during range commission), lead abatement and construction, smelting, metals processing and finishing, and battery manufacture and recycling. The PSI monitor has also demonstrated applicability to other metals and other applications. These include total chromium monitoring in hard chrome plating facilities and lead and chromium continuous emissions monitoring in stack gases. These latter applications and extensions are being pursued by PSI independently, principally with IRAD funding.

4.1 Commercialization Plan

SIBS toxic metal monitoring instruments represent a substantial business opportunity, and PSI is currently seeking partnerships with established environmental and occupational safety and health companies to commercialize a family of SIBS instrument products. Our strategy is to team with established instrument companies to take advantage of existing marketing, sales and distribution networks, with manufacturing to be provided by PSI or by our partners under license from PSI, to be negotiated. We have prepared a comprehensive business plan for commercialization of SIBS instruments and have introduced the plan to over a dozen medium to large instrument companies.

SIBS instruments address annual markets of over $100 million in four different applications:

- Industrial health monitoring
- Environmental field engineering
- Continuous emissions monitoring
- Monitoring radioactive materials at DOE remediation sites.

The industrial health monitoring market is the most attractive of these markets for the near term. Our target selling price for continuous, real-time, on-site SIBS monitor for lead (or chromium or cadmium) is $3K to $5K, which should be an affordable range for plant operators and remediation contractors. Although no definitive market studies are available for toxic metal monitors, we estimate that the annual U.S. market is on the order of $25 million or greater. This consists of at least 10% of the $120 million spent annually for monitoring lead exposures in the construction industry, plus another $12 million annual market for the thousands of industrial plants and indoor firing ranges where lead exposures must be monitored.

PSI has been conducting partnering discussions with the MSA Instruments Division of Mine Safety Appliances to introduce SIBS monitors to occupational health markets. MSA is the leading supplier, with extensive foreign operations, of industrial safety and health equipment, including respirators and protective clothing. The Instrument Division sells a well-established line of workplace monitors for O_2, CO, NO_2, H_2S, and combustible gases. MSA has thoroughly evaluated the SIBS prototypes, both at PSI and at MSA. It has currently commissioned a study by the well-known market research firm of Frost & Sullivan on the market for occupational health monitors for lead and other toxic markets. With sufficient confirmation from this market study, we anticipate that MSA and PSI will execute an agreement under which MSA will market and sell SIBS monitors to the occupational health market.

The proposed Phase II funding will play a critical role in getting SIBS instruments to the market. This funding will enable PSI to develop and thoroughly test instruments that will meet the market needs and will satisfy the regulatory requirements of NIOSH and OSHA. Our plan is to develop manufacturable prototypes under the Phase II funding, at which point the engineering development is turned over to our corporate partner. Conversely, we are not optimistic that MSA or other commercial partners would agree to invest the funds to cover the necessary development work to be performed in the Phase II effort. The market for environmental instrumentation has been very disappointing in recent years, with the result that established instrument companies have limited budgets for new product development.

Letters of user interest and commercialization partnership are provided in the Appendix. Included among these are letters from Mine Safety Applicances, our commercialization partner, and Batta Environmental Associates Inc., a leading company in lead abatement projects.

PSI has filed a patent application on the SIBS technology. This application pre-dates the NIOSH Phase I program.

4.2 Summary of Instrument Capabilities

Species measured - Lead in airborne aerosols and particulate

Technology basis - Atomic emission from spark-induced breakdown spectroscopy

Spark energy - 5J

Sampling frequency - 0.5-1 Hz with data reported at 60 shot average intervals

Detection limit - 5-10 ppbw

Dynamic range - Linear response from 0-1000 ppbw with detection extending to >10 ppmw

Response time - 2-3 minutes (at 1 Hz operation)

Interferences - No known interferences for lead encountered at firing ranges. Non-interference from iron, silicon, aluminum, calcium or magnesium demonstrated in the laboratory.

ACKNOWLEDGMENTS

The authors would like to thank Greg Lord of PSI for use of the Smith and Wesson handgun and primer-only shells for the lab tests. We would also like to thank the folks at the firing range for their cooperation and enthusiasm.

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6. LIST OF PRESENT AND POSSIBLE FUTURE PUBLICATIONS

PSI has presented results of the SIBS technology development and the NIOSH Phase I program at Pittcon 98 and an AWMA conference entitled "Environmental Innovation in the Metals Industry for the 21st Century" in March 1998. Copies of the presentation materials for these two conferences were included in the Phase II proposal appendix.

We have recently submitted an abstract entitled "Real Time Monitoring of Airborne Metals" to the SPIE conference on Environmental Monitoring and Remediation Techniques scheduled for 1-6 November 1998. We have not yet been informed of the status of this planned presentation.

DEPARTMENT OF HEALTH AND HUMAN SERVICES

Public Health Service
Centers for Disease Control
and Prevention (CDC)

Memorandum

Date: March 27, 2001

From: Roy M. Fleming, Sc.D., Director, Research Grants Program RMF
Office of Extramural Programs, NIOSH, D30

Subject: Final Report Submitted for Entry into NTIS for Grant 1 R43 OH003505-01

To: William D. Bennett
Data Systems Team, Information Resources Branch, EID, NIOSH, P03/C18

The attached final report has been received from the principal investigator on the subject NIOSH grant. If this document is forwarded to the National Technical Information Service, please let us know when a document number is known so that we can inform anyone who inquires about this final report.

Any publications that are included with this report are highlighted on the list below.

Attachment
cc: Sherri Diana, EID, P03/C13

List of Publications - None

Title:	Airborne Heavy Metal Monitor
Investigator:	Mark E. Fraser, Ph.D.
Affiliation:	Physical Sciences, Inc.
City & State:	Andover, MA
Telephone:	(508) 689-0003
Award Number:	1 R43 OH003505-01
Start & End Date:	9/30/1997–5/31/1998
Total Project Cost:	$97,162
Program Area:	Exposure Assessment Methods
Key Words:	

Abstract:

During the Phase I effort, key issues of sensitivity and calibration were to be explored and a prototype monitor for lead constructed and tested. The objectives of the proposed Phase I program were outlined in the proposal as follows: (1) Build a simple, field monitor for lead-laden aerosols based on PSI experience with spark-induced breakdown spectroscopy. (2) Calibrate the monitor with lead-containing aerosols generated by PSI's in-house dry aerosol generator, and 3) test the unit offsite at a firing range.

Phase I achievements include: (1) A fully functional lead monitor was assembled. It is composed of a spark power supply, spark and sample chamber, radiometer detection package, and computer for data acquisition and display. (2) The monitor has been calibrated, and shown to be independent of the chemical forms of lead tested. The detection scheme has been proven to be free of potential spectral interferences from iron, silicon, aluminum, magnesium and potassium. (3) The functionality of the lead monitor was tested in-house by detecting the lead from handgun blanks containing lead styphnate primer. Simultaneous data from the monitor and lead collected on an internal filter compared well (analyzed by an environmental laboratory). The lead monitor was tested at a firing range, and data collected correlated well with shooting events. The observed concentrations were consistent with typical levels reported in the literature.

The Phase I prototype is basically a "brass-board" device, intended for extensive laboratory characterization of the SIBS technology and sufficiently portable for field tests under well-controlled conditions. This instrumentation consists of several separate boxed. Only the spark electrode assembly is custom-made; the power supply is an off-the-shelf commercial unit and data processing and storage is performed on a stand-alone Pentium PC.

Publications

No publications to date.