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GEOENVIRONMENTAL SITE CHARACTERIZATION TO TREATMENT: LEAD CONTAMINATED FIRING RANGE CASE STUDY

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ABSTRACT

The methodologies employed and the results obtained during the performance of a comprehensive geoenvironmental site characterization case study are presented. The study demonstrates the need to integrate research tools from various disciplines including geotechnical, analytical and mineralogical specialties in order to develop a thorough understanding of both the nature and extent of the environmental issues associated with the site and the most viable alternatives for its remediation. Particle size distribution coupled with contaminant fractionation studies and mineralogical and micromorphological analyses were performed on the soil samples collected onsite to identify the metals present, their concentrations and the mechanisms of transformation. Lead fragments found in the soil samples were analyzed by means of X-ray diffraction (XRD) and scanning electron microscopy (SEM). Quantitative phase analysis studies showed that the fine soil fractions contained considerable amounts of lead carbonates, which owing to their colloidal nature could not be readily removed using gravitational methods. To mitigate this deficiency, a bench-scale chemical treatment experiment based on dissolution of the Pb was performed. Although the study is still in progress, the benefits derived from using the multi-disciplinary approach for site characterization described herein warrant consideration by others who may face similar challenges in the future.

INTRODUCTION

Site characterization studies for small arms firing ranges are performed to identify whether the facility is in compliance with environmental regulations and/or if the facility poses an environmental hazard to its users or those who live or work nearby. In many cases the site characterization, while compliant with U S Environmental Protection Agency (EPA) requirements, is conducted from a limited perspective. The performance and interpretation of a series of regulatory analytical tests that measure total metals concentrations and Toxicity Characteristic Leaching Procedure (TCLP) levels, using samples collected at selected locations across the site, will identify whether an environmental problem exists. However, without benefit of additional data necessary to characterize the soil, groundwater and overall site conditions, neither the source nor the extent of the problem or the viability of various remedial alternatives can be reliably addressed.

This paper presents a case history involving a site characterization of the soil projectile impact berms at the Armaments Technology Facility (ATF) at Picatinny Arsenal, New Jersey. The integration of data collected from geotechnical, analytical and mineralogical studies was vital in both the identification of the environmental issues related to the site as well as providing a focused coherent approach to their mitigation. While the authors do not believe that all sites warrant the level of investigation, testing and analysis presented below, the recognition that such options are available and can be applied when circumstances dictate will benefit those responsible for the performance of such efforts.

BACKGROUND

The site characterization methodology presented in this paper was developed during the implementation of a research effort at the Picatinny Arsenal (Picatinny) in New Jersey. Picatinny is part of the US Army Research, Development and Engineering Command (ARDEC) whose mission includes the design of new weaponry and the improvement of existing weapons used by the Army. An integral element used in this effort is the Armaments Technology Facility (ATF) located at Picatinny. The ATF consists of two indoor firing ranges, one a 100-m long facility and the other a 300-m long facility. The ranges are located immediately adjacent to each other and are used in the testing and evaluation of small arms designs and/or modifications.

Soil berms are located at the extreme far end of each range and are used to control and collect the projectiles fired within the ranges. Due to the high usage rate of the facility, the soil contained within the first five feet of the impact face of the berm had become significantly pulverized and laden with heavy metal fragments. These conditions posed serious safety problems due to the increased level of dust emitted from the face during firing as well as the greater tendency for uncontrolled ricochet of the projectiles upon impact with the metal fragments.

To mitigate the problem, it was decided that the soil comprising the first five feet of the berm would be removed and replaced with fresh material. However, the only option available for final disposition of the contaminated soil appeared to be landfill disposal. This problem was

brought to the attention of the RangeSafe group located at Picatinny whose mission is the identification, testing, validation and implementation of new technologies that enhance the safety, functionality and economic aspects of firing range operations. Following an evaluation of the problem and its potential solutions, RangeSafe personnel realized that from an economical perspective, landfill disposal was the most cost-effective means of for final disposition of the contaminated soil. However, consistent with their mission, it was decided to initiate a research effort intended to identify alternate options that both minimized costs and at the same time were more environmentally compliant.

To this end, Stevens Institute of Technology was awarded a research contract by ARDEC to conduct such a study. The method of identifying and integrating relevant aspects of the geotechnical, analytical and mineralogical disciplines forms the basis of what the authors believe to be an effective approach for use when implementing environmental site characterizations.

EXPERIMENTAL METHODOLOGY

The methods used to collect and test the soil and metals samples are discussed below.

Sample Collection

The soil samples used in this study were collected prior to, during and subsequent to the soil removal effort conducted at both the 100-m and 300-m firing ranges at the ATF. As indicated above, the removal and replacement of the soil located in the first five feet of each berm was necessary due to the pulverization of the soil that had resulted from repeated projectile impacts and the accumulation of numerous projectiles at and near the impact faces. The pulverization was causing an unacceptable quantity of dust to be released during firing activities and the buildup of projectiles was increasing the likelihood of ricochet.

The initial sampling effort involved the collection of soil samples (in 5-gal plastic containers) directly from the surface of each range and a sample of virgin soil (not previously fired upon) that had been stored in a supply hopper located on the roof of the range. Information provided by the ATF operations personnel indicated that soil used to construct both berms had been supplied from a single source and over the same time period and should be essentially the same for each range.

A second sampling effort was performed during soil removal activities at each berm. In order to determine the variation in the soil conditions as a function of the depth into the berms, it was decided that the soil would be removed in successive 1-ft. thick layers parallel to the face of the berm. A schematic of the removal process is shown in Fig. 1.

During berm excavation, individual stockpiles of the soil in each layer were created. The material was then screened to remove oversized soil particles, projectiles and any undetonated rounds that may have existed within the soil. The screening process was initiated using two screens. A 1/8-in. upper screen was used to remove the larger materials and reduce the loading imposed on the lower, finer #10 (2.36-mm) screen.
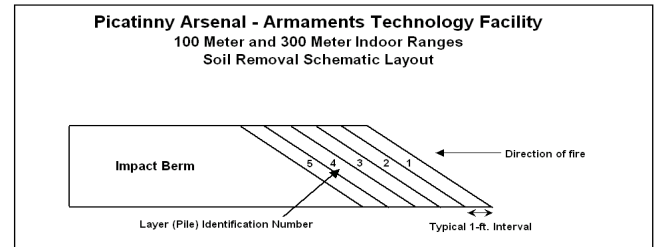

Fig. 1 – Material Removal Schematic for ATF Soil Impact Berms

At the outset of work on at the 300-m range, excessive clogging of both screens occurred and was caused by the presence of moisture within the soil. The moisture existed as a result of range maintenance activities that involved the use of a water spray applied to the surface of the berm to provide dust control during test firing operations. Attempts to mitigate the problem using a ¼-in. upper screen and a 1/8-in. lower screen were likewise unsuccessful. Following a review of the historical usage of the range, it was decided that munitions fired at this location that posed a potential safety issue could be removed by using a 3/8-in. by 4-in. slotted screen. This screen was then used to process the remainder of material from this range. However, approximately 3-tons of soil that had been successfully screened over the #10 sieve prior to changing the screening method were stored separately from the remainder of the processed material. The over-sized soil and projectile particles collected on the 3/8-in. by 4-in. slotted screen were stored in 90-gal metal drums. A photograph showing the typical contents of the drums is presented in Fig. 2.

Fig. 2 – Oversized Projectile Fragments Removed From ATF Soil Impact Berms

After screening, the soil was loaded into 20-CY waste containers, with each layer being placed in separate containers, to the extent practical. One soil sample of the contents of each container was collected in a 5-gal. plastic bucket during loading by periodically obtaining small portions of material from the bucket of the loader as the soil was being placed in the container.

Essentially the same procedures were used for the removal and sampling of the soil located at the face of the 100-m range. However, due to the drier condition of the soil found in this range, it was possible to use a standard 3/8-in. square screen instead of the 3/8-in slotted screen used in the 300-m range. This change is believed to be relatively insignificant since the overall quantity of material coarser than the 3/8-in. sieve was

less than 5% by dry weight.

Additional sampling of the containers for both ranges was also conducted later in the research program. In this case the samples were collected by using a hand auger to retrieve material over the entire depth of the soil present in each container at 4-6 locations equally spaced along the centerline of the container.

Testing Procedures

In order to properly characterize the various materials (soil and metals) present in the berms, a comprehensive testing program was performed involving the collection of geotechnical, analytical and mineralogical data. The types of tests performed in each category are listed below along with a brief description of any modifications/deviations that may have been employed during implementation of standardized procedures.

Geotechnical Testing.
- *Water Content – ASTM D2216-92*: No deviations from the standard method were required.
- *Liquid Limit, Plastic Limit and Plasticity Index – ASTM D4318-84*: No deviations from the standard method were required. However, it should be noted that the liquid limit ("LL") and plastic limit ("PL") tests were conducted on samples that were prepared in their "as-sampled" state; i.e. not air-dried.
- *Particle Size Analysis – ASTM C136-96a*: No deviations from standard method were required.
- *pH – ASTM D4972-95*: Five deviations from the standard were employed. These were:
  a. The maximum particle size of the test specimens was finer than the 3/8-in. sieve and not the #10 sieve;
  b. The soil was tested in its "as sampled" condition and was not air dried prior to testing;
  c. Deionized water was used instead of distilled water;
  d. A second pH measurement using 0.01 M $CaCl_2$ was not performed;
  e. pH measurements were also made at a 2:1 deionized water to soil (by dry weight) ratio in addition to the 1:1 ratio specified in the test procedure.

Analytical Testing.
- *Total Digestion Test* – EPA Method 3050B: No deviations in the test procedure were made.
- *Toxicity Characteristic Leaching Procedure (TCLP)Test* – EPA Method 1311: No deviations in the test procedure were made.

For both of the analytical tests indicated above, concentrations of the soluble lead were determined using an inductively coupled plasma optical emission spectrometer (ICP-OES) (Varian Vista-MPX, Palo Alto, CA)

Mineralogical Testing. The mineralogical testing performed for this study involved both X-Ray Diffraction (XRD) Analysis and optical and Scanning Electron Microscopy. (SEM). The procedures used in the execution of these analyses are as follows:

X-Ray Diffraction Analysis. The XRD analyses of the ATF soil were performed on material obtained within the first two feet of the 100-m range impact surface. Sample preparation involved an initial screening of the material through a #4 (4.76-mm) sieve, since the material present in the fraction coarser than #4 sieve consisted predominately of projectiles, projectile jackets and various other metallic fragments associated with test firing. The –#4 mesh soil materials (soil that passes the #4 sieve) were weighed and fractionated in accordance with the method described below.

Duplicate sub-samples (Sample A and Sample B) of about one hundred grams of the soil were obtained using a sample splitter. Each sub-sample series was mixed with 250 ml of deionized (DI) water and stirred using a magnetic stirrer. The magnetic materials collected on the stirring bar were removed in order to form a separate fraction, identified as the "Magnetics Fraction". After mixing, the suspension containing the finer fractions was collected in a separate beaker. The coarse fractions were washed ultrasonically to achieve a thorough separation of the finer particles from the coarse fractions. Subsequently, the water used for fractionation was combined with the water and soil collected from the ultrasonic treatment. The total volume of water used during fractionation was approximately 1±0.1L. This process led to separation of each soil sub-sample (Samples A and B) into nine fractions identified in accordance with the range of particle sizes (based upon ASTM standard sieve mesh numbers) that they represented. The magnetic fraction was not separated on a size-basis. The nine fractions are identified as follows: magnetic, +4, -4+10, -10+40, -40+100, -100+200, -200+325, -325 down, -325 up. Fractions -325 down and -325 up were obtained from the material which passed through a #325 mesh sieve in the following manner: the –#325 mesh suspension (~ 1000ml) was left to settle for 16 hours, then the settled materials (-325 down) were separated from the particles still in suspension (-325 up fractions). Each fraction was then collected on a 0.45µm membrane filter under suction, washed with a small amount of acetone and air dried at room temperature. Soil fractions of Sample A were used for optical, SEM and XRD studies, while Sample B fractions were used for chemical analyses. Independently prepared samples were also chemically analyzed for quality assurance purposes and were designated as Sample C.

The XRD analyses of the soils conducted as part of this study were performed using a computer-automated diffractometer (Rigaku DXR-3000) that uses Bragg-Brentano geometry. The X-ray source was a Cu anode operating at 40 kV and 40 mA with graphite-monochromated CuKα radiation. Data were collected between 3 and 70° in 2 theta (Θ) with a step size of 0.05° and count time of 5 sec per step. Silicon powder (NBS 640 a = 5.43088 Å) was used to correct 2Θ values.

Scanning Electron Microscopy. Analyses of the soil particles and metal fragments were made using a Karl Zeiss Model LEO890 Scanning Electron Microscope with Energy Dispersive X-ray Analysis (EDAX) capability for compositional analysis of the specimen being analyzed.

Physical Separation Testing

The physical separation testing phase of the study was initiated following a comprehensive review and analysis of the geotechnical, analytical and mineralogical characterization data. One of the key items identified in the review was that 71% to 86% of the total amount of lead contained in the soil exists in the gravel and sand sized soil fraction. Based upon this factor, a broad review of existing literature and discussions with treatment equipment manufacturers, it was decided that an investigation as to the viability of removing the lead and other heavy metals using physical separation techniques common to the "soil washing" technology was justified. Given that the degree of difficulty for effective separation typically increases with decreasing particle size, it was further decided to initiate work using material finer than the #10 sieve and larger than the #200 sieve (medium to fine sand size range). Furthermore, consistent with the intent of the study to identify cost-effective alternatives to landfill disposal, a spiral concentrator was selected to be the first of the candidate components to be evaluated since this type of device is one of the least expensive separation mechanisms to purchase and operate. This choice was also in line with the -#10 +#200 particle size range selected for investigation, because this range is at the lower end of the operational range of conventional concentrators.

Spiral concentrators are common to the mining industry and are used routinely to fractionate materials containing particles of different densities. Basically, the spiral concentrator is a continuous helically-shaped channel located around a central axis. The configuration and number of flights or "turns" incorporated into the system is dependent upon the specific application for which the concentrator will be used. A typical five-turn unit is shown in Fig. 3. During operation, the material is discharged in slurry form at the top of the spiral and in the course of the downward travel through the spiral the denser, larger particles tend to collect along the interior of the channel while, due to centrifugal forces, the lighter particles report to the outer edges of the channel. A series of finger levers is located at the bottom of the device and the segregation of the discharged materials is done manually using these levers. The selection of the lever settings or "cut points" is made by the operator and is based upon both the visual inspection of the discharge and experience gained following analysis of post-treated material. While appearing crude in its level of sophistication, this equipment has been proven to provide an effective, low-cost means of material separation.

While the device is simple in appearance, its performance is based upon numerous parameters such as the "pitch" or angular orientation of the flights, the number of flights, the size distribution and shape of the particles being separated, the magnitude of the density differences between components, and the solids concentration and flow rate during operation. Other, less obvious but much more complex phenomena cited in the literature (Atasoy and Spottiswood, 1995; Davies et. al., 1991; Holtham, 1992;) such as hindered settling, Bagnold forces, and existence of various flow regimes within the slurry as it travels down the spiral all but eliminate a theoretical prediction of the effectiveness of the separation process. As such, pilot-scale tests are usually necessary to reliably evaluate the performance of a specific spiral concentrator for the soil under consideration.

The initial round of spiral concentration testing was performed using a

Linatex five-turn spiral concentrator with a medium pitch orientation. A composite sample created by combining equal masses of material from the Layers 1&2 container of the 100-m range and the Layer 1 soil from the 300-m range was used in the evaluation. The soil was prepared for testing by first removing the material coarser than the #10 sieve and finer than the #200 sieve since this was reported by the manufacturer to be the particle size range over which this unit was to be most effective.

Fig. 3 – Five-Turn Spiral Concentrator

A second series of tests was performed following analysis of the results obtained during the initial evaluation. For this work, a Linatex seven-turn spiral concentrator was used in place of the five-turn unit. The change was made to determine if the increased length of travel provided in the seven-turn system would enhance the efficiency of the metals removal. Further, the basic -#10 +#200 feed material was separated into two particle size ranges instead of one. This was done to establish if the degree of the metals segregation could be enhanced if the soil feed was composed of particles having a greater uniformity in size. Therefore, the basic feed was separated into one size range consisting of -#10 to +#50 material and a second containing the -#50 to +#200 particles. The material separations at the #10 and #200 sizes were made using sieves, however an elutriation system was used to effect the separation at about the #50 sieve size. Therefore, it is likely that finer lead particles were present in the coarser size fraction since being denser, they would report with the coarser soil particles.

Chemical Extraction Testing.

A bench-scale testing program to assess the efficacy of chemical extraction of the heavy metals, particularly lead, was initiated as a result of, and subsequent to, a review of the findings provided in the physical separation studies. The initial spiral concentrator results revealed that only two of the 19 treated soil fractions contained total lead concentrations below the target value for the program of 600-mg/kg. Therefore, while additional study options were being considered regarding the spiral concentrator and other physical separation processes, it was decided to initiate studies aimed at identifying the optimal conditions for chemical extraction of the lead should further reduction by a means other than physical separation be required.

An initial investigation using both acetic and nitric acids was performed to assess the lead solubilization capacity for each. While it would be expected that the nitric acid, at the same molarity, would provide a more thorough dissolution and reach thermodynamic equilibrium in a shorter time than acetic acid, the latter was included in the study based upon a number of other considerations including:
- Ions (acetate) are non-hazardous.
- Acetic acid is more selective in the dissolution process of the soil – lead carbonate (cerrussite) matrix.
- Acetic acid is weaker than either nitric or hydrochloric acid and would be less hazardous to personnel and less corrosive to equipment.
- Lead complexes generated during solution are strong with high solubility in water.

The experiment involved first the preparation of individual sample aliquots 10-g to 12-g in weight which were obtained using a riffle splitter. The soil used in the testing was taken from the 100-m range (Layers 1 & 2 sample) and was representative of the entire distribution of particle sizes present in the sample. The acid solutions were prepared at molecular concentrations and resulting pH values required to cover the range of interest for the study. For the acetic acid the pH of the solutions ranged from approximately 5.0 to 2.6, while for the nitric acid the pH range was from about 4.5 to values too low for reliable pH measurements to be obtained (<0.9).

The soil aliquots were then placed in 125-ml capacity polyethylene bottles and the test solution was added using a 10:1 liquid to solid ratio in all cases. Each bottle was mixed over a 24-hr. period using the same rotating tumbler used in TCLP testing. Mixing by means of the tumbler was selected in lieu of a glass beaker-magnetic stirrer alternative so as to minimize the potential for breakdown of the particles. Such a reduction in particle size could lead to erroneous results (e.g., a higher solubility potential of the finer particles that could result from stirrer-induced grinding during the mixing process). At the conclusion of the mixing period, the samples were vacuum-filtered over a 0.45 µm glass fiber filter. Aliquots of the aqueous extract were taken for both measurements of the post-treatment pH and the lead concentration in solution. The lead concentration measurements were obtained using an ICP-OES.

RESULTS AND DISCUSSION

The following sections present the various test results obtained from the geotechnical, analytical and mineralogical analyses performed for this study and discuss their significance.

Geotechnical Test Results

The results from the geotechnical analyses are summarized in Table 1, with graphical presentations of the particle-size data in Figs. 4 through 7.

Water Content. The water content of the soil removed from the impact berm at the 100-m range varied from 0.8% to 4.0% while the range of water content at the 300-m impact berm was from 1.0% to 12.1%. Water content variability can be attributed to range maintenance operations. More specifically, standard operating procedures at each range required that a continuous water spray be applied to the soil located at the face of the berm, in essentially Layer 1, while firing was in progress to control dust emitted as a result of projectile impact. This is reflected in the water content data with the Layer 1 values being larger than the values for other layers for both ranges. Given the relative ease with which water can flow through the sandy soil comprising each berm, it is likely that the interior areas of the berms become wetted and remain so, particularly since the ranges are both indoors and not subject to conventional evapo-transpiration cycles as in the case of soils found in outdoor ranges. The presence of water within the soil along with the isolation of the wetted soil within an enclosed area having limited ventilation and climate control essentially creates an incubator within which transformation of the heavy metals into various species is facilitated and accelerated. It should be noted however, that due to the need to combine various layers within a given storage container, a conclusive statement as to the variation of water content as a function of the distance into berm cannot be reliably made.

Particle-Size Analysis. The results of the particle-size analyses performed for this study are presented in Figs. 4 through 7. Figure 4 presents the grain size distribution of soil samples collected at the face of the impact berm at each range in their pre-removal condition. Also shown in this figure is the grain size distribution of soil used to construct the berms prior to its being fired upon. Examination of this data reveals the following:
- The soil prior to use in the berm (virgin soil) is a medium to fine sand containing mica and occasional sea-shell fragments. No metal fragments or other deleterious materials were observed.

- A significant quantity of material larger than the #10 sieve is present in the soil samples collected at the face of both berms. This is attributed to the projectile fragments that collect on the berm surface after firing. Visual examination revealed that essentially all of this material is heavy metal fragments.

Table 1. Geotechnical Index Property Test Summary for ATF Impact Berm Soils from 100-m and 300-m Ranges

Range	Layer No.	Storage Container No.	Scalping Screen Size	Water Content (as-sampled) %	pH Water to Solids Ratio 1:1	2:1	3/4-in	3/8-in	#4	#10	#40	#200	#400	USCS Symbol for Total Sample
PRE-REMOVAL DATA														
---	Virgin Soil	---	---	---	7.64	7.87	100.0	100.0	100.0	99.8	84.2	0.8	---	SP
100-M-1	1- Face	N/A	Pre-scalp	---	---	---	100.0	97.5	95.3	91.7	80.8	34.1	---	SM
100-M-2	1- Face	N/A	Pre-scalp	---	---	---	100.0	98.9	92.3	88.3	78.1	24.3	---	SM
300-M	1-Face	N/A	Pre-scalp	---	---	---	100.0	86.0	81.1	75.8	64.3	28.9	---	SM
POST-REMOVAL DATA														
100-M	1 & 2	9697	3/8-in.	4.0	8.72	8.90	100.0	100.0	93.3	90.7	77.4	19.0	15.5	SM
100-M	2 & 3	9891	3/8-in.	0.8	8.90	8.99	100.0	100.0	98.3	97.1	81.6	8.6	6.0	SP-SM
100-M	4	9715	3/8-in.	2.9	8.83	8.98	100.0	100.0	99.7	99.4	84.3	2.6	1.7	SP
100-M	5	9939	3/8-in.	0.9	8.48	8.40	100.0	100.0	99.8	99.8	83.7	1.8	1.3	SP
300-M	1	8841	3/8-in. x 4-in.	12.1	8.59	8.76	100.0	96.2	92.1	89.3	75.3	16.3	15.0	SM
300-M	2	9872	3/8-in. x 4-in.	3.5	8.61	8.85	100.0	97.1	95.9	94.8	80.6	9.9	7.2	SP-SM
300-M	2 & 4	0038	3/8-in. x 4-in.	2.2	8.63	8.78	100.0	99.1	95.8	94.5	80.1	6.5	4.4	SP-SM
300-M	3	9114	3/8-in. x 4-in.	3.7	8.58	8.65	100.0	99.6	98.1	97.7	83.4	8.1	5.8	SP-SM
300-M	4	9112	3/8-in. x 4-in.	1.0	8.66	8.91	100.0	98.8	97.4	96.8	81.4	3.4	2.3	SP
300-M	3,4,5	9814	3/8-in. x 4-in.	1.8	8.67	8.81	100.0	100.0	99.5	99.2	83.6	2.9	2.1	SP
300-M	5	9470	#10	1.0	8.56	8.80	100.0	100.0	100.0	97.7	83.4	8.1	5.8	SP

- The soil collected at the face of the berms has undergone a significant degree of particle breakdown resulting from numerous projectile impacts. This effect is shown clearly in Fig. 5, which presents the particle-size distribution of the same samples as shown in Fig. 4, but without the +#10 size metal and projectile fragments. As can be seen from this comparison, there is a substantial increase in the percentage of material passing each sieve size. For example, the quantity of soil finer than the #200 sieve (0.074-mm) increased from 0.8% in the original soil to as much as 35.8% for the 100-m-1 sample and 35.6% for the 300-m sample.
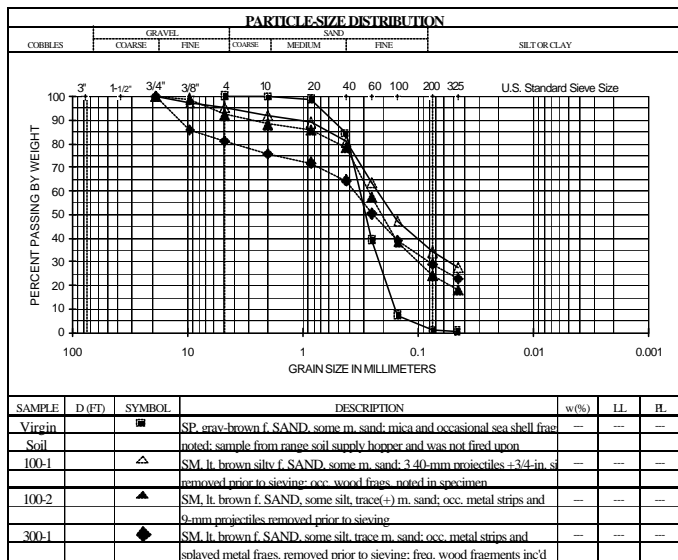

Fig. 4. Particle Size Distribution of ATF Impact Berm Soil From Face of 100-m and 300-m Berms
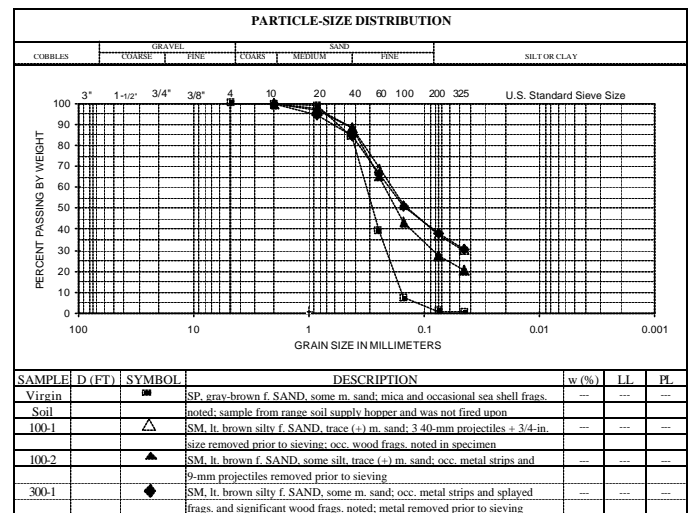

Fig. 5. Particle Size Distribution of ATF Impact Berm Soil From Face of 100-m and 300-m Berms (Material Passing #10 Sieve Only)

The results of the sieve analyses performed using the post-removal screened material stored in the 20-CY waste containers are shown in Figs. 6 and 7 for the 100-m and 300-m ranges, respectively. A review of these data reveals the following:

- The soil nearest the impact surface of each berm has sustained the greatest amount of particle breakdown as is shown by the gradual decrease in the fineness of the soil gradation as a function of distance into the berm (increasing layer number). Since some storage containers have a mixture of various layers, the magnitude of the breakdown versus depth from the impact face cannot be reliably assessed, however the trend is valid.
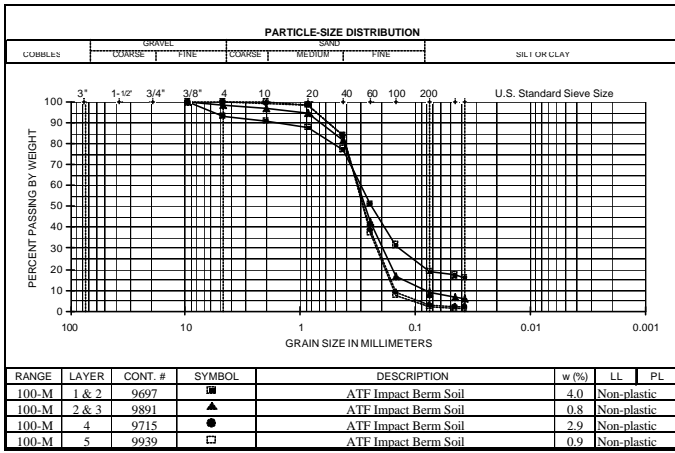
PARTICLE-SIZE DISTRIBUTION

RANGE	LAYER	CONT. #	SYMBOL	DESCRIPTION	w (%)	LL	PL
100-M	1 & 2	9697	▣	ATF Impact Berm Soil	4.0	Non-plastic	
100-M	2 & 3	9891	▲	ATF Impact Berm Soil	0.8	Non-plastic	
100-M	4	9715	●	ATF Impact Berm Soil	2.9	Non-plastic	
100-M	5	9939	□	ATF Impact Berm Soil	0.9	Non-plastic	

Fig. 6. Post-Removal Particle-size Distribution of Impact Berm Soil from 100-m ATF Range

• The quantity of projectile fragments coarser than the #10 sieve decreases with distance into the impact surface for both berms indicating a reduction in the number of projectiles that were present at these depths. It must be noted that some variation in the projectile concentrations is also likely since different screens having different opening sizes were used during the removal and screening process for each range. However, since the same type of screen was used during removal within a given range, comparisons of data for samples collected within a range are believed to more reliable than comparisons made between ranges.

PARTICLE-SIZE DISTRIBUTION

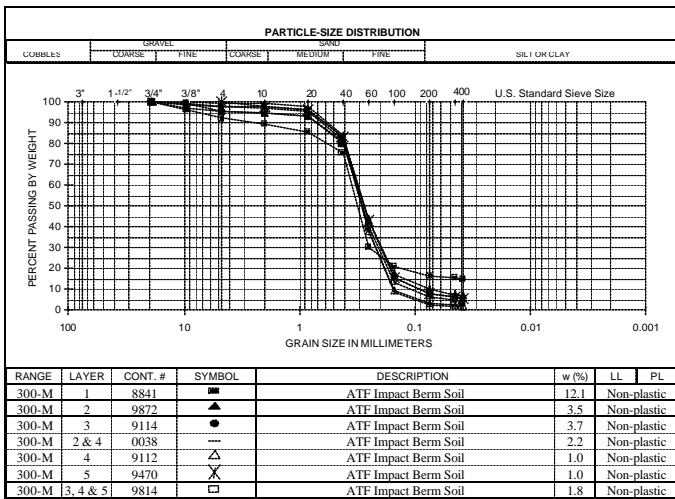| RANGE	LAYER	CONT. #	SYMBOL	DESCRIPTION	w (%)	LL	PL
300-M	1	8841	▥	ATF Impact Berm Soil	12.1	Non-plastic	
300-M	2	9872	▲	ATF Impact Berm Soil	3.5	Non-plastic	
300-M	3	9114	●	ATF Impact Berm Soil	3.7	Non-plastic	
300-M	2 & 4	0038	----	ATF Impact Berm Soil	2.2	Non-plastic	
300-M	4	9112	△	ATF Impact Berm Soil	1.0	Non-plastic	
300-M	5	9470	✕	ATF Impact Berm Soil	1.0	Non-plastic	
300-M	3, 4 & 5	9814	□	ATF Impact Berm Soil	1.8	Non-plastic	

Fig. 7. Post-Removal Particle-size Distribution of Impact Berm Soil from 300-m ATF Range

Liquid Limit, Plastic Limit and Plasticity Index. The liquid limit, plastic limit and plasticity index are geotechnical index properties that are indicative of the clay content of a soil. In all cases the soil samples did not contain sufficient quantities of clay particles to exhibit plasticity and are therefore classified as non-plastic. The absence of clay particles in the samples, as inferred by the absence of plasticity, is corroborated by results obtained from the XRD analyses, which indicated the same finding.

Soil pH – The soil pH test results are summarized in Table 1. A review of this information indicates the pH of the original soil in its unused state when measured at a ratio of 1:1 (water:solids) is 7.6 while values for the same soil following use in the impact berms range from 8.5 to 8.9 indicating an increase in the alkalinity of the soil. This is believed to have occurred as a result of the prolonged exposure to moisture and the subsequent physicochemical transformation of the various metal fragments that were deposited in the soil during firing operations. A slight increase in the pH was measured for all samples at an increased water to soil ratio of 2:1. This could be explained by the reduction of the hydrogen ion concentration due to the introduction of the additional water.

Analytical Test Results

The results of the analytical tests performed during the site characterization phase of this study are presented Table 2. Total lead concentrations measured using subsamples representative of the entire range of particle sizes contained in the respective samples range from 20,000 mg/kg to 480 mg/kg and generally decrease with increasing distance from the face of each berm.

Total lead concentration data are also presented for selected particle size ranges and vary from 132,000 mg/kg to 6,510 mg/kg. While the magnitude of the values varies between the two range berms, the concentration trends are similar with highly elevated lead concentrations in the fine gravel to medium sand particle sizes (-3/8-in. to +#40) where the material found is primarily metallic projectile fragments. Lower lead concentrations are found in the fine sand particle size but increase substantially with decreasing particle size. However, given that the quantity of soil comprising each particle size range varies, it is necessary to determine not only the concentration of lead in each size fraction, but the percentage of the total quantity of lead contained in each fractionated range. These values are presented in Fig. 8 and reveal a similar trend for both soils. However, a slightly greater quantity of lead is found in the coarser size fractions at the 300-m range and the finer lead fraction is substantially higher in the 100-m range. One explanation for this behavior may be that the projectiles undergo a greater degree of fragmentation in the 100-m range due to a higher impact velocity given the shorter distance traveled and lesser energy lost prior to impact.
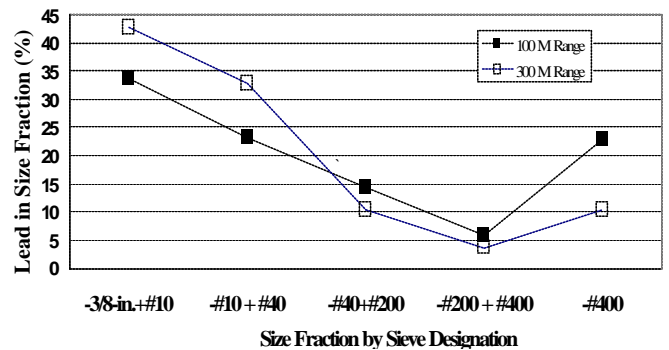

Fig. 8 – Percentage of Total Lead in Size Fraction for ATF Impact Berm Soil From 100-m and 300-m Ranges

Table 2. Analytical Data for ATF Soil Impact Berms 100-m and 300-m Ranges

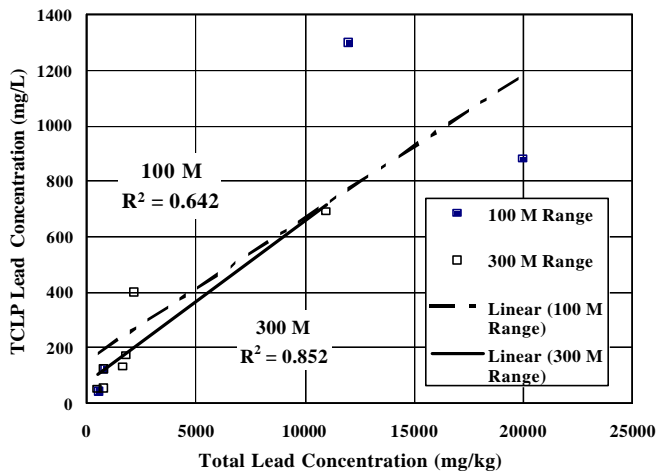| Range No.	Layer No.	Entire Sample		Total Pb Concentration by Particle Size Range (mg/kg)				
		Total Pb mg/kg	TCLP Pb mg/kg	-3/8-in. +#10	-#10 +#40	-#40 +#200	-#200 +#400	-#400
100-M	1 & 2	20,000	880	132,000	47,200	6,510	33,200	54,000
100-M	2 & 3	12,000	1300					
100-M	4	570	40					
100-M	5	840	120					
300-M	1	11,000	690	129,300	84,100	7,060	19,000	25,300
300-M	2	2,200	400					
300-M	2 & 4	1,700	130					
300-M	3	820	120					
300-M	4	1,800	170					
300-M	3,4,5	480	50					
300-M	5	780	54					

Fig. 9 – Total Lead Concentration versus TCLP Lead Extraction for ATF Impact Berm Soil at 100-m and 300-m Ranges

The TCLP data from the berm soils range from 1,300 mg/L to 40 mg/L. Linear correlations of the these data with corresponding total lead concentrations are shown in Fig. 9 which shows correlation coefficients (R^2) of 0.642 and 0.852 for the 100-m and 300-m range data, respectively. Given the limited number of tests performed and the potential for sample variation caused by the presence of larger sized metal fragments, the reliability of these correlations is deemed reasonable. In all cases, these results exceed the EPA TCLP regulatory limit for lead of 5-mg/L and the soil therefore would be classified as a hazardous waste.

Mineralogical Test Results

During the initial characterization phase of this work, the composition of both the soil and metal fragments was determined using XRD and SEM analyses. The results for each are discussed below.

XRD Results. X-ray diffraction data of the various particle size fractions of the material contained in the first two feet of the 100-m range are illustrated in Fig. 10.
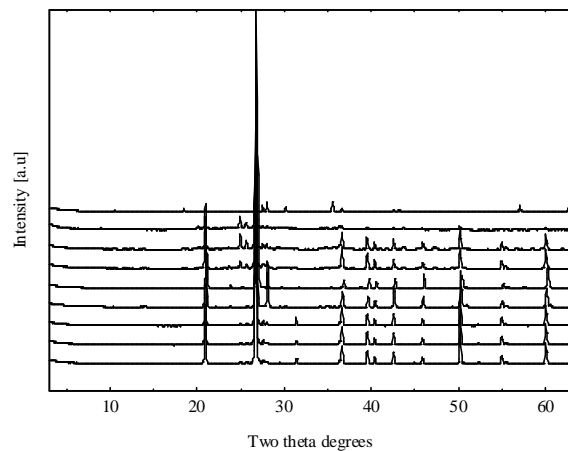

Fig. 10. XRD powder diffraction pattern for various size fractions of ATF soil; from bottom to top +4, -4+10, –10+40, -40+100, -100+200, -200+325, -325down, -325up, and Magnetic phases.

The mineral assemblage consisted primarily of granite origin quartz, feldspar, muscovite and typical granite accessory minerals (minerals in granites that undergo weathering at different rates and form weathering products consistent with their chemical composition). The soil also contains a small amount of magnetic material (1.5 wt %), which was later found to be a magnetite-hematite mixture. Quartz, one of the most stable minerals, is known for its high resistance to weathering. Mica minerals may also remain relatively unweathered in many granite residues. Feldspar, on the other hand, weathers quite rapidly forming clay minerals like kaolinite. Ferromagnesian minerals (most of the black minerals rich in iron and magnesium) also weather rapidly to form clays and iron oxides. However, because of the soil origin (dredged sand) most of the clay minerals that may have formed had been subsequently washed away. No significant organic fraction was identified.

Table 3. Rietveld-based mineralogical composition of the ATF soil fractions

Fractions	+4	-4+10	-10+40	-40+100	-100+200	-200+325	-325 down	-325 up	Magnetic
Fraction, wt%	0.94	2.86	16.48	36.69	22.27	5.29	14.62	0.69	0.16
Albite	0	0	0.1	0	2.7	3.4	0.7	2.5	0
Quartz	93.9	96.2	95.8	78.3	92.2	85.3	88	67.9	36.8
Cerussite	0	0	1.3	0	0.8	2.5	6.3	15.4	0.3
Anorthoclase	6.1	3.8	2.8	21.7	4.3	8.9	4.9	14.2	26.3
Hametite	0	0	0	0	0	0	0	0	1.5
Magnetite	0	0	0	0	0	0	0	0	35.2

Table 3 presents the results of the Rietveld-based mineralogical quantification of the various particle size fractions. The first row in this table shows the percent of total sample (wt %) represented by each fraction. The subsequent rows show the percent content (wt %) of each individual mineral identified within the individual fractions. Moreover, as shown in Fig. 11, comparison of the total lead quantities measured using the analytical (ICP-OES) method with the lead quantities computed based on the cerussite contents (determined by the Rietveld quantification method) correlated well for all of the finer fractions (-40 to -325 up). This is an indication that in the finer fractions most of the lead exists in the form of cerussite. Lead concentrations obtained by ICP-OES using independently prepared samples (shown as a "Sample C" in the figure) were also in good agreement. The curve of lead concentrations based on cerussite contents does not agree well with the analytical lead curves (Samples B and C) for the coarser fractions (-4 to +40). This is attributed to the fact that in the ATF soil coarser fractions Pb primarily exists in metallic fragment forms with only a limited amount of cerussite present.
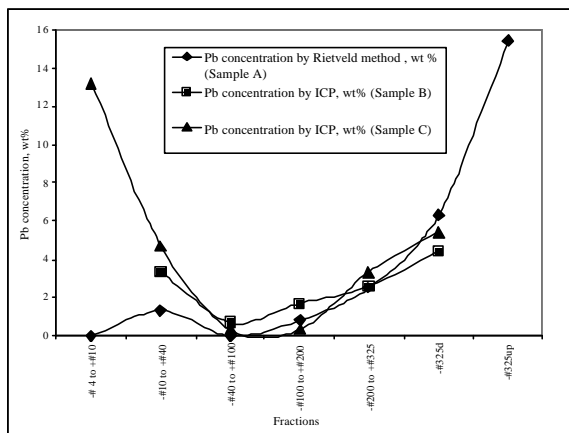

Fig. 11. Plot of total Pb concentration versus size-fractions. Pb concentrations are either calculated based on the cerussite content as determined by the Rietveld method (Sample A) or measured by ICP-OES (Samples B and C)

Scanning Electron Microscopy. In this phase of the study, several lead fragments were collected during analysis of the soil using the optical microscope and then examined using the SEM. Overall, SEM analyses revealed that the metallic lead fragments were mostly covered by a white surface layer of cerussite. Figures 12 and 13 show SEM pictures of selected Pb fragments and associated cerussite formations. The identification of the presence of cerussite had a profound impact on the results of the lead removal studies as will be subsequently discussed.
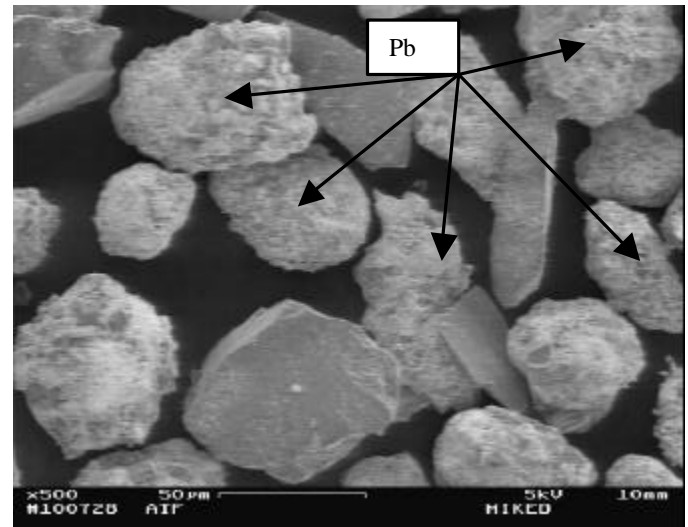

Fig. 12. ATF soil SEM picture of Pb fragments. White surface layer covering the Pb fragments is cerussite.
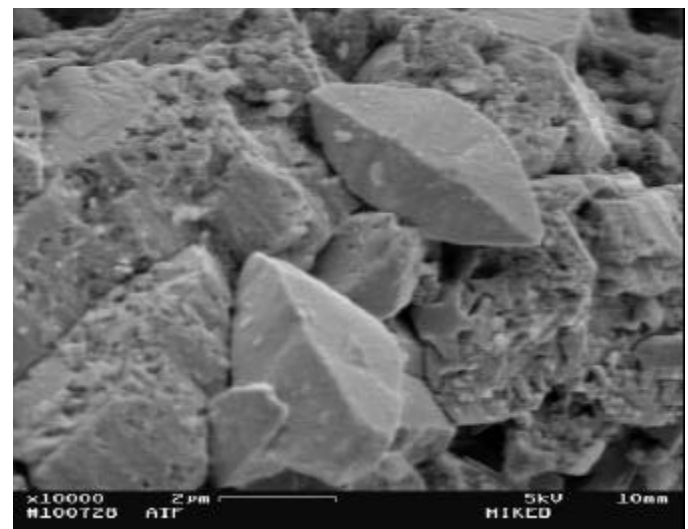

Fig. 13. ATF soil SEM picture close-up showing cerussite crystals forming on Pb fragment surfaces.

Physical Separation Test Results. The results of the physical separation testing associated with this study are presented in Tables 4 and 5 for the first and second pilot-scale evaluations, respectively.

Table 4. Post-Treatment Total Lead Concentration Using Five-Turn Spiral Concentrator

Middlings (2)	Heavies (3)	Lights (4)	Solids Loading (%)
1670	122400	8760	5% Solids Loading
2590	153100	4800	10% Solids Loading
7180	110600	4240	15% Solids Loading
9130	149900	6300	17.5% Solids Loading

Notes:
1) Initial lead concentration of -#10 +#200 feed material = 18,600 mg/kg
2) "Middlings" represent Treated Soil
3) "Heavies" represent waste metal stream
4) "Lights" represent predominantly -200 fine materials

In the first test series, performed using a 5-turn medium pitch spiral and a material feed consisting of the entire -#10 +#200 fraction of the composite sample, the total lead concentration of the treated soil (middlings) was reduced from an initial value of about 18,600 mg/kg to as low as 1670 mg/kg. The data also indicated that the effectiveness of the lead removal decreases with increasing solids concentration. While a mass balance was not performed during this initial assessment, visual observations of the "heavies" or concentrated metals stream indicated the quantity of sand reporting to this stream did not appear to be substantial.

Table 5. Post-Treatment Total Lead Concentration Using Seven-Turn Spiral Concentrator

Feed Size	Solids Concentration, %	Flow Rate, gpm	Post-Treatment Pb in mg/kg
-#10 + #50	7.8%	20	1175
		25	1078
		30	698
		35	584
		39.5	1885
-#50 +#200	5%	20	1121
		27.7	735
		30	533
		35	670
		40	1456
-#50 +#200	10%	20	925
		25	864
		30	877
		35	860
		40	858
-#50 +#200	20%	20	873
		25	827
		30	934
		35	812

Note:
-#10 +#50 Initial Pb Concentration = 25300 mg/kg
-#50 +#200 initial Pb Concentration = 10900 mg/kg

The results obtained in the second test series, performed using a seven-turn medium pitch spiral and two hydraulically separated particle size fractions for the system feed, are presented in Table 5. These results show that for the single test series conducted using the coarser size feed, approximating a -#10 +#50 particle size range, the total lead concentration was reduced from 25,300 mg/kg to as low as 584 mg/kg

with the unit operating at a solids concentration of 5% and a flow rate of approximately 35 gpm.

Using the same seven-turn device, a more detailed evaluation of the finer fraction approximating the -#50 to +#200 particle size range was performed. These data reveal that the total lead concentration was reduced from a pre-treatment level of approximately 10,900 mg/kg to as levels as low as 533 mg/kg. The data also indicate that lower lead levels are achieved at lower solids concentrations as was observed for the coarser feed. In addition, at solids concentrations of 7.8% for the coarser feed and 5% for the finer feed, there appears to be an optimum flow rate in the 30-gpm to 35-gpm range outside of which removal efficiency decreases at both lower and higher flow rates. This effect is not observed at the higher solids concentrations of 10% or 20% where the data indicate that removal efficiency appears to be relatively insensitive to flow rate.

The data show that the removal efficiency obtained using the seven-turn spiral with a greater particle size uniformity of the feed material exceeds the removal efficiency obtained using the five-turn unit and a wider particle size feed range. However, because both the test apparatus and feed conditions were changed concurrently it is not possible to identify the extent to which either or both were responsible for this improvement.

Since lead concentrations in the treated soil revealed that the removal goal had not been reliably attained, SEM and XRD analyses of these materials were made in order to identify the source of the elevated lead concentrations in the soil. These analyses revealed the presence of cerrussite. Since this compound has a density of only 6.6 g/cm^3 versus 11.3 g/cm^3 for elemental lead, it is possible that surface formations of this material could be slaking from the weathered lead particles during the treatment process (transport pumping and travel over the spiral concentrator) and being smaller and lighter than the elemental lead, may be reporting with the treated soil. Additional testing is planned to assess the effects of using an attrition scrubber to remove the looser cerrussite materials as part of the feed preparation step and thereby reduce the potential for release during treatment.

Chemical Extraction Test Results. The findings obtained in the bench-scale chemical extraction study are presented graphically in Fig. 14. This figure contains a summary of the quantity of lead removed per quantity of soil treated as a function of the molarities of both the nitric and acetic acids. The results indicate that at similar molarities, the nitric acid has a significantly greater capacity for solubilizing the particles of lead and/or lead species. Furthermore, the quantity of lead removed using the nitric acid approaches the total lead concentration level of approximately 50,000 mg/kg contained in the soil prepared for this experiment. It should be noted that this concentration is greater than the values obtained in both the initial characterization phase and mineralogical analyses presented above. It is believed that this difference as well as the scatter in the data shown are due in large part to experimental variation resulting from the inclusion of the -3/8-in. +#4 portion of the material in the limited-sized (10-g to 12-g) test specimens used in this study. Validation of these results will be addressed in subsequent analyses yet to be conducted. However, it is clear that acetic acid must be tested at higher molarities such that sufficient hydrogen ions are made available to

overcome the buffering capacity of the soil and bring about additional dissolution of the lead.
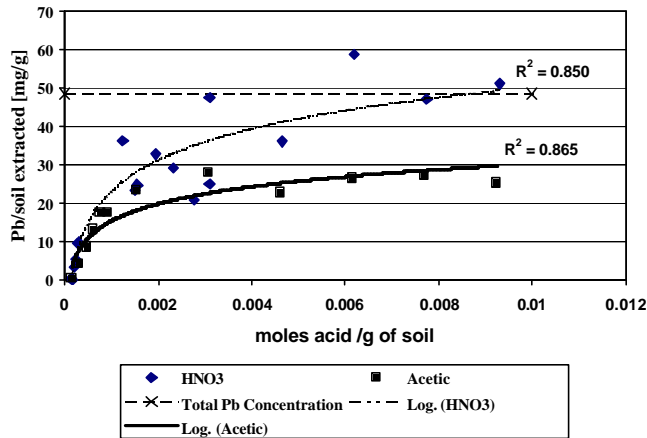

Fig. 14. Total Lead Extraction Results Using Nitric and Acetic Acids

CONCLUSIONS

The integration of geotechnical, analytical and mineralogical research tools in the site characterization study presented herein provided the data necessary to develop a comprehensive understanding of the environmental conditions that exist within the soil at the subject site as well as invaluable guidance in the selection of viable alternatives for remedying them. While important data were provided from each discipline individually, it was the manner in which the data sources complemented each other that was of greatest significance. The

quantification of the lead distribution throughout the soil as a function of particle size was determined using procedures founded in the geotechnical and analytical disciplines. However, the impact of this data on the overall project, specifically on the assessment of remediation options, was greatly enhanced by the understanding of the lead speciation provided by the mineralogical analyses. Continuation of this multi-disciplinary approach in the evaluation of potential remedies also provided insight as to possible explanations for the performance of the components and/or processes and readily identified alternatives that warranted investigation. While much work in the areas of physical separation and chemical extraction is yet to be performed in the pursuit of a cost-effective and practical alternative to landfill disposal for this and other firing range soils, the continuation of such an integrated multi-disciplinary approach will ensure that such efforts are effective and focused on attaining the desired goal.

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