SERDP FINAL REPORT
(Project PP/1057/78)

PROJECT TITLE: Elimination of Toxic Heavy Metals from Small Caliber Ammunition

SERDP PROJECT IDENTIFIER: 1057/78

RESEARCH CATEGORY: 6.2 Applied Research

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AGENCY: U.S. ARMY

LAB: Army Armament Research, Development, and Engineering Center - Picatinny

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THRUST AREA: Pollution Prevention

CRADA DEVELOPMENT: No

INTERNATIONAL: No

TOTAL ALLOCATED FUNDS: 500K

PROJECT OBJECTIVE:

The objective of this program was to obtain technical solutions for producing non-toxic small caliber ammunition, which would meet U.S. and NATO performance standards for all calibers (5.56mm, 7.62mm, 9mm, .50 caliber). This effort focused on eliminating toxic components in the projectile core, primer, and manufacturing processes. All proposed solutions must be economical and feasible while meeting all environmental regulatory guidelines and standards over the life cycle of the cartridge.

BACKGROUND:

Hazardous heavy metals are present in two major areas of current conventional small arms cartridges: the bullet core and the chemical compositions contained within the primer. The objective of this program is to investigate and develop technically feasible alternatives that have a lesser (preferably zero) environmental impact over the lifecycle of the product.

TECHNICAL APPROACH & RISKS:

Projectile core: The approach taken was to conduct environmental studies of candidate projectile core materials to ensure their viability for use in non-toxic projectiles, and provide methods by which the recovery of the material is optimized and release is minimized. Upon identification of a materials substitute for the lead cores currently being used, environmental testing was conducted.

The next generation of bullets and other projectiles for use in small arms is relying on new and different materials and combinations thereof to reproduce the density and properties of lead. Composite materials such as metal powders in nylon, or metal particulates with light metal binders, are currently being examined to produce "non-toxic" replacements for lead in small caliber bullets. The primary materials of choice for the high-density component in these composite simulants are tungsten and tungsten alloys. As with most materials, the data concerning the environmental and toxicological behavior of tungsten is incomplete. The project objectives are to complete the life cycle studies of tungsten to ensure its viability for use in non-toxic projectiles, and help provide guidance by which the release of the metal into the environment is minimized, and thus recovery is optimized.

Environmental stability, mobility, leaching and biological uptake studies were conducted from bullets fabricated from the new non-lead materials of tungsten-nylon (TRI-1) and tungsten-tin (ESP-1) composites, under various environmental conditions. Bullets were also 'fired' into different soils to characterize the basic impact behavior of lead and non-lead bullets, with particle sizes and material form noted.

The major areas of concern regarding the projectile core replacement are the terminal ballistic performance (lethality/penetration, addressed under a separate effort) and the environmental mobility/toxicity of the materials. The SERDP effort explored concerns with the mobility of the

alternate materials, the synergistic effect of these materials with other projectile components when exposed to various environmental conditions, toxicity of these materials, recyclability, and the degree of material uptake by indigenous flora.

Cartridge primer: This effort utilized a new class of non-toxic energetic materials called Metastable Intermolecular Composites (MIC) as a replacement for current primer materials, which typically include lead styphnate, lead azide, barium nitrate, and antimony sulfide. A MIC material is an engineered energetic consisting of two or more chemical species that are exothermically reactive with each other. The MIC fabrication technology relies on the use of Nano-particles (Nanotechnology), such that the reactant species are almost atomically intermixed to form a metastable reactive system that reacts many orders of magnitude faster than traditional mixtures of those reactants. Primer mixtures were developed and underwent ballistic testing to assess the performance of the MIC based primer as compared to conventional percussion primers.

There are three areas of concern with MIC with respect to current primer materials. First, the MIC compounds have never been used in small arms percussion primers. Unlike current primer compositions, the MIC materials produce heat, but not gas, upon ignition and this affects energy transfer into the main propellant charge. Second, the MIC compositions must be thoroughly characterized, such as by its output temperature, with means developed that would be relevant to a production environment. Third, the materials must eventually demonstrate extremely high reliability, such as when subjected to firing rates of 6,000 rounds/minute in the M134 Minigun, while concurrently meeting extreme environmental storage and operational conditions.

The primer work consisted of two discrete efforts. The first was the scaling up of the base material output, performed by Los Alamos National Lab (LANL) and ARDEC prototype reactors. The second was to utilize this output to fabricate, mix, assemble and test various primer chemistries for the applied product and to examine these processes themselves.

PROJECT BENEFIT:

This project will develop a non-toxic cartridge that will eliminate the environmental and hazardous effects that are associated with current ammunition. The need for costly range cleanups will be eliminated without sacrificing the proficiency and readiness of Armed Forces personnel. Specifically, it is anticipated that approximately $2.5 million required for waste removal at each outdoor firing range, as well as the $100 K annual cost for lead contamination monitoring, will be eliminated. Furthermore, there will be a facilities cost avoidance for over 600 indoor National Guard ranges which are currently closed due to high levels of lead, which will also improve military readiness. Lake City Army Ammunition Plant (LCAAP) yearly costs will also be reduced significantly by the incorporation of non-toxic materials in the projectile cores and primer mixtures. Over $100 K per year will be saved due to the elimination of lead sludge treatment and a reduction in fifteen operating personnel is estimated once the automated MIC primer and process is fully implemented. This will result in a $750K savings annually.

MANAGEMENT STRUCTURE:

This program has been a coordinated team effort between the Department of Defense (the U.S. Army Armaments, Research, Development and Engineering Center (ARDEC) at Picatinny Arsenal, the Naval Surface Warfare Center (NSWC) at Crane & Indian Head), the Department of Energy (DOE) National Laboratories at Oak Ridge and Los Alamos, Lake City Army Ammunition Plant (LCAAP) and its private industry operator, Alliant TechSystems (ATK). The team has been lead by ARDEC, who also Chairs the Joint Working Group for Non-Toxic Ammunition, which serves as a peer review committee with representatives from the US Army Infantry Center, Naval Surface Center, US Air Force, US Marines Corps, US Coast Guard, Army National Guard, US Army Industrial Operations Command, Materials Acquisition Command, US Army Environmental Center, and the Center for Health Promotion and Prevention Medicine.

The project's projectile R&D efforts have consisted of: ARDEC for general program management, product design & operational requirements; DOE for projectile core design and impact characterization, R&D of standardized test methods, short and long term environmental studies in various soil/rain compositions, and uptake studies in rye grass, beans and earthworms; LCAPP/ATK for legacy production process compatibility and cartridge loading; NSWC/Crane for cartridge testing; and Army Environmental Center & Materials Acquisition Command, as an advisory panel for outdoor ranges' requirements.

The primer R&D efforts have consisted of: ARDEC for general program management, product design, operational requirements analysis and primer testing; DOE, Indian Head and ARDEC for UFAL Reactor development, technology transfer and limited UFAL production, and material characterization test methods development and demonstration; Indian Head and ARDEC for MIC mix development, Primer loading and testing; and LCAAP/ATK for Primer metal parts.

SUMMARY:

- The environmental stability and mobility, and biological uptake of tungsten from bullets fabricated employing the new non-lead materials [tungsten-nylon (TRI-1) and tungsten-tin (ESP-1) composites] was examined.

- Bullets fired into different soils were characterized to provide a basic understanding of the behavior of lead and non-lead bullets upon impact. Particle sizes and form of the materials were noted and used to develop simulated fragmented bullets for test samples.

- Modified leaching procedures and aging experiments were used to evaluate the chemical stability and mobility of tungsten in bullets. Up-flow and hold techniques were examined to simulate different conditions, utilizing multiple types of soil and waters. Precision studies of the up-flow and hold techniques were conducted to examine technique reproducibility.

- Sand was a very porous media, allowing sub-micron particles to pass from the bed, through the filters. Concentrations of tungsten in leachant from experiments using sand were thus the

highest. Little or no tungsten was leached from columns using topsoil as the media.

- Special runs were conducted to evaluate interactions with other materials that could be present at shooting ranges (e.g. lead), with no interactions were observed.

- Long-term (one year) outdoor exposures and accelerated aging tests were employed to examine the stability of the new materials. It appeared that the tungsten powder oxidizes to form tungsten oxide, which is insoluble in water, and thus relatively stable in the environment.

- Biological uptake was studied employing earthworms. Earthworms were not adversely affected by exposure to soil contaminated with the tungsten-containing bullet materials. Uptake of tungsten by the earthworms in the short-term study was either minimal or zero. Tungsten was found in the worms from the three-month study, but as noted, the worms did not appear to be adversely affected. High concentrations of lead were found in worms exposed to lead-contaminated media in both the short and long term studies.

- Biological uptake was studied employing earthworms and rye grass. The tungsten-tin composite did not adversely affect the germination and growth of the rye grass. Rye grass growth, however, was inhibited for the sample contaminated with lead and the tungsten-nylon composite bullets. Tungsten was found in all plants grown in soil contaminated with tungsten. No correlation between tungsten concentration in the soil and in the plants was observed.

- Recovery and recycle experiments were conducted on the Tungsten based projectiles. Tungsten powder was easily recovered from the tungsten-tin composite by simply heating the material and melting the tin. The density differences produced rapid separation of the materials. Tungsten-nylon matrix recovery is recognized as a more challenging task.

- MIC primer mix collaborative efforts to demonstrate tech-transfer of UFAL reactors to ARDEC and Indian Head were successful. Batch scaling efforts have been partially successful (400% increase from 5g to 20g. Final goal is 1kg/hr).

- UFAL measurement technologies successfully designed and characterized material: BET gas absorption Isotherm, Highest Velocity, Transmission Electron Microscopy (TEM), & Thermal Gravimetric Analysis (TGA).

- The Long/Short timeframe chemical sensitivity of MIC to water was characterized under varying conditions, including temperatures. Results are very promising for the development and validation of a very cost-effective MIC decontamination procedure that will be valuable in a production environment.

- Primer evaluations have shown promising results in the viability of a ‘drop-in’ primer mix solution for current cartridge designs. Tailored test equipment has been developed for characterizing MIC material against the current baseline in a highly controlled environment. Conclusions are that the MIC primer mix appears to deliver near-nominal Peak Pressure

values and it currently has a slower response (Action Time) than the baseline primer.

- Taguchi studies on the Primer design have aided in identifying the interaction of the geometry of Metal Parts, gaining insight on the cartridge ignition process. Predicted results from the response tables were validated.

- High Temperature/High Humidity storage testing was conducted on unprotected MIC based primers. Results show that the MIC material does appear to have some moisture sensitivity, but not elevated temperature storage.

- Safety tests were performed on MIC samples. As expected, the material has a very high electrostatic sensitivity. It also had a high reactivity to friction; the exact values were below the measuring capabilities of both of these standard tests and could not be definitively quantified at this time.

- Scaling up of the batch sizes of MIC primer mix compositions was initiated, with a variety of mix compositions. The MIC primer composition is not considered to be finalized due to its Action Time performance, which currently meets Cartridge-level standards, but not component level performance standards. Nevertheless, up to 10g/batch has been demonstrated, which represents an order of magnitude improvement.

- The MIC primer performance optimization finished with the initiation of efforts to investigate a hybridized material to improve Action Time. The effort is showing progress through the use of gas-generating materials, to compliment the pure heat output of the MIC.

ACCOMPLISHMENTS:

I. Bullet Related Efforts:

The principal performer for this task was Department of Energy's Oak Ridge National Lab (ORNL). The investigation of the chemical stability and mobility of tungsten as contained in non-lead bullets continued throughout the program. The environmental stability and mobility of the powdered tungsten as part of fragmented bullets was examined employing combinations of leaching and aging (corrosion) experiments. Materials used in the non-lead bullets were exposed to simulated environments (soil, solvents, temperatures, etc.) to determine what compounds would form and their solubility and mobility were examined. Real and simulated bullet designs were used with various soil targets to provide a basic understanding of bullet behavior on impact, including particle sizes and form.

The next generation of bullets and other projectiles for use in small arms is relying on new and different materials and combinations thereof to reproduce the density and properties of lead. Composite materials such as metal powders in nylon, or metal particulates with light metal binders, are currently being examined to produce "non-toxic" replacements for lead in small caliber bullets. The primary materials of choice for the high-density component in these composite simulants are tungsten or tungsten alloys. As with most materials, the data concerning the environmental and toxicological behavior of tungsten is incomplete. The objectives of this project was to complete the life cycle studies of tungsten to ensure its viability for use in non-toxic projectiles, and help provide guidance by which the release of the metal is minimized, and thus recovery is optimized.

Background

The negative environmental safety and health aspects of lead are well documented, and are addressed by numerous federal laws and regulations. The use of tungsten-containing projectiles may offer simple remedies to the toxicological and environmental concerns associated with lead. Unfortunately, the information concerning the environmental and toxicological properties of tungsten and its compounds is incomplete. The life cycle of a projectile is not limited to fabrication, shooting, and possible recovery and recycling, but includes all aspects of the processing and use of the bullet. Design, manufacture, loading, storage and transport, firing, flight, impact, recovery, and recycling must be considered to fully examine the life of a bullet. Changes to one part of the cycle will influence all others in the chain.

Development of the information regarding environmental stability and mobility is needed to support recycling or "closed-loop" use of the materials. The morphology and chemistry of spent projectiles will be examined to begin the process of understanding the new materials when used in bullets. Materials in various forms from spent projectiles fired into different medias will be characterized. For materials that cannot be readily recovered, the morphology and chemistry will be used as a starting point for environmental and biological up-take studies. Information on the amount of residual material, and its chemistry and morphology is currently not available.

As the data concerning the form and chemistry of the materials and the nature of the applicable environments becomes available, leaching and accelerated aging tests will be conducted. The stability of the residual tungsten-containing materials in environments of concern must be determined prior to further studies including biological up-take. Leaching and other corrosion studies of the different materials combinations will be conducted (e.g. see Toxicity Characteristic Leaching Procedure (TCLP) test methods as described in EPA Publication SW-836). The enhanced environmental information will be used to determine risks, and establish a foundation for further development of bullets that utilize high-density metals other than lead.

The information regarding the form and composition of the species that remain in the environment is crucial for realistic predictions of mobility and interpretation of environmental models. Speculation with regard to this information is not advisable. The variability in reported data, and the lack of detail for the chemistry of the new tungsten-containing systems, makes utilization of the available information impossible. The biological uptake of tungsten from the environment is uncertain. This data is essential in understanding the long-term potential for biological up-take and mobility of tungsten in animals and plants throughout the food chain.

The results of the accelerated aging experiments will also provide guidance for optimizing the stability, and thus maximizing recovery and recyclability of this next generation of projectile materials. Projectile design, constituent materials, and processing will be used to minimize the generation and release of unrecoverable materials. Constituent materials may be modified to maximize recovery and minimize release. Encapsulation and modification of compositions are two possible options for optimizing the recyclability of the high-density component in the non-toxic bullets.

The final product is a more complete understanding of the environmental issues concerning tungsten-containing materials that are being considered for use in bullets and projectiles for small arms ammunition. The interest in tungsten is not limited to small arms but encompasses a large family of projectiles from small arms to missile warheads, and from training rounds to armor penetrators. The results of this project will be used to develop a comprehensive life cycle plan, with options for minimizing release and optimizing the recovery and recyclability of the constituent materials. The project will close the loop for the use of tungsten in bullets for small arms, prior to acceptance and wide spread distribution.

Fired Bullet Characterization:

The chemical stability and mobility of tungsten used as the “heavy” (high density) constituent in newly developed lead replacement materials are being examined. The tungsten-containing compositions under evaluation were down selected during an earlier study of seven different non-lead formulations. The current non-lead materials are homogeneous composites composed of tungsten powder in either a nylon or a tin matrix. The plan for this environmental stability effort was devised using the two materials and their most likely applications. Two military bullet designs were chosen to be examined, the 9mm, M882 Full Metal Jacket (FMJ) Ball cartridge (124 grain projectile), and the 5.56mm, M855 Ball cartridge (62 grain projectile, including steel penetrator tip), with emphasis on the latter. The lead replacement materials are under development thus adequate quantities of bullets for the study could not be readily

procured, so simulants for fired bullets were used in the environmental studies. The form and quantities of the materials used in the "fired bullet" simulants were determined through examination of real bullets shot into different soil types.

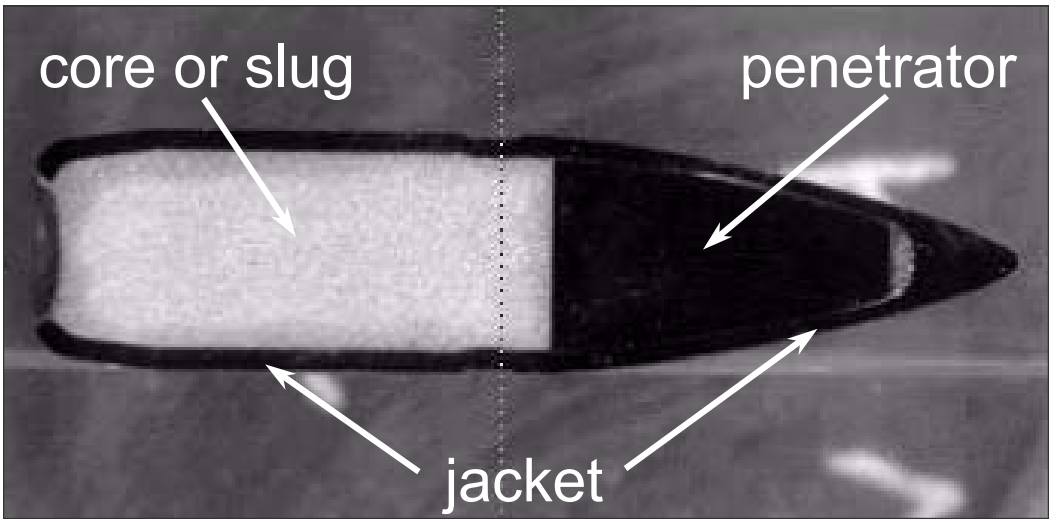

Figure 1: Cross-section of 5.56 mm M855 highlighting the three components: copper-alloy jacket, steel penetrator, and the high-density slug, which is currently lead.

M855 bullets fabricated employing the two lead replacements were fired into different soils at the Navy's facility in Crane, Indiana. Bullets were shot into sand, soil and mixtures containing gravel, and deflected off a steel plate into the same materials to mimic conditions found at various shooting ranges. Bullets were also fired into a shock absorbing concrete material developed by the Army. The fired bullets and fragments were removed from the medias and characterized. The size and shape of the recovered fragments were measured and the condition of the materials noted.

This information was used to design the simulants for use in the chemical stability and mobility experiments. It was found that the penetrators remained intact regardless of the media. The jackets peeled, expanded, and fragmented. The cores separated from the jackets and also fragmented. Analysis of the jacket fragments from the fired bullets showed that jacket trimmings (scrap materials from the fabrication process for the bullets) are very similar in size and shape to the fragments found in the sand and soil, and thus could be used in the simulant.

Figure 2: Similarity in fragmentation - M855 projectile samples recovered after firing into sand

The simulants were fabricated employing a combination of blending, pressing, and crushing of the bullet components. Scrap from the injection molding process for the tungsten-nylon composite was obtained and crushed to match the fragments found in the medias. The same was done for the tin-tungsten composite. In this manner, intimate contact between constituents' exposed surface area is achieved, and thus the simulant better represents the fired bullet. Details regarding the composition of the bullet simulants are given in Table 1, below:

Bullet	**Jacket (Cu alloy)**	**Core (composite)**	**Penetrator (steel)**
5.56 mm ball – rifle (per bullet weight)	32 wt % (20 grains)	52 wt % (32 grains)	16 wt % (10 grains)
9 mm ball – pistol (per bullet weight)	16 wt % (20 grains)	84 wt % (104 grains)	---

Table 1: Composition of the bullets selected for the study

Figure 3: Materials used to mix the simulant from fired bullets. Only the largest particles of the non-lead composite materials are shown.

Examination of Chemical Stability and Mobility:

Leaching studies

A review of the literature and established procedures for "leaching", such as Toxicity Characteristics Leaching Procedure (TCLP), revealed deficiencies with regard to evaluating the environmental stability and mobility of tungsten. Most procedures have been developed to determine what compounds or pollutants are present in the soil or other absorbent material. Digestion and leaching are involved, and in some cases acids and organic solvents are employed. These procedures are not adequate for the examination of the environmental stability and mobility of tungsten. The corrosion behavior of tungsten must be studied using specified quantities of materials mixed with various medias and exposed to different 'natural' solvents. The established procedures are not entirely applicable thus alternate methods were pursued.

A modified environmental exposure/leaching procedure was developed to examine both the leaching of the materials from various media (soil, sand, etc.) and the corrosion of the materials under simulated outdoor conditions. The "leaching" procedure and equipment were modeled after those described in ASTM D 4874-95, "Standard Test Method for Leaching Solid Material in a Column Apparatus." Polyethylene was substituted for glass or stainless steel for the column body and endplates for reasons of cost and robustness. All other aspects of the apparatus are consistent with the ASTM standard. The columns were fixtured in sets of six to best implement the experimental plan.

Figure 4: ASTM D 4874-95 derived leaching apparatus highlighting the gravity feed & peristaltic pump system for controlling flow.

The ASTM standard for leaching describes the up-flow method in which the fluid or solvent is introduced through the bottom of the column and forced up through a packed bed out the top of the apparatus. Although this is the "standard," other techniques have been used to study the corrosion and leaching behavior of materials in packed beds. It is recognized that different conditions will exist at various locations, and no single test can simulate all cases. However, the potential conditions to which fired bullets are exposed can be determined by reviewing past, present, and suggested shooting range designs. Two different leaching techniques were selected for use in this study.

Up-Flow - Leachant is forced into the bottom of a column and through the media at a constant rate (typically 1 void volume per day). The leachant is forced out of the top of the column and collected for analysis. This method is described in the ASTM standard.

Hold – Leachant is forced into the bottom of the column as in the Up-Flow technique but when the bed is saturated, the column is sealed and allowed to stand for a given period. After a specified time segment, the leachant is drained and sampled. The process is then repeated.

Each of the techniques can be operated, as long as is necessary to gather desired information. A total time of 30 – 35 days (approximately one month) was chosen for this study. The leaching units were operated at ambient indoor conditions, typically 72°F. As mentioned, the columns were assembled in sets of six to accommodate the experimental plan. Six leaching experiments using a given soil/leachant combination were conducted in parallel. This ensured consistency and simplified operation. The six columns were loaded as follows and summarized in Table 2, below: (1) media, no simulant, (2) media with lead, copper alloy, and steel, (3) media with tungsten-tin composite, copper alloy, and steel, (4) media with tungsten-nylon composite, copper

alloy, and steel, (5) media with tin-tungsten composite and copper alloy, and (6) media with tungsten-nylon composite and copper alloy. The simulants added to the columns represented: (1) control, no bullets, (2) lead M855, (3) W-nylon 9 mm, (4) W-nylon M855, (5) W-Sn 9 mm, and (6) W-Sn M855.

Column Number	Simulant **Material**	Bullet **Type**
1	Empty (Control)	None
2	Lead, Jacket Alloy, Steel	Lead M855
3	W-Sn, Jacket Alloy, Steel	ESP-1 M855
4	W-nylon, Jacket Alloy, Steel	TRI-1 M855
5	W-Sn, Jacket Alloy	ESP-1 M882
6	W-nylon, Jacket Alloy	TRI-1 M882

Table 2: Leaching Columns and Bullet Types

The soils selected for the studies included sand and topsoil. Initial leaching experiments were conducted using sand as a media. Sand is frequently used as a berm material at many shooting ranges and is relatively inert, thus was a useful starting material. It packs and percolates well, and provides an excellent media for examining chemical interaction between the leachants and bullet materials.

Soils that may be found at different shooting ranges were also explored and collected. The soils were selected based upon chemistry and permeability. A Soil Survey of Anderson County, Tennessee, was used to locate different soil types in the local area. Using the soil maps, a soil commonly used for berms in this region, loamy clay, was located and collected. The soils were dried and characterized in the laboratory. Unfortunately, but not unexpectedly, the loamy clay exhibited excessively low water permeability that limited (stopped) leachant flow. This type of soil was deemed unacceptable for use in the columns. It was evident that although loamy clay would be suitable for berms due to its resistance to erosion and water permeation, it was not an appropriate choice for the study. Water does not flow though the clay and thus bullets trapped in this media would not be exposed to conditions simulated by the columns. Therefore, other soil types were located, obtained and characterized. Topsoil from the local region was selected as an appropriate media for leaching studies. It too exhibits acceptable percolation and packing behavior. Limestone gravel and ground tires are also of interest to the user and thus may be used in the future.

Three leachants were chosen for the chemical stability and mobility studies: deionized water, simulated rain (acidic), and salt water. The deionized water provided a neutral solvent and thus accentuated the interaction between the soil and the bullet materials. The ocean water was produced from a mix specifically designed for saltwater fish tanks. This leachant was used to simulate conditions expected at coastal facilities. The composition of the acid rain was determined from information reported by the National Atmospheric Deposition Program for the Walker Branch Monitoring Station (TN00) located on the Oak Ridge Reservation for the period January 3, 1995 through January 2, 1996. Information concerning rainwater including amount, pH and composition, i.e. metals and salt concentrations have been collected by the NADP and is

available for use. The TN00 monitoring station is located on the Oak Ridge Reservation and thus the rainwater collected represents that which would be encountered at the on-site shooting ranges. The rain is relatively acidic with an average pH of 4.4. The local rainwater was simulated employing mixtures of de-ionized water, salts and acid solutions. Composition and pH for each batch were measured and adjusted to ensure reproducibility and consistency between experiments. It is important to note that rainwater compositions representative of most any region of the country can be duplicated if necessary.

Effluent or leachant flow for the up-flow experiments was fixed at 1 void volume per day, or a daily volume sufficient to completely fill the porosity in the media packed into each column. The columns were designed to produce a void volume of approximately 1-l for the selected soil types. The same volume was needed to fill the column for the hold method. For the up-flow technique, the leachate was collected and sampled after 1, 2, 4, 8, 16, and 32 days. The columns were drained after 5, 10, and 20 day for the hold technique for a total of 35 days exposure. Analysis of the leachates was conducted by R. J. Lee Group of Monroeville, PA, which is EPA certified for the analysis of fluids and other materials.

Literature sources suggested that each column contain between 1% and 5% by weight of the bullet materials to simulate the amount found in the berms at most ranges. However, it was realized that controlling the weight fraction of contaminant could introduce additional variables when using different soil types. In other words, the amount of the bullet material in the column would be different for each media. In order to control the quantity of tungsten in the columns, and maximize reproducibility, a fixed number of bullets per column were used instead of a weight percentage of media. The chosen numbers of bullets per column were 10 for the M855 and 5 for the 9mm ball. The total weight of bullet material used in each column was fixed at ~ 40 grams (620 grains).

The solid media, e.g. sand, was added to each column in three even portions (i.e. top, middle, and bottom). The column was shaken during filling to settle and compact the mixture. The bullet simulant was added to a portion of the media equal to one-third the total weight. The bullet fragments and media were blended in a separate container before addition to the column to ensure uniform mixing and distribution. The bullet-media mixture was placed in the middle portion of the column.

Fluid from the columns was collected and analyzed for metals or other constituents, and bullet materials were recovered after exposure for further examination. Samples of the soil from different sections of the columns were gathered to analyze chemical interactions and movement of materials.

Standard Runs:

- Experiments that simulate the exposure of the new bullet materials to wet soils and different leachants such as salt water and acid rain are being conducted. These are the standard experiments with bullet fragments buried in soil and then exposed to the leachants. The details are listed in Table 4 with the results summarized in later sections.

Special Runs:

- A number of experiments to examine materials interactions or accelerated aging have been and will be conducted. The leaching experiments involve either special preparation of the materials or combinations of materials. The "special" leaching runs are summarized in the second section of Table 4. Short descriptions of some of these experiments are given in the following sections.

Legacy Lead Interaction:

The new tungsten-containing bullets may be fired on legacy ranges that still have lead contamination. This has caused concern with regard to the stability of lead when mixed with tungsten-containing materials. It was anticipated that the new bullets will not cause lead to leach from the ranges rates greater than currently being measured. A study using mixtures of lead and non-lead bullets was completed. The equivalent of 10 each of lead and non-lead bullets, either tungsten-nylon or tin-tungsten, were mixed with media and leaching studies conducted using rain water and the hold technique. Both sand and soil were used and the materials were exposed for a total of 35 days. Columns containing only media, and single bullet materials were also examined for comparison. A summary of the results is given a later section. The lead concentrations measured for the sand and soil columns did not exceed 0.05 and 0.1 ppm, respectively. For both sand and soil, the control, the column containing no bullets, only media showed the highest lead levels. It is concluded that mixing the new materials with lead will not affect leaching on existing ranges.

Precision Study:

As noted, the chemical mobility and stability of the new bullet materials were evaluated employing a combination of corrosion (aging) and leaching. Due to the significant variations in materials, soil types, and solvents, only one repetition has been conducted for each chosen mixture of soil, bullet type, and solvent. To ensure the validity of the approach and results, a series of leaching experiments have been conducted to examine reproducibility and statistics of the procedure. Three columns each of tungsten-nylon and tin-tungsten M855 simulant were tested using soil and sand as medias, and acid rain as the solvent. Both the up-flow and hold methods were evaluated. The leaching runs have been completed and samples submitted for chemical analysis. The details are listed in Table 4 and indicate acceptable experimental test repeatability.

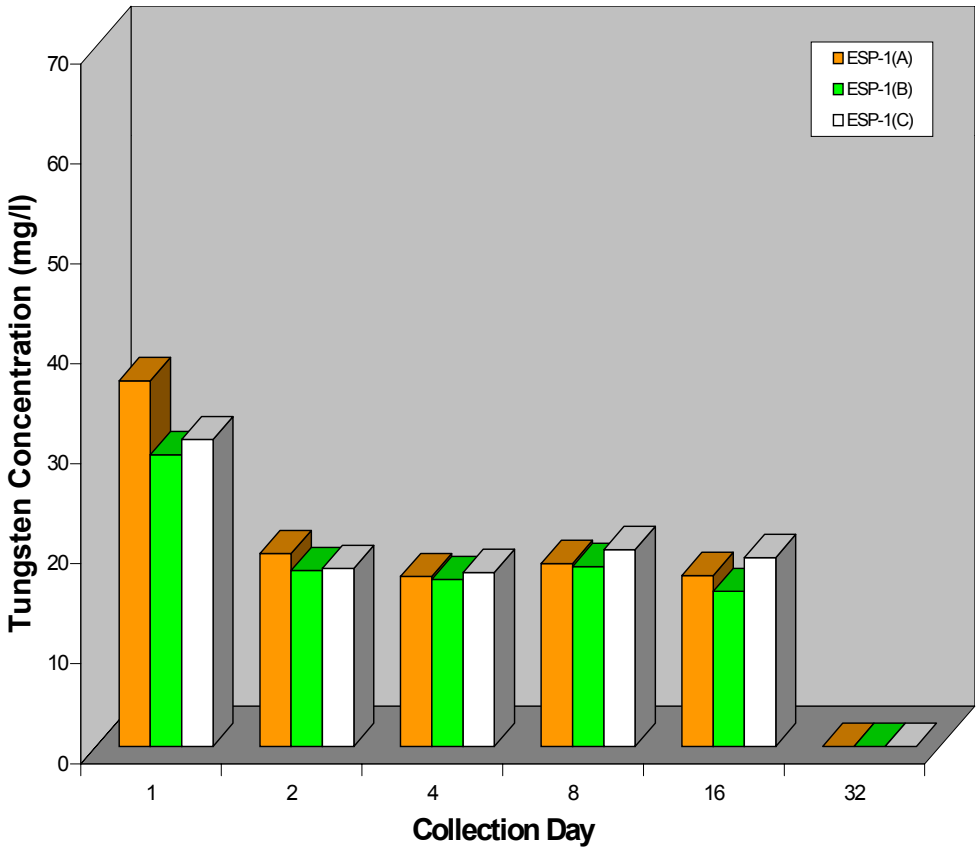

Table 3: Precision Study repeatability evaluation.

Accelerated Aging:

A study in which tungsten oxide and tungstic acid in different ratios were substituted for the tungsten powder in the tungsten-tin bullet material was conducted to simulate the longer term corrosion of tungsten in a moist environment. Mixtures of tungsten, tungsten oxide, and tungstic acid were blended with tin to simulate the corrosion products for the W-Sn bullets. Although this method provided an indication of trends that could be experienced, it may not fully represent the products that will form during environmental corrosion, as bullets fired at shooting ranges will be buried in soil and will experience a variety of environmental conditions. Additional aging studies of the materials in various soils will be necessary to ensure a complete understanding of the chemical stability and mobility of tungsten as found in the new bullet materials.

The corrosion of simulated, as-fired non-lead M855 bullets was accelerated using an environmental chamber. The behavior of both the nylon-tungsten and tin-tungsten materials in an elevated-temperature, water-saturated environment was examined. Mixtures of non-lead core fragments, jacket scrap, and penetrators, in proportions equal to those as used in the M855 bullet, were blended with 500 grams of sand and placed in Pyrex dishes. The equivalent of approximately 62 bullets

(250 grams) was added to each dish. Deionized water (~ 250 ml) was added to the dishes until the mixtures were thoroughly soaked. The dishes were then placed in the chamber that was preheated to 130°F. A dish with deionized water was placed in the oven to ensure full saturation of the environment with water vapor, and the water level in the dish was maintained throughout the test. Water was added to the mixtures as needed and the mixtures were stirred every three to four days. The sand-bullet mixtures were kept in the oven for 48 days. After removing from the chamber, the mixtures were air dried, and placed in leaching columns. The remaining volume of the columns was filled with sand per standard procedures and guidelines. Simulated acid rain was used as the solvent, and samples were taken at the usual intervals.

Environmental Aging: Outdoor Exposure

Although laboratory corrosion tests provided valuable insight into materials interactions and the processes associated with the degradation of the new bullet materials under controlled conditions, outdoor exposure is the optimal method by which chemical stability and mobility in the environment can be examined. Outdoor exposure studies of simulated M855 bullets using lead, tungsten-nylon, and tin-tungsten were conducted. Ten each of the bullets have been mixed with sand or soil in amounts necessary to fill a leaching column. Control samples with no bullets have also been prepared for each time step. The mixtures were placed outdoors with full exposure to the elements. Samples were removed from the study at 1, 3, 6, 9, and 12 month intervals and tested using the hold leaching method.

The leachate, or solvent extracted from the columns, is collected and sent to RJ Lee Group Inc. of Monroeville, PA for analysis. The samples are analyzed for six metals: tungsten, copper, iron, zinc, tin, and lead using an inductively coupled plasma technique. A summary of the chemical stability and mobility experiments is presented in Table 4 (following page). The results for the listed runs compiled and presented in the *Appendix*, in Table sections A-1 through O-4.

Standard Runs	Solvent	Media	Technique	Status
A	**De-ionized Water**	**Sand**	**Up-flow**	**Complete**
A	**De-ionized Water**	**Sand**	**Hold**	**Complete**
B	**De-ionized Water**	**Soil**	**Up-flow**	**Complete**
B	**De-ionized Water**	**Soil**	**Hold**	**Complete**
C	**Ocean Water**	**Sand**	**Up-flow**	**Complete**
C	**Ocean Water**	**Sand**	**Hold**	**Complete**
D	**Rain Water**	**Sand**	**Up-flow**	**Complete**
D	**Rain Water**	**Sand**	**Hold**	**Complete**
E	**Rain Water**	**Soil**	**Up-flow**	**Complete**
E	**Rain Water**	**Soil**	**Hold**	**Complete**

Specialty Runs	Solvent	Media	Technique	Status
*	Rain Water	Chipped Tires	Hold	Cancelled
F	**Rain Water**	**Soil + Limestone**	**Hold**	**Terminated**
*	Rain Water	Soil + LEADX™	Hold	Cancelled
*	Rain Water	Tracer Rounds in Sand	Hold	Cancelled
G	**Rain Water**	**Sand (mixed mat'ls)**	**Hold**	**Complete**
G	**Rain Water**	**Soil (mixed mat'ls)**	**Hold**	**Complete**
H	**Rain Water**	**Sand (precision test)**	**Up-flow**	**Complete**
H	**Rain Water**	**Sand (precision test)**	**Hold**	**Complete**
I	**Rain Water**	**Soil (precision test)**	**Up-flow**	Cancelled
I	**Rain Water**	**Soil (precision test)**	**Hold**	Cancelled
J	**Rain Water**	**Bullets Fired into Sand**	**UP-flow**	**Complete**
J	**Rain Water**	**Bullets Fired into Sand**	**Hold**	**Complete**
K	**Rain Water**	**Bullets Fired into Soil**	**Up-flow**	**Complete**
K	**Rain Water**	**Bullets Fired into Soil**	**Hold**	**Complete**

Aging Studies	Solvent	Media	Technique	Status
L	**Rain Water**	**Aged Material in Sand**	**Up-flow**	**Complete**
M	**De-ionized Water**	**Sim. Aged Mat'l in Sand**	**Up-flow**	**Complete**
N	**Rain Water**	**Sand (1 month)**	**Hold**	**Complete**
N	**Rain Water**	**Sand (3 months)**	**Hold**	**Complete**
N	**Rain Water**	**Sand (6 months)**	**Hold**	**Complete**
N	**Rain Water**	**Sand (9 months)**	**Hold**	**Complete**
N	**Rain Water**	**Sand (12 months)**	**Hold**	**Complete**
O	**Rain Water**	**Soil (1 month)**	**Hold**	**Complete**
O	**Rain Water**	**Soil (3 months)**	**Hold**	**Complete**
O	**Rain Water**	**Soil (6 months)**	**Hold**	**Complete**
O	**Rain Water**	**Soil (9 months)**	**Hold**	**Complete**
O	**Rain Water**	**Soil (12 months)**	**Hold**	**Complete**

Table 4: Summary of Leaching Experiments
Detailed tables for each are listed in the *Appendix*

Biological Uptake

Earthworms – Short-term

B. Konetsky and A. Stewart of ORNL's Environmental Sciences Division conducted earthworm toxicity studies involving the tungsten-containing replacements for lead in bullets. The method employed in the study was developed to assess the sublethal effects of contaminants in soils on earthworms.[1] Earthworms, Eisenia foetida, were introduced to soil samples containing material mixtures similar to those employed in the leaching studies. The earthworms were kept in the environments for 20 days and growth analyzed.

Worms in each sample had negative growth, i.e. lost weight, including those in the control sample; the soil without metals (Table 5). However, worms in the lead-containing samples lost significantly more weight than the other specimens. The ranking, in order from best to worst for weight loss, was ESP - 9 mm, TRI - 9 mm, control, ESP M855, TRI M855, and lead M855. The three tests simulating the M855 bullet exhibited more weight loss than the control and those using mixtures mimicking a 9-mm configuration. It is possible that the iron from the penetrator affected worm growth, but this has not been fully investigated.

Contaminant	Average Growth (mg)
ESP – 9 mm	-0.007095
TRI – 9 mm	-0.010025
Control – none	-0.010113
ESP – M855	-0.010804
TRI – M855	-0.016464
Lead – M855	-0.030413

Table 5: Comparison of Earthworm Growth

Earthworms are a valuable part of the food chain; thus metal concentrations in the remains of the earthworms were measured to examine biological up-take. The dried worms were sent for analysis and the results are listed in Table 6, below. High levels of lead were found in the worms exposed to lead-contaminated soil. No tungsten or tin was found in the worms, and zinc and copper levels were essentially the same for all specimens. There were notable differences in iron concentrations; however, as noted before, correlation between contaminant and iron content have not been examined.

Contaminant	Lead (ppm)	Tungsten (ppm)	Tin (ppm)	Zinc (ppm)	Iron (ppm)	Copper (ppm)
ESP – 9 mm	0	0	0	145	1240	17
TRI – 9 mm	0	0	0	112	4310	14
Control – none	0	0	0	139	3500	14
ESP – M855	0	0	0	123	2180	17
TRI – M855	0	0	0	181	6980	23
Lead – M855	752	0	0	122	180	11

Table 6: Chemical Analysis of Earthworms

Earthworms – Long-term

In collaboration with a student from a local institution, an additional experiment to examine the effects of the non-lead compounds on earthworms has been completed. The earlier study involved ten repetitions of two worms in each soil-contaminant mixture. In this latest experiment, colonies of worms were exposed to contaminated soil for approximately three months. Growth and the biological uptake of the various metals were examined.

One pound (455 grams) of earthworms, *Eisenia foetida*, was placed in each of four "composting bins" with mixtures of soil, newspaper, and organic matter. The worms were given a period of two weeks to acclimate to the environment. Once the worms had adapted to the environment, 150 grams of bullet core material (50 grams of coarse and 100 grams of fine particles) were added to the soil/compost mixture. Lead and the tungsten-tin and tungsten-zinc composites were used in this study, with one colony left uncontaminated as a control. Copper jacket scrap, steel penetrators, and the tungsten-nylon materials were not included in this experiment. The worms were fed organic matter when needed, about once a week. Care was taken to minimize the amount of available food as to force the earthworms to continually scavenge the soil in search of nutrients, thus maximizing exposure to the metals.

During feeding, quantities of worms in the container contaminated with the mixture of zinc and tungsten were observed on the walls of the bin instead of in the soil-compost mixture. In the bin contaminated with lead, the worms could not be easily found and appeared to move more slowly than the worms in the other bins. The worms in the control and the bin with tin and tungsten were extremely active. The pH of the soil was checked periodically using indicator strips and appeared to remain unchanged throughout the experiment.

The worms were separated from the soil 80 days after the metals were added. As expected, the worm populations, as measured by total weight, for all of the colonies decreased during the study (Table 7). Very few worms were found in the bin with lead contaminated soil. The quantity of worms in the control and the soil with tungsten-zinc were about the same, however, the most worms were found in the sample contaminated with the tungsten-tin composite. The size and number of worms from the colonies was also examined. Measuring the length and diameter of live worms was impossible; thus size was determined using the number of worms per unit weight. In this manner, a high value indicates smaller worms; a small number denotes larger worms. Results are shown in Table 8.

Metal Contaminant	Weight of Worms In (grams)	Weight of Worms Out (grams)	Worm Weight loss (grams)	Worm Weight Ratio (to control)
Tin plus tungsten	455	174.7	-280.3	1.61
Zinc plus tungsten	455	123.3	-331.7	1.14
Control	455	108.4	-346.6	1.00
Lead	455	59.3	-395.7	0.55

Table 7: Growth Behavior as Determined from Weight of Worms Removed

Metal Contaminant	Number per 15 g	Number per gram	Number per 10 g	Number per gram
Control	83	5.53	51	5.10
Tin plus tungsten	90	6.00	59	5.90
Zinc plus tungsten	97	6.47	63	6.30
Lead	104	6.93	73	7.30

Table 8: Size as Determined from Number per Unit Weight of Worms
(Note: a smaller numerical value = larger individual worms)

Dried worms were analyzed for metal uptake with the results summarized in Table 8. High concentrations of lead were found in the worms from the lead-contaminated soil. Small amounts of tungsten were found in the worms from the containers contaminated with the tungsten-tin and tungsten-zinc compounds, with higher concentration for those from the tungsten-zinc bed. Elevated levels of tin and zinc were noted in the worms from the bins contaminated within the materials containing these metals. Concentrations of iron and copper in all of the worms appeared to be constant. Although some tungsten was found in the worms from the bins contaminated with materials containing this metal, the levels were low, and did not seem to adversely affect the worms (Tables 8 and 9).

Metal Contaminant	Lead (ppm)	Tungsten (ppm)	Tin (ppm)	Zinc (ppm)	Iron (ppm)	Copper (ppm)
Control	95	10	1	111	263	10
Lead	2760	16	73	127	332	11
Tin plus tungsten	79	213	285	117	289	11
Zinc plus tungsten	43	457	27	1240	248	16

Table 9. Chemical Analysis of the Worms

There were notable differences in the texture of the soils from the different bins. The soil from the bin with tungsten-tin had a fine particle size and was uniform in color and particle size distribution. There was no evidence of residual newspaper or organic matter. The same was found for the control bin. The soil from the bins with lead and tungsten-zinc was coarse and had quantities of newspaper and organic matter. The pH of the soil from each bin was also measured and the results are listed in Table 10. The reasons for the differences in the pH of the soils are not known, however, additional analysis will be conducted to examine variations in the chemical content of the soils.

Metal Contaminant	pH
Lead	6.25
Tin plus tungsten	4.29
Zinc plus tungsten	6.10
Control	4.28

Table 10: pH of the Soils from the Worm Study

Biological Uptake: Bean Plants

In collaboration with a local institution, a preliminary plant uptake study was conducted. Tungsten and tungsten oxide powders were blended with soil, and bean plants were germinated and grown in the contaminated soils. Soil mixtures containing 1, 5, and 10-weight % tungsten or tungsten oxide powders were used. A control sample containing no metal or oxide was also included. Germination rate, plant growth as measured by height, and tungsten concentration in the plants are being examined. This initial effort will provide insight into the procedures and analytical needs for future plant uptake studies.

The entire bean plants, including stems and leaves but not roots, were dried, charred, and dissolved in an acid mixture for analysis. Tungsten content was measured using an inductively coupled plasma technique. Three samples of each group of plants grown in soil with a given concentration of tungsten or oxide powder were analyzed. The tungsten concentrations found in the plants are summarized in Table 11. The linear relationship between the concentration of tungsten in the plant and soil shows the bean plants are "indicators" for tungsten. These are preliminary results and do not indicate a problem or concern. Tungsten does not appear to be detrimental to plants, and is listed as essential for life processes.

Concentration of Powder in Soil (wt %)	Concentration in Plant for W powder in Soil (mg/kg)	Concentration in Plant for WO_3 powder in Soil (mg/kg)
Control	170	170
1	620	340
5	920	660
10	1100	820

Table 11: Concentrations of tungsten in bean plants

The uptake of tungsten from the tungsten powder was higher than for the oxide. This was expected for the oxide is essentially insoluble in water and acids thus would be less mobile. Although these initial tests showed that tungsten can be absorbed by plants, tungsten and tungsten oxide powder were used, not a simulant containing the other materials such as nylon, tin, steel, and copper. Future uptake studies will utilize materials as determined from the leaching (environmental corrosion) tests, and would include much greater detail concerning growth, uptake, and suggested consequences of said.

Biological Uptake: Rye Grass

Bean plants are not commonly found on shooting ranges thus other plants to be used in the investigation of the biological uptake of tungsten have been explored. It was determined that rye grasses are typically planted on ranges to control erosion. The effects of different metals; copper, nickel, manganese, lead, cadmium, zinc, aluminum, mercury, chromium, and iron, on germination and root growth for rye grass, *lolium perenne*, have been examined[1]. The study did not include analysis of metal uptake or the terminal harvest information such as plant or root mass.

[1] M. H. Wong and A. D. Bradshaw, "A Comparison of the Toxicity of Heavy Metals, Using Root Elongation of Rye Grass, Lolium Perenne", New Phytol. 91, 255-162 (1982).

Perennial rye grass was therefore selected for the additional biological uptake studies. Rye grasses are some of the most widely grown grasses in the world. They establish quickly, have long growing seasons, and are high yielding under favorable conditions. Perennial rye grasses are bunch-type grasses and will generally survive several seasons in harsh conditions. These grasses will grow on soils with a wide range of pH, but prefer a pH range of 5 to 8.

Samples were added to topsoil and rye grass grown in the contaminated soil, along with a Control. The grasses were allowed to grow to maturity, approximately 12 weeks. Germination and harvest information such as plant and root mass were investigated to determine the effects of the non-lead bullets on rye grass growth behavior, with root and plant samples analyzed for metal content. Sample trays were 200 square inches in area.

For harvesting, the individual rye grass trays were divided into ten (10) sectors, and plants were harvested from each sector. Three (3) sectors were chosen for also harvesting plant roots (Figure 5). Growth did not occur in all sectors of every tray, but growth did occur in all trays.

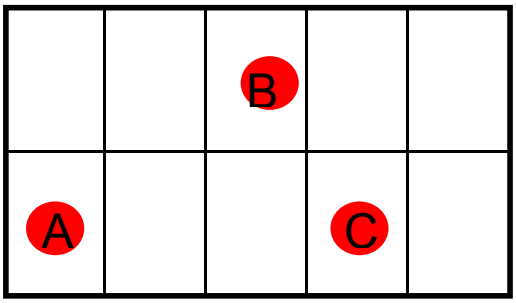

Figure 5: rye grass sample trays (Total area = 200 in^2)
Plants AND roots harvested from A, B & C (from balance, only plants)

Growth appeared to be consistently higher in the Control and non-lead samples than in areas with leaded projectiles (Figures 6, 7). Additional analysis of the results is pending.

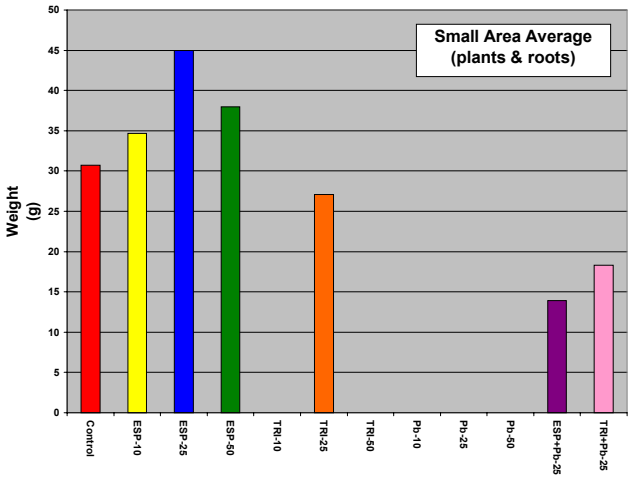

Figure 6: rye grass weights – plants & roots

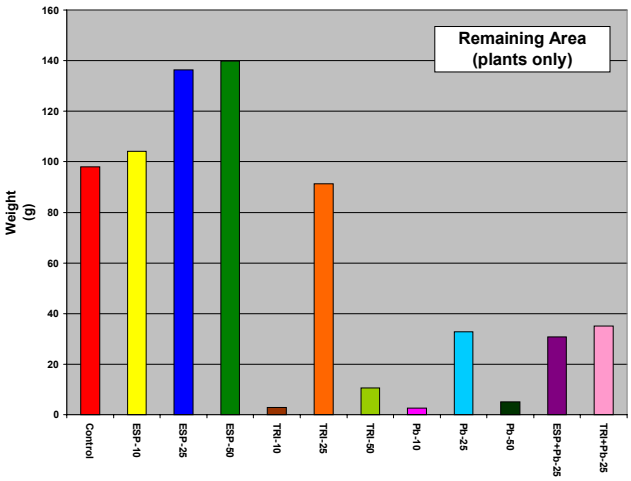

Figure 7: rye grass weights- plants only

II Primer Related Efforts:

This effort is utilizing a new class of non-toxic energetic materials called Metastable Intermolecular Composites (MICs) as a replacement for current primer materials which include lead styphnate, lead azide, barium nitrate, and antimony sulfide. Primer mixtures were evaluated using ballistic testing to assess the performance of the MIC based primer as compared to conventional percussion primers. The primary thrusts were the continuing development of prototype Ultrafine Aluminum (UFAL) reactor facilities and the use of the MIC output to fabricate and evaluate MIC-based primers.

UFAL Reactors:

One effort was to concurrently scale-up the batch size and to support the related Technology Transfer effort to establish reactors at ARDEC and Indian Head. The latter part of this effort has been successful with the demonstration of product production at both remote sights that has been verified through primer testing. UFAL reactors have experienced downtime due to operational difficulties, such as power supply cooling system corrosion, but as of the time of this writing, there are three operational reactors. The batch size scale-up portion of this effort is and will continue to be ongoing and includes collaboration and data sharing. To date, it has had moderate successes: techniques such as enlarged crucibles and semi-continuous methods such as the introduction of wire feeders has resulted in increased batches from 5g to 20g. The interim goal for this related work is 200g, with a final full-scale production objective of 1kg/hour.

The other primary effort was the design, characterization and demonstration of several product characterization and measurement processes, which was also a collaborative effort. The Aluminum's particle size/diameter was successfully quantified by a BET gas absorption process and by Transmission Electron Microscopy (TEM). The Aluminum's passivation layer thickness was successfully quantified through Thermal Gravimetric Analysis (TGA) and through a Highest Velocity (optimum stoichiometry) processes.

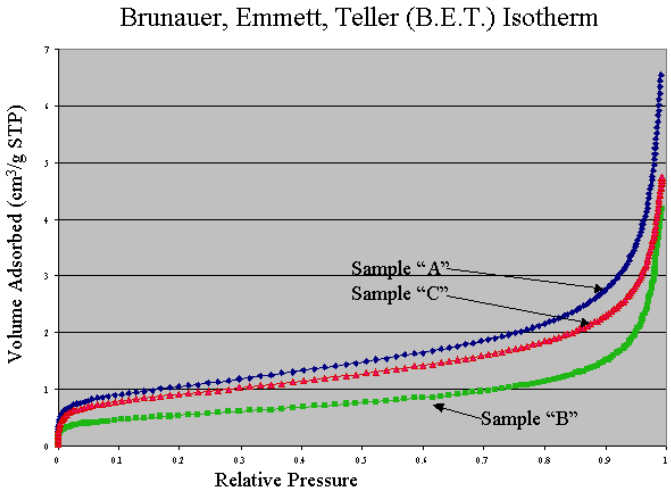

Table 12. BET Isotherm

Water Sensitivity Evaluation

The objective of this effort was to investigate the water sensitivity of MIC powders and to quantify its sensitivity in a production environment. Studies were conducted to determine if MIC materials lose chemical energy upon exposure to water over longer exposure periods and at elevated water temperatures. LANL examined the effects of water on the MIC material, which revealed that the Ultra Fine Grain (UFG) Al component of MIC could be degraded and made functionally inert by direct exposure to water. "Worst Case" testing was conducted with UFG powder dispersed in water.

In the fixed temperature (20°C), variable time exposure tests, no detectable degradation took place until 22 hours of exposure was reached. At the 22-hour mark, the MIC lost all reactivity and was essentially 'dead' (chemically inert). In fixed time, variable temperature exposure tests, the energy of the MIC degrades substantially at approximately 35°C (95°F) after 30 minutes exposure, and faster at higher temperatures. The differences in reaction between UFAL and conventional Aluminum powders are illustrated in Figures 8 and 9 (below). The information generated in this study and its findings is significant and is invaluable for development of safe and effective production process control and clean-up protocols.

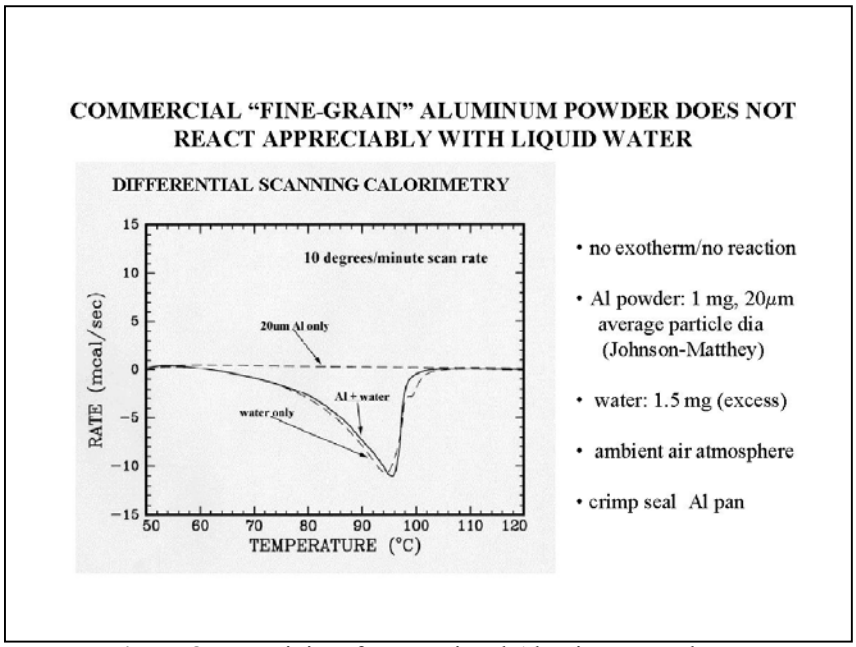

Figure 8: reactivity of conventional Aluminum powder

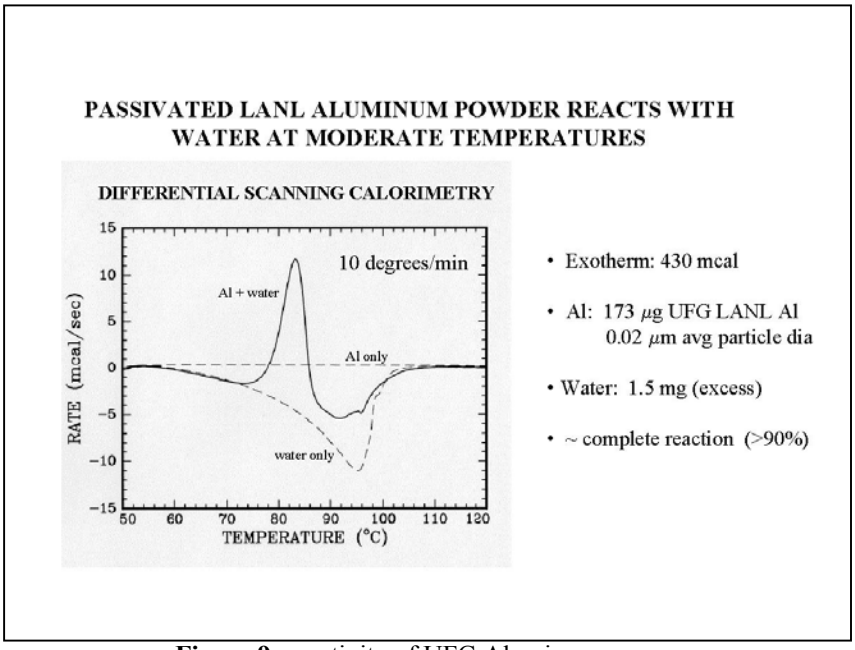

Figure 9: reactivity of UFG Aluminum

This study revealed that UFG Al is reactive with water, where larger particle size Al is not. Another discovery is that at elevated temperature UFG Al completely reacts with water yielding a significant exotherm. This information could prove useful for future spin-off applications for the MIC. An additional advantage of this transformation is a color change from black to white:

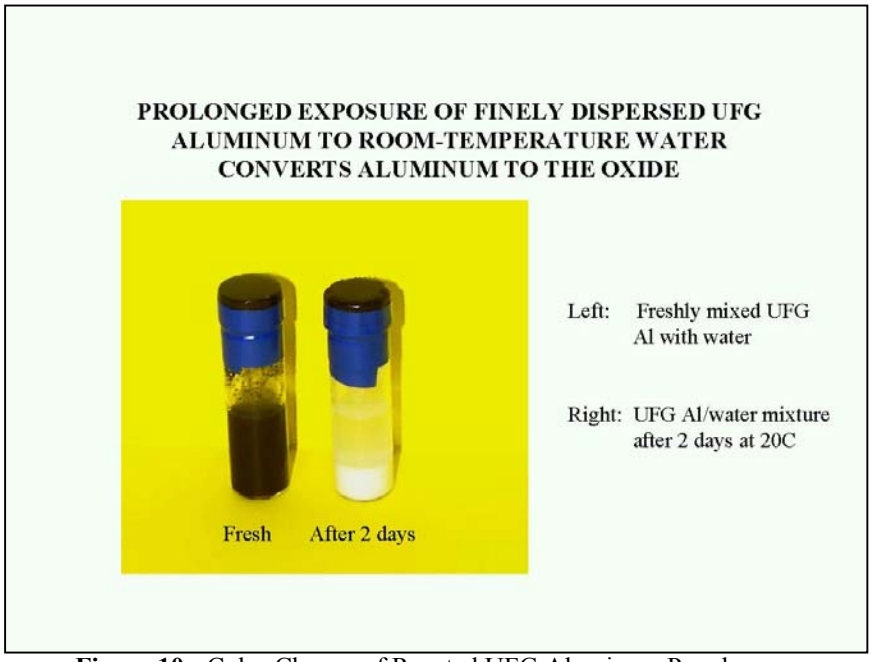

Figure 10: Color Change of Reacted UFG Aluminum Powder

Primer Evaluation

The intent of this effort was to keep the MIC composition the same, while determining if primer performance could be optimized by modifying the primer's metal parts configuration (5.56mm primer configuration: Figure 11). However, during early manufacturing of MIC material at LANL, different burn rate material was produced. Normal "fast-burning" MIC exhibits a characteristic loose-powder burn rate of 760 meters/second, whereas a "slower-burning" MIC exhibited a burn rate of 560 meters/second. The difference in the MIC burn rates was attributed to an increased average particle size for the aluminum particles in the slower-burning MIC. Therefore, the burn rate of the MIC material was added as a parameter for analysis.

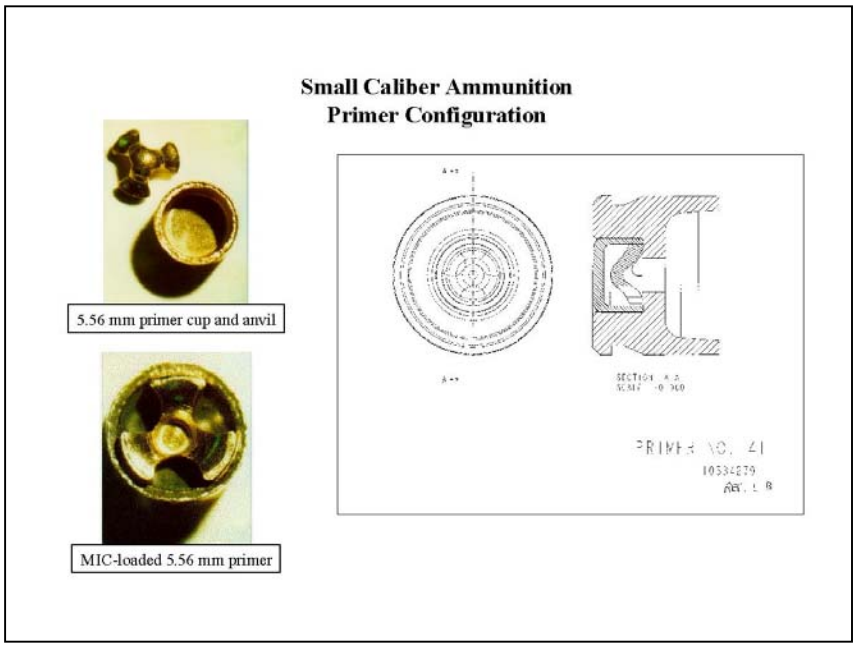

Figure 11: 5.56mm cartridge primer configuration (#41 primer)

A Taguchi Array was created to examine the physical configuration of the primer and systematically analyze each system parameter's affect on performance. Utilizing this experimental design required the testing of a fraction of the primer component combinations necessary to allow analysis. Specialized primer hardware components could not be culled from Lake City's normal production, so custom-made components were produced, loaded and evaluated.

While the custom parts were being manufactured for the Taguchi experiment, MIC material was used in standard component primers and other issues evaluated, such as MIC burn rate, effects of high and low temperature, storage, the effect of the cartridge case flash hole size, anvil geometry, etc. Testing was conducted on a new device developed for this study called a CAD Tester (Figure 12). The CAD Tester integrates and combines a standard primer "drop test" equipment setup with a closed bomb, instrumented with a dual transducer setup that allows pressure vs. time curves to be captured without firing 'full-up' cartridges:

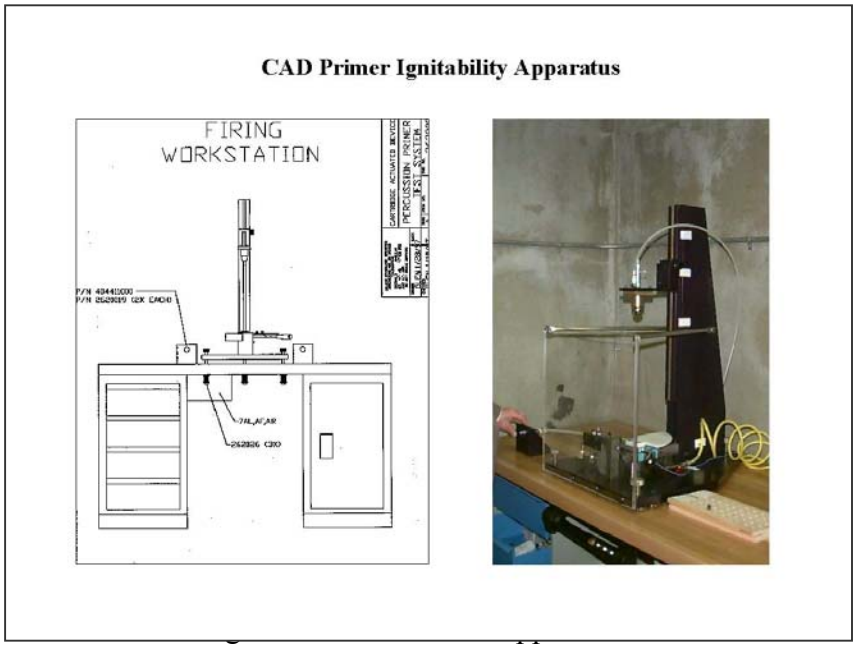

Figure 12: CAD Tester Apparatus

Historically, extreme cold temperature (-65°F and below) has been the defining problem for non-toxic primer mixtures, with any temperatures lower than 0°F typically causing the current generation of lead-free primers to become unreliable or to cease functioning entirely. Consequently, the affect of temperature extremes, as well as the affect of a larger flash hole, was chosen as the first phase of testing.

The flash hole is the vent between the primer pocket and the propellant chamber. In conventional primer mixtures, hot particles and hot gas flow generated by the primer flow through this vent into the cartridge case body and cause the propellant bed to be ignited. Since MIC yields no hot gas, the affect of the flash hole size on how quickly the hot particles flow into the propellant bed was a parameter worthy of analysis.

The key metric used to quantify results was Action Time. Action Time has various definitions and the one used in ammunition production is the time from firing pin strike to the time that the bullet exits the muzzle (or triggers the 'Port Pressure' transducer near the muzzle). The general Action Time requirements in small arms are typically 4 milliseconds at Cartridge Lot Acceptance and a lower value, such as 2.5 mSec, at Propellant (component level) Lot Acceptance. For this test, the definition of Action Time being used in the CAD closed bomb is from firing pin initiation until peak pressure. The average Action Time results and two representative pressure vs. time curves are illustrated below (Figures 13& 14; Table 13):

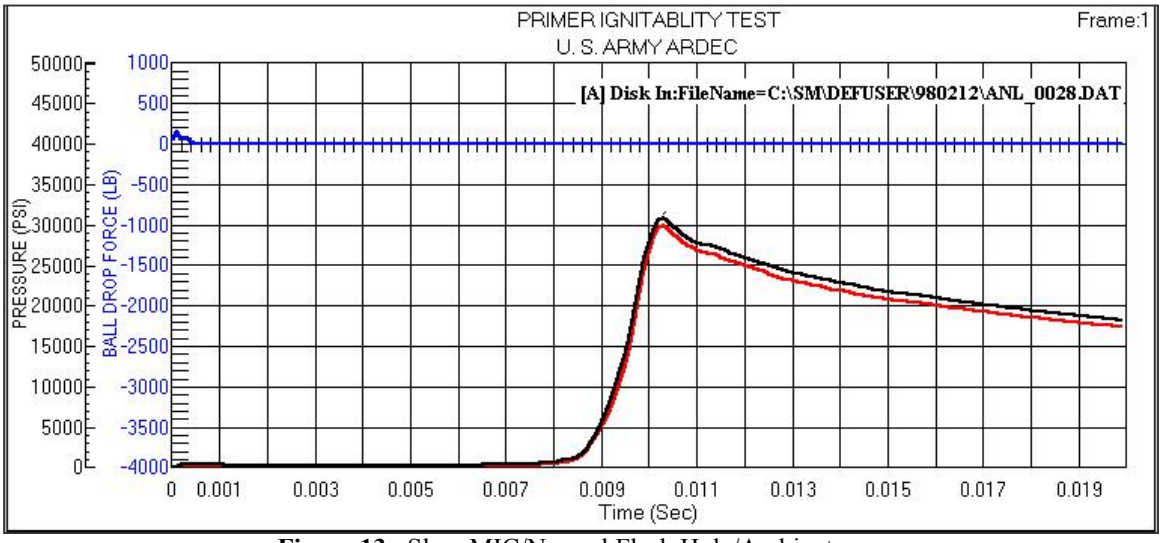

Figure 13: Slow MIC/Normal Flash Hole/Ambient

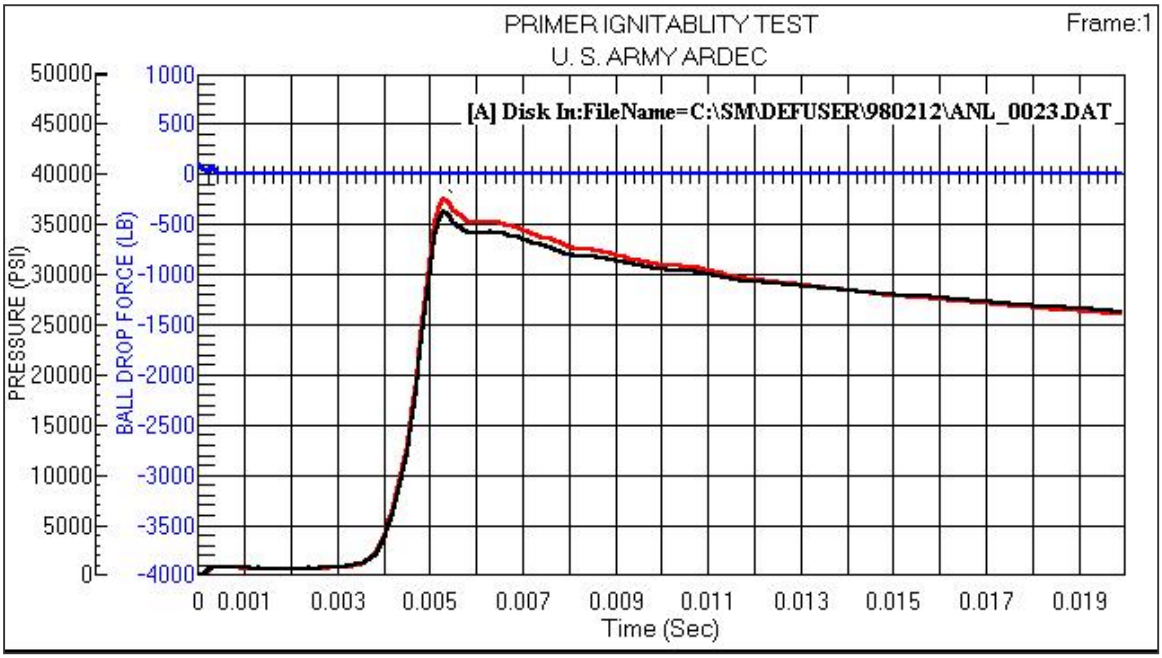

Figure 14: Normal MIC/Normal Flash Hole/Ambient

	Normal MIC			Slow MIC		
	Hot	Cold	Ambient	Hot	Cold	Ambient
Normal Flash Hole	6	7.8	5.6	18.5	20.2	11.4
Large Flash Hole	3.7	7.1	4.7	12.8	16.7	14.6

Table 13: Summary of Average Action Times (Milliseconds)

As can be seen in the above, a time delay of up to several additional milliseconds until peak cartridge pressure was observed for primers that were fabricated using slower-burning MIC compared with that of primers that were fabricated using "fast-burning" MIC.

This test result is significant because it confirms that the microscopic character of the MIC reactants must be carefully controlled if consistent, high performance is to be achieved and maintained in MIC primer manufacturing processes. In particular, the test reveals that primers fabricated using MIC powder that exhibits a substandard, loose-powder burn rate can be expected to also exhibit poor primer performance. Additionally, results revealed that the flash hole size has little or no affect on action time and temperature

Pressure/Action Time Baselining:

This effort developed a set of baseline information to quantify the performance of the current Lead Styphnate primer (FA-956) in the CAD tester, so that comparisons could be made to the various MIC compositions being developed:

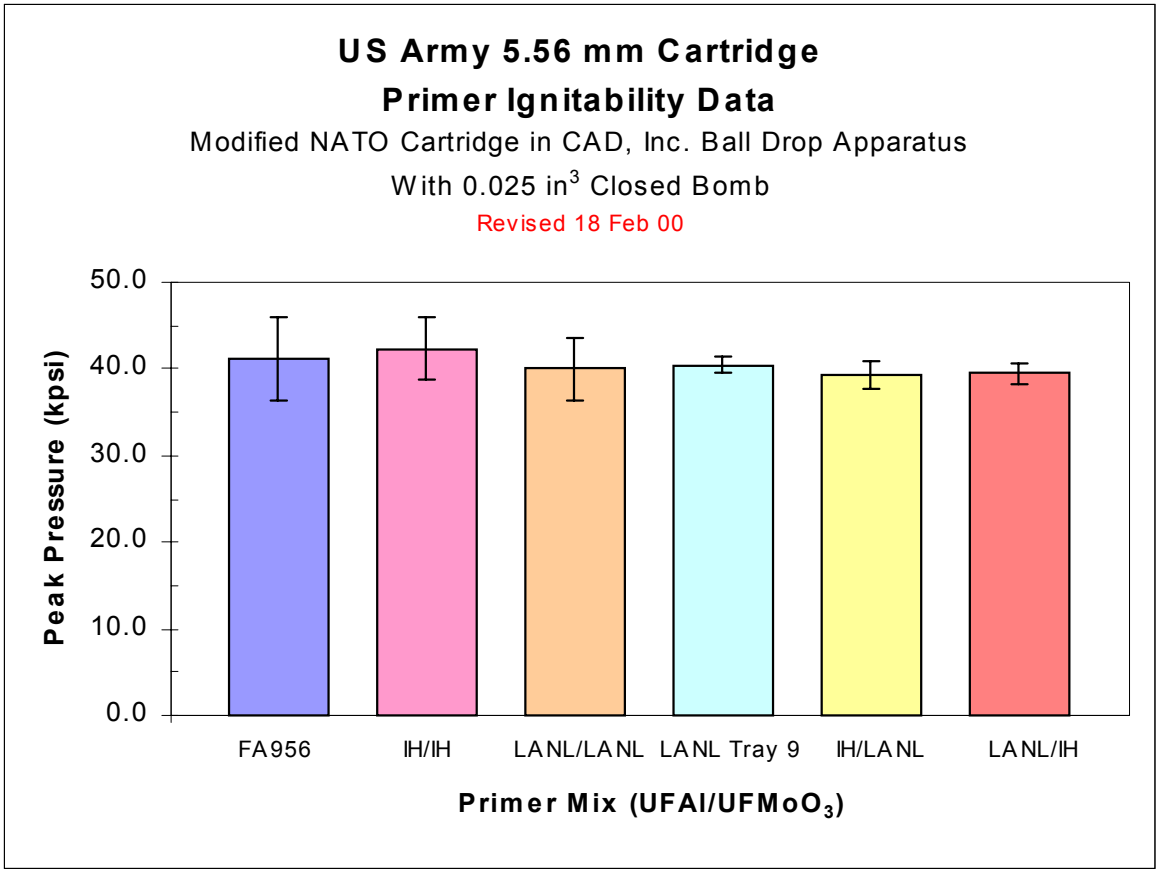

Figure 15: Peak Pressure versus primer compound

The goal of this evaluation was to determine if the proposed change from the baseline (FA-956 mix) to a MIC alternative would not significantly change the internal ballistics performance of

the weapon system, and thus be a "drop in" replacement. Figure 15 (above) illustrates that for the metric of Peak Pressure, the MIC alternatives appear to be feasible and satisfactory, with both nominal means and standard deviations well within the norm.

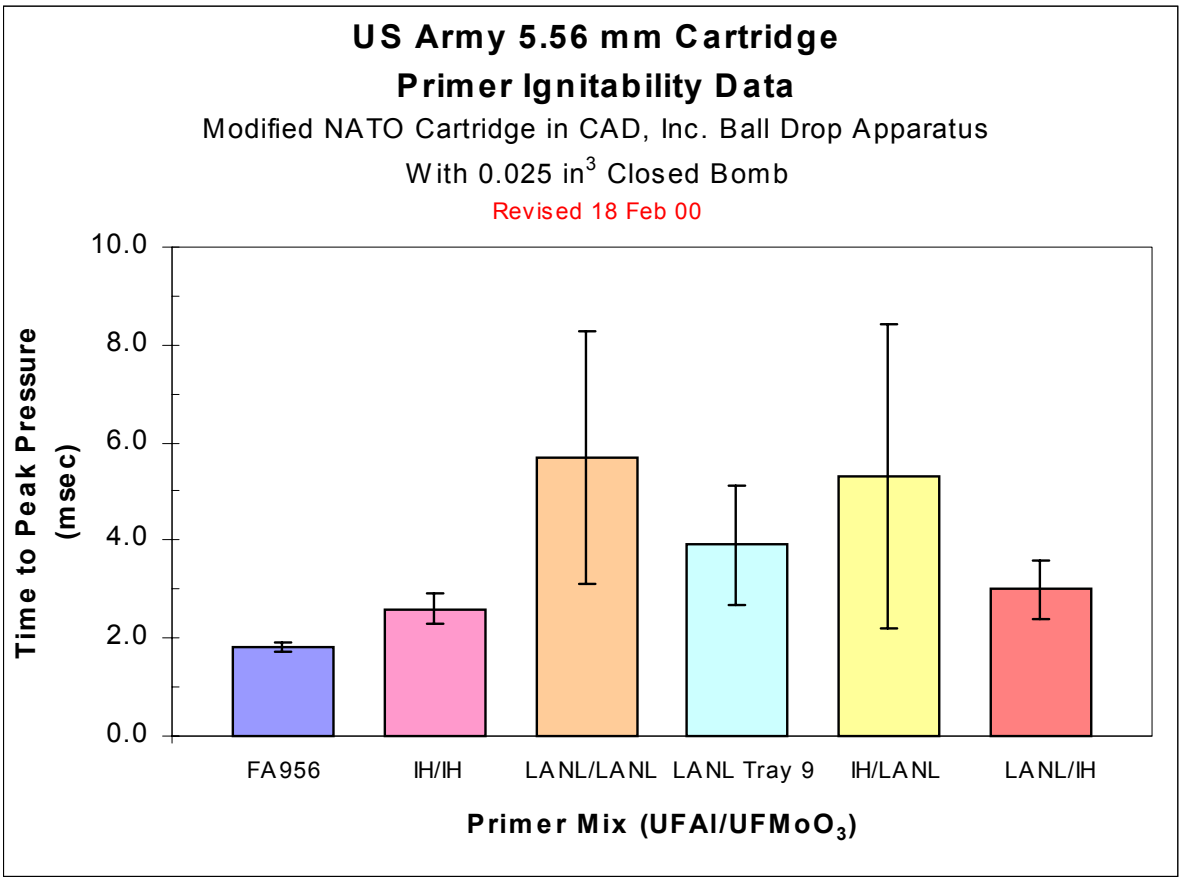

Figure 16: Action Time versus primer compound

The situation with Action Time, however, indicates that additional work is required. In small caliber ammunition, the cartridge level specifications call for a maximum response time of 4.0 milliseconds and component-level specifications add a safety margin to this and require a maximum of 2.5 msec. Consequently, additional performance optimization and characterization of the most promising production batches is required, predominantly to increase the overall rate of response; it is opinioned that the need for increased product uniformity is an artifact of the one-off handcrafted manufacturing environment of these experimental samples.

Taguchi – Phase II

The second phase of the Taguchi tests focused on the affect of temperature extremes as well as the affect of a larger flash hole than was used in Phase One. A parameter that was added to this phase of the testing was anvil geometry. In a spin-off effort with the Navy (discussed in the next section) it was discovered that the anvil used in their primers, the PVU anvil, had a different physical geometry. The PVU has a bipod configuration, rather than a Lake City (LC) anvil tripod, and has a sharply pointed, triangular apex. The PVU drawing is illustrated below:

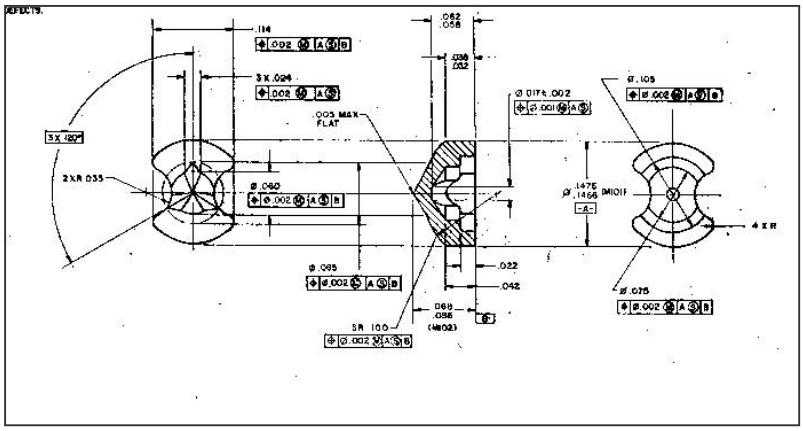

Figure 17: PVU anvil

This phase of testing was designed to provide more extensive information on the effects of these parameters than Phase One and the results are summarized below.

	Fast MIC			Slow MIC		
	Hot	Cold	Ambient	Hot	Cold	Ambient
Normal Flash Hole/LC Anvil	2.3	3.9	3.7	5.8	12.8	9.7
Normal Flash Hole/PVU Anvil	2.3	6.0	4.5	4.7	13.1	9.0
Large Flash Hole/LC Anvil	2.2	7.0	6.3	6.1	12.9	7.9
Large Flash Hole/PVU Anvil	2.2	6.7	4.6	6.0	20.5	9.7

Table 14: Average Action Time (Milliseconds)

Again, an unacceptable delay was observed for primers that were fabricated using slower-burning MIC. Additionally, results revealed that the flash hole size increase actually hindered action time, the PVU anvil did not aid the reduction of the action time and temperature had no significant affect on performance.

Taguchi 2^{15} Matrix Experiment

After LCAAP completed production of the custom primer components, LANL fabricated 256 M41-style MIC-based primers in support of the comprehensive Taguchi Experiment. The primers were fabricated in configurations consistent with the L_{16} (2^{15}) orthogonal array shown below. The Taguchi Array was created to examine the physical configuration of the primer and systematically analyze each system parameter's affect on performance. Utilizing this experimental design required the testing of a fraction of the primer component combinations necessary to allow analysis. The key parameters under investigation were pellet weight, anvil shape, anvil height, paper, primer cup base thickness, flash hole size, and primer seating depth.

	A Pellet Weight	B Anvil Shape	C Anvil Height	D Paper/ No Paper	E Primer Cup Base Thickness	G Seating Depth	F Flash Hole Size
1	17 mg	Flat	0.0750	Yes	0.0250	0.02	0.0820
2	17 mg	Flat	0.0750	Yes	0.0290	0.04	0.0950
3	17 mg	Flat	0.0820	No	0.0250	0.02	0.0950
4	17 mg	Flat	0.0820	No	0.0290	0.04	0.0820
5	17 mg	Pointed	0.0750	No	0.0250	0.04	0.0950
6	17 mg	Pointed	0.0750	No	0.0290	0.02	0.0820
7	17 mg	Pointed	0.0820	Yes	0.0250	0.04	0.0820
8	17 mg	Pointed	0.0820	Yes	0.0290	0.02	0.0950
9	21 mg	Flat	0.0750	No	0.0250	0.04	0.0950
10	21 mg	Flat	0.0750	No	0.0290	0.02	0.0820
11	21 mg	Flat	0.0820	Yes	0.0250	0.04	0.0820
12	21 mg	Flat	0.0820	Yes	0.0290	0.02	0.0950
13	21 mg	Pointed	0.0750	Yes	0.0250	0.02	0.0820
14	21 mg	Pointed	0.0750	Yes	0.0290	0.04	0.0950
15	21 mg	Pointed	0.0820	No	0.0250	0.02	0.0950
16	21 mg	Pointed	0.0820	No	0.0290	0.04	0.0820

Table 15: Taguchi $L_{16}(2^{15})$ Orthogonal Array

The testing was conducted at ARDEC and the data was analyzed and reduced and a response table formulated exhibiting each parameter's effect on performance. The response tables were formulated looking at two specific primer performance parameters: action time and sensitivity. Action time is the time between firing pin strike and peak pressure, whereas, sensitivity is a measure of the amount of firing pin energy required to function the primer. The response tables are shown below.

Taguchi Response Tables

Action Time

	A	G	E	C	AxE	AxF	AxC	BxF
Level 1	0.04930	0.04804	0.04078	0.04711	0.04154	0.04660	0.04654	0.04204
Level 2	0.03903	0.04029	0.04755	0.04123	0.04679	0.04173	0.04179	0.04629
Delta	0.01027	0.00775	0.00676	0.00588	0.00525	0.00486	0.00475	0.00425
Delta (xK)	10.27	7.75	6.76	5.88	5.25	4.86	4.75	4.25
Rank	1	2	3	4	5	6	7	8

Sensitivity

	B	E	C	BxE	AxF	BxC	G	D
Level 1	17.50	12.60	15.30	15.20	14.80	14.70	14.60	13.50
Level 2	10.60	15.50	12.80	12.90	13.30	13.40	13.50	14.60
Delta	6.90	2.90	2.50	2.30	1.50	1.30	1.10	1.10
Rank	1	2	3	4	5	6	7	8

Table 16: Response Tables to Taguchi $L_{16}(2^{15})$ Experiment

In summary, the key parameters identified by the Taguchi test, in priority order, were as follows:

Key Critical Parameters for Action Time
1) Pellet Weight (Heavier)
2) Seating Depth (Deeper)
3) Cup Thickness (Thinner)

Key Critical Parameters for Sensitivity
1) Anvil Shape (Pointier)
2) Cup Thickness (Thinner)
3) Anvil Height (Taller)

Validation Testing

Based on the results of the Taguchi Experiment, validation tests were conducted. The best configuration, based on the response table parameters, was fabricated at LANL. The best configuration utilized a thin base primer cup. Since there is some risk that using a thinner than normal cup could lead to pierced primers when firing in actual weapons, a second configuration was also loaded with identical parameters but with a standard thickness primer cup.

Configuration A

Pellet Weight - 21mg
Anvil Shape - Pointed & Tall (Small Radius 0.024, Tall Height 0.823)
No Paper in the Cup prior to loading MIC
Primer Cup - Thin Base

Configuration B

Pellet Weight - 21mg
Anvil Shape - Pointed & Tall (Small Radius 0.024, Tall Height 0.823)
No Paper in the Cup prior to loading MIC
Primer Cup - Standard Thickness

Each configuration was sent to ARDEC and tested using the CAD tester, at three different temperatures, Hot (+155°F), Cold (-65°F), and Ambient (70°F) Testing. Both configurations successfully met all cartridge requirements, although the MIC mix did not strictly equal the Action Time performance of the Lead Styphnate based primer material slated for replacement.

High Temperature/High Humidity Environment Experiment

Loaded and unprotected MIC primers were exposed to a continuous high temperature (+125F) environment, at both low and high (100%) humidity levels, and surveillance tested over a period of 49 days (7 weeks). The hot/dry test indicated no measurably adverse affect on performance. Not unexpectedly, the high humidity test did show an adverse affect, both to the action time and to the reliability of the primers:

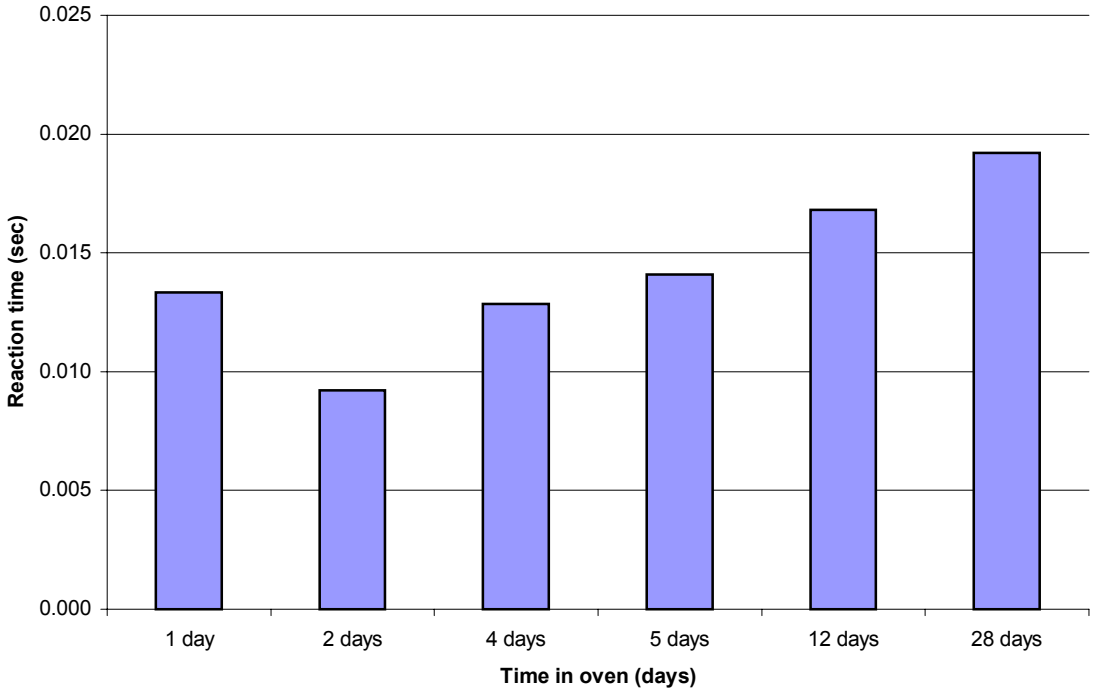

Figure 18: Average Action Times from prolonged Humidity exposure

Not shown in the above figure was that a very high 40% misfire rate was observed in both the high-humidity's 28-day (2 out of 5) and 49-day (6 out of 15) samples. There was also an increasing incidence of hangfires whose action times could not be precisely measured, because their response time was so delayed such that it exceeded the instrumentation's 40millisecond time measurement window. Hangfires are a particular concern in externally driven weapons, such as the 7.62mm, M134 'Minigun' (GAU-2).

MIC Safety Testing

The following safety tests were performed on a sample of MIC:

a. BOE Sensitivity Test
b. Electrostatic Sensitivity Test
c. BAM Small Friction Sensitivity Test
d. Ball Drop Impact Test

The BOE Impact Test Method is given in the department of Defense Explosives Hazard Classification Procedures, ARMY TB 700-2. The sample had no reaction at impact height of 3 ¾-inches. The impact height was increased to 9.75 then 15 inches. At 15 inches, the sample reacted in 2 out of 10 trials.

The electrostatic sensitivity test was performed accordance with MIL-STD-650, Method 512.1, "Approaching-Electrode Electrostatic Sensitivity Test". The MIC material was found to be EXTREMELY sensitive to electrostatic stimulus compared to the other primary explosive. For example, a person can generate approximately 0.02 joule by walking across a carpet. The MIC material reacted at 0.00010 joule (the lowest this tester can handle), so its true value is lower. Proper grounding of personnel and equipment will be a major consideration with MIC production, as will likely be environmental humidity control within the appropriate areas of the production facilities.

The BAM Small friction tester was used to determine the friction sensitivity of the samples. A sample was placed on the porcelain plate. The porcelain pin was lowered onto the sample and a weight was placed on the arm to produce the desired load. The tester was activated and the porcelain plate was reciprocated once to and from. The results are observed as either a reaction (i.e. flash, smoke, and/or audible report) or no reaction. Testing is begun at the maximum load of the apparatus (1000 gm) or lower if experience warrants it. If a reaction occurs in ten trials, the load is reduced until no reactions are observed in ten trials. The minimum load value at which reaction occurs is reported in grams. The MIC material reacted at 10 grams load (the lowest load value that this tester can handle), so its true value is lower. Due caution is again appropriate.

The ball drop impact test was used to determine the impact sensitivity of the samples. Approximately 30 mg of MIC sample is placed on a steel block and spread into a thin layer. A steel ball (0.934 inch diameter) weighing 55.0 grams is placed into a ball track above the steel block. The ball track is set to the desired height. The steel block is positioned under the ball track. The steel ball is released and impacts the MIC sample. An iterative procedure is used to determine the maximum height at which no positive results are obtained in 20 trials. The ball track height is adjusted in increments of one inch. There was no positive result at a height of 30 inches for the MIC sample (the maximum height for the ball impact tester), which is a favorable result.

Primer Mix Scaling Experiments

The demonstrated manufacturing process of the MIC primer mix consists of the precise measurement of UFAL and MoO_3 in the proper blend ratio, to which is added a specified amount of Cyclohexane solvent and blended in a cooled ultrasonic bath. The blended mixture is then thinly spread on a metal plate, which is then mildly heated to accelerate the evaporation of the Cyclohexane to dry the primer mix. The dried material is then slowly and carefully scraped off the plate by hand with a razor blade. This powder is then stored, or it can be used immediately. Loading is accomplished by a controlled pressing (consolidation) of the MIC powder, dry, into the primer cup.

The standard batch size by this process is 0.5 grams and takes approximately one hour to produce. The process has been successfully scaled up to 0.75g, but there are practicality limitations on the drying plate that discourages any further scaling. Consequently, alternatives to the 'hot plate' have been under investigation.

Discrete experiments of alternative processing methods were conducted over the period. Currently, the most promising of these is a 10g batch size, which is filtered and then slowly dried for ~10 hours under ambient laboratory conditions ($20^{o}C$). This line of experimentation has been nominally successful, although two issues are known to be present: the first is an solitary occurrence where the drying sample apparently underwent a partially exothermic reaction and blew itself apart without burning the filter paper. This line of experimentation was immediately suspended until additional instrumentation was obtained and installed to monitor the MIC blend's internal temperatures during the drying cycle and it is now in use on all subsequent 'filter and dry' experiments. In subsequent trials to date, the singular event has not recurred and no irregularities have been detected which would provide insight. The second challenge is that this process results in partial clumping of the dried MIC blend, which makes it unsuitable for loading into primers. One method of follow-on processing that has been tested is to reapply the Cyclohexane solvent to the reducd mass (the clump), break up the clump while wet, and then redry the material, although alternative solvents are also under investigation.

While scaling-up is occurring, an additional philosophical goal is to maximize the percentage of the process in which the MIC material remains 'wet', as this generally affords a safer manufacturing process.

Improvement of Action Time – Gas Generators

Related to the above processing experiments are changes in solvent, or more properly 'chemically non-reactive carrier solutions'. It was found that replacing Cyclohexane with Isopropyl Alcohol resulted in a slight improvement in Action Time, to a nominal average of ~3 msec. It has, however, been found that the Isopropyl Alcohol is less compatible with the hotplate drying method than the Cyclohexane, which limits its utility in baselining changes through the smaller but faster current 'hotplate' fabrication method. Additionally, a different approach to improving Action Time has been yielding better results, so Isopropyl Alcohol is currently shelved while the more promising approach is being pursued.

The most promising approach at present to improve the action time of the MIC blend is to hybridize the base MIC chemistry by introducing a gas generation substance. In doing so, the MIC primer compound more closely resembles the traditional Lead Styphnate primer mix in that its brisance includes components of heat, hot particles and hot gasses. Literature searches and subject matter expertise has been used to identify suitable materials and through limited experimentation, the most promising composition blend to date is MIC with a +10% additive of Ethyl Cellulose (Cyclohexane solvent). This composition blend has reduced action times to ~2.5msec and, while testing is still very limited, appears to also be reducing the frequency of both misfires and hangfires which have been spuriously occurring over the course of the program. Both metrics – action time and reliability – are indicative of an improved ignitability of the propellant bed without any process or chemistry changes to the current propellant compositions.

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Appendix

Results of Chemical Stability & Mobility Experiments (Sections A through O).

STANDARD RUN A. Sand and De-Ionized Water

Media: *Sand*
Solvent: *De-ionized water*
Technique: Up-flow and Hold
Bullet types: Lead M855
ESP-1 M855
ESP-1 9 mm Ball (FMJ)
TRI-1 M855
TRI-1 9 mm Ball (FMJ)

Summary: Tungsten was leached but at levels of < 20 ppm and with indication of decreasing concentration with time. Highest levels were for columns with the highest concentrations of tungsten, i.e. TRI-1 9 mm Ball.

Comments: The leachant was more closely examined before shipping. Some amount of sediment was evident. It appeared that some particulate matter had passed through the filter. It is believed that small particles of sand and tungsten, < 0.7 microns, could have passed through the packed bed and filter. This could explain the higher concentrations of tungsten in the experiments using sand as the media.

Table A-1: Metals Analysis from Up-Flow – Sand + Deionized Water
(Concentrations in mg/L)

Detection limits

W	Sn	Zn	Fe	Cu	Pb
0.05	0.005	0.005	0.01	0.005	0.01

Deionized water

W	Sn	Zn	Fe	Cu	Pb
< 0.05	< 0.005	< 0.005	0.0532	< 0.005	< 0.01

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	0.05	< 0.005	0.0169	0.115	< 0.005	< 0.01
2	0.05	< 0.005	0.0086	< 0.01	0.0065	< 0.01
4	< 0.05	< 0.005	< 0.005	0.168	< 0.005	< 0.01
8	0.05	< 0.005	< 0.005	0.1200	< 0.005	< 0.01
16	< 0.05	< 0.005	< 0.005	0.0824	< 0.005	< 0.01
30	< 0.05	< 0.005	< 0.005	0.0636	< 0.005	< 0.01

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	3.61	< 0.005	0.0051	0.4010	0.0065	< 0.01
2	3.42	< 0.005	< 0.005	0.0106	0.0056	< 0.01
4	3.31	< 0.005	< 0.005	< 0.01	0.0095	< 0.01
8	3.19	< 0.005	< 0.005	< 0.01	0.0059	< 0.01
16	4.41	< 0.005	< 0.005	0.0150	0.0061	< 0.01
30	4.46	< 0.005	< 0.005	0.0529	0.0066	< 0.01

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	7.11	< 0.005	0.0139	0.0115	0.0105	< 0.01
2	6.53	< 0.005	0.0103	0.0258	0.0113	< 0.01
4	4.59	< 0.005	0.0104	< 0.01	0.0118	< 0.01
8	4.91	< 0.005	< 0.005	0.1130	0.0082	< 0.01
16	6.38	< 0.005	< 0.005	0.0398	0.0089	< 0.01
30	5.19	< 0.005	< 0.005	0.0464	0.0093	< 0.01

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	15.60	< 0.005	0.0125	0.0107	0.0091	< 0.01
2	7.40	< 0.005	< 0.005	< 0.01	< 0.005	< 0.01
4	8.52	< 0.005	< 0.005	0.0149	< 0.005	< 0.01
8	7.28	< 0.005	< 0.005	< 0.01	< 0.005	< 0.01
16	7.25	< 0.005	< 0.005	< 0.01	< 0.005	< 0.01
30	10.00	< 0.005	< 0.005	0.0367	< 0.005	< 0.01

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	14.30	< 0.005	0.0084	< 0.01	0.0058	< 0.01
2	6.12	< 0.005	0.0061	< 0.01	0.0067	< 0.01
4	11.30	< 0.005	< 0.005	0.0151	0.0083	< 0.01
8	13.40	< 0.005	< 0.005	0.2050	0.0056	< 0.01
16	11.80	< 0.005	< 0.005	0.0621	< 0.005	< 0.01
30	9.10	< 0.005	0.0068	0.0656	< 0.005	< 0.01

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	0.06	< 0.005	0.0129	0.0188	< 0.005	< 0.01
2	< 0.05	< 0.005	0.0123	0.0744	< 0.005	< 0.01
4	< 0.05	< 0.005	0.0255	0.0717	< 0.005	< 0.01
8	< 0.05	< 0.005	0.0100	< 0.01	< 0.005	< 0.01
16	< 0.05	< 0.005	< 0.005	0.0116	< 0.005	< 0.01
30	< 0.05	< 0.005	0.0064	0.0819	< 0.005	< 0.01

Table A-2: Metals Analysis from Hold - Sand + Deionized Water
Concentrations in mg/L

Detection limits

W	Sn	Zn	Fe	Cu	Pb
0.05	0.005	0.005	0.01	0.005	0.01

Deionized water

W	Sn	Zn	Fe	Cu	Pb
< 0.05	< 0.005	< 0.005	< 0.01	< 0.005	< 0.01

Control

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.005	0.111	1.14	0.0079	< 0.01
15	< 0.05	< 0.005	0.146	1.46	< 0.005	< 0.01
35	< 0.05	< 0.005	0.0587	0.115	0.0051	< 0.01

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	3.70	0.0148	0.241	1.61	0.0119	< 0.01
15	1.74	0.0596	0.383	8.90	0.017	0.0429
35	1.10	< 0.005	0.0561	0.359	< 0.005	< 0.01

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	4.49	0.0292	0.0901	0.311	0.012	< 0.01
15	1.81	0.113	0.437	7.280	0.0215	0.0394
35	1.46	< 0.005	0.0825	0.167	0.0061	< 0.01

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	11.9	< 0.005	0.041	0.0464	0.0165	< 0.01
15	5.39	< 0.005	0.0747	0.0484	0.0051	< 0.01
35	2.79	< 0.005	0.0722	1.010	0.0065	< 0.01

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	5.56	0.0149	0.400	5.71	0.0207	0.0293
15	3.01	< 0.005	0.385	5.76	0.0118	0.0266
35	5.07	< 0.005	0.429	8.40	0.0170	0.0338

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.005	0.0958	0.0709	0.0156	< 0.01
15	0.0573	< 0.005	0.139	1.370	0.0087	0.0887
35	< 0.05	< 0.005	0.0936	0.0426	0.0063	< 0.01

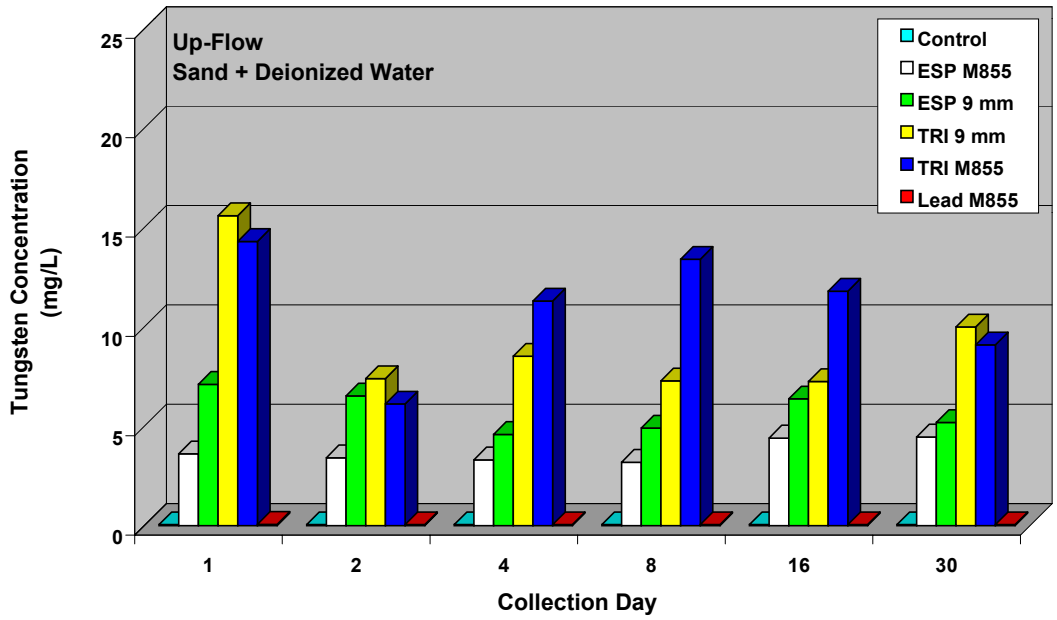

Figure A-1. Tungsten concentration in leachate for up-flow test using sand and de-ionized water.

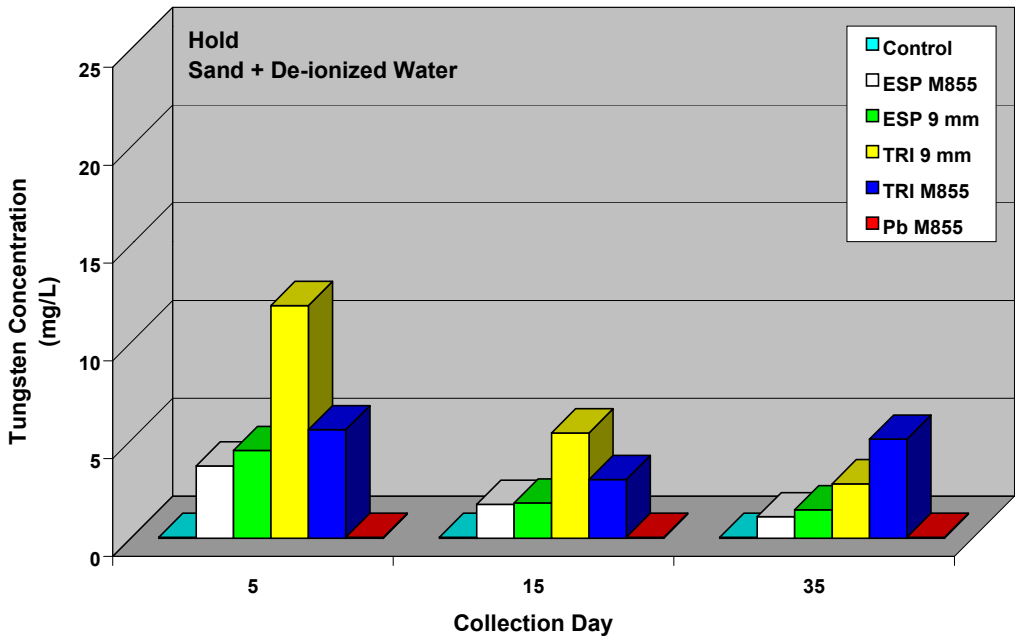

Figure A-2. Tungsten concentration in leachate for hold test using sand and de-ionized water.

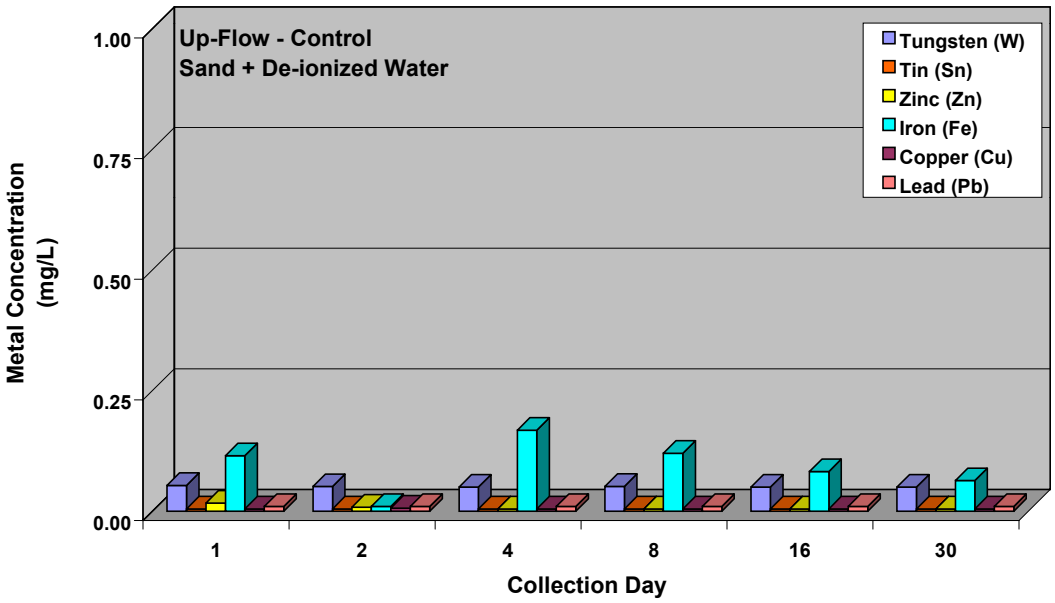

Figure A-3. Metal concentrations in leachate for control sample from the up-flow test using sand and de-ionized water.

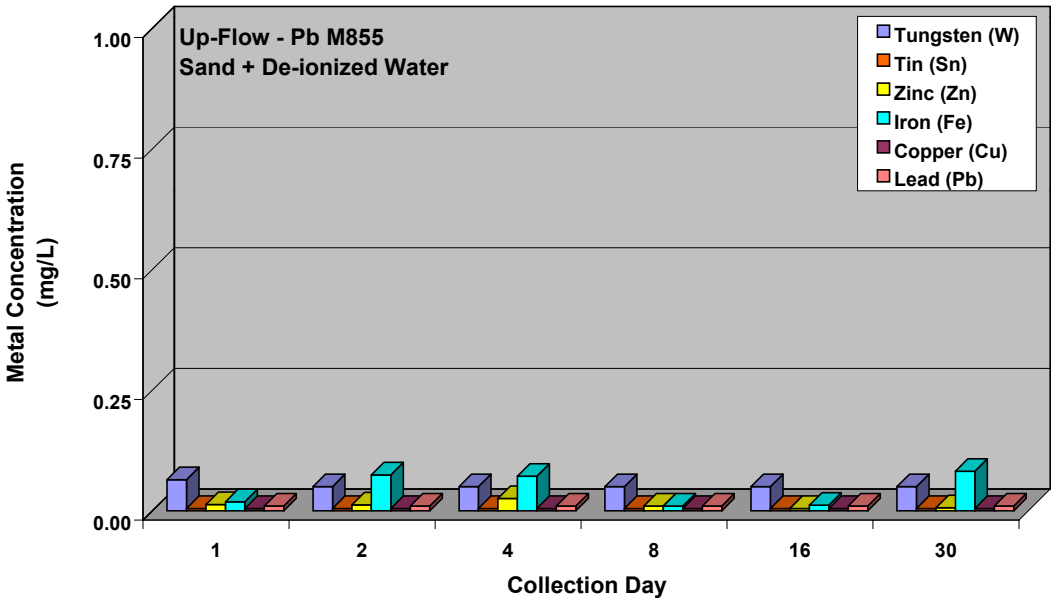

Figure A-4. Metal concentrations in leachate for lead M855 sample from the up-flow test using sand and de-ionized water.

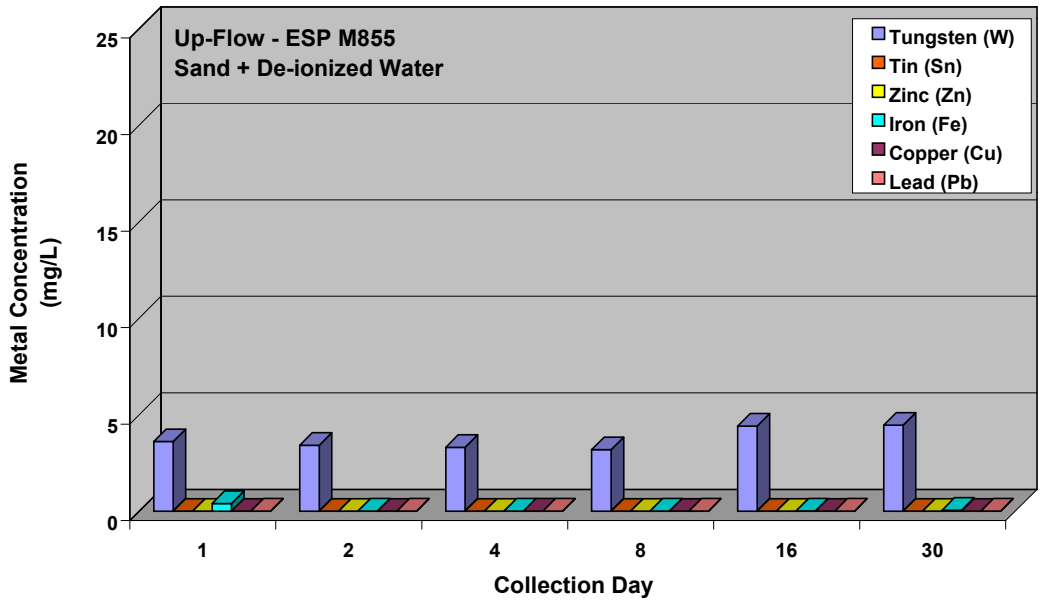

Figure A-5. Metal concentrations in leachate for ESP M855 sample from the up-flow test using sand and de-ionized water.

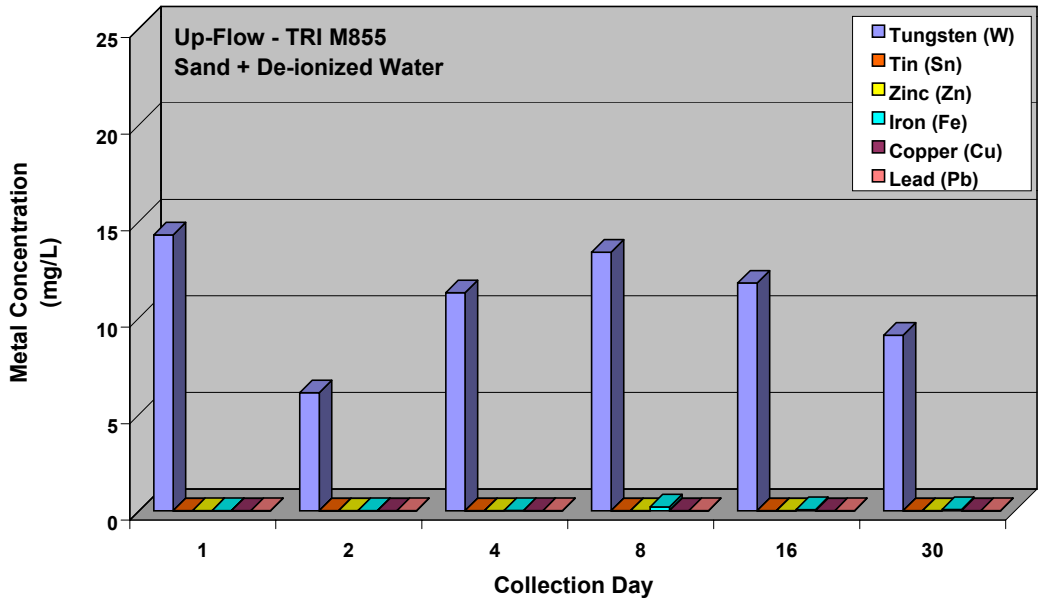

Figure A-6. Metal concentrations in leachate for TRI M855 sample from the up-flow test using sand and de-ionized water.

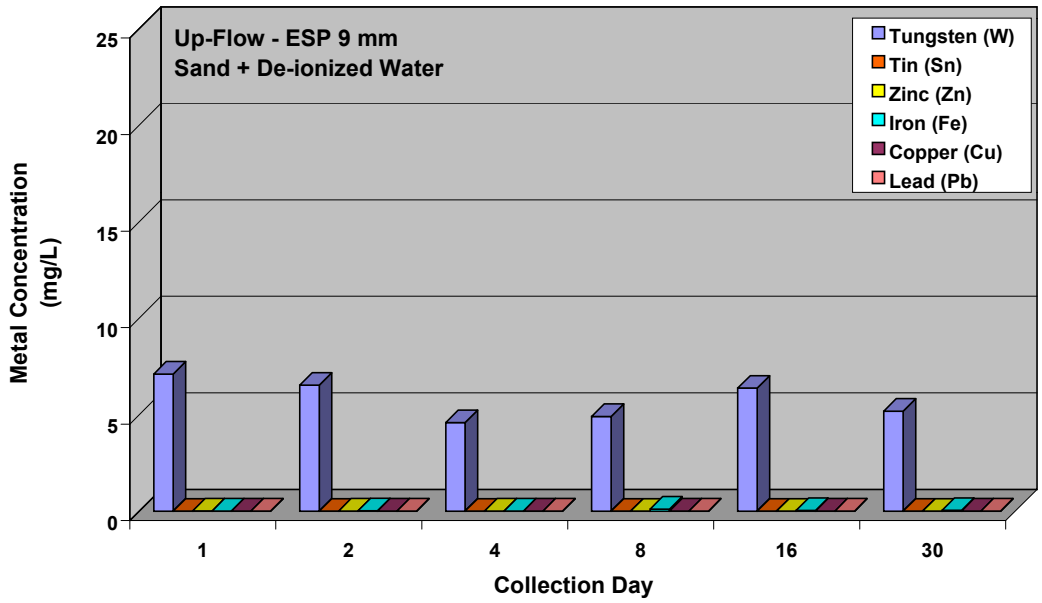

Figure A-7. Metal concentrations in leachate for ESP 9 mm sample from the up-flow test using sand and de-ionized water.

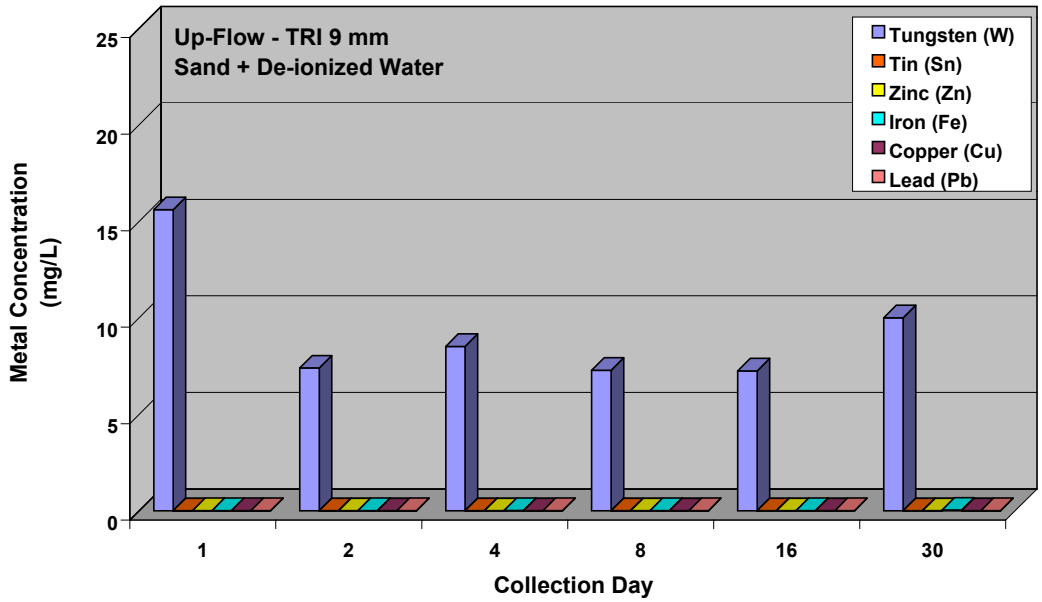

Figure A-8. Metal concentrations in leachate for TRI 9 mm sample from the up-flow test using sand and de-ionized water.

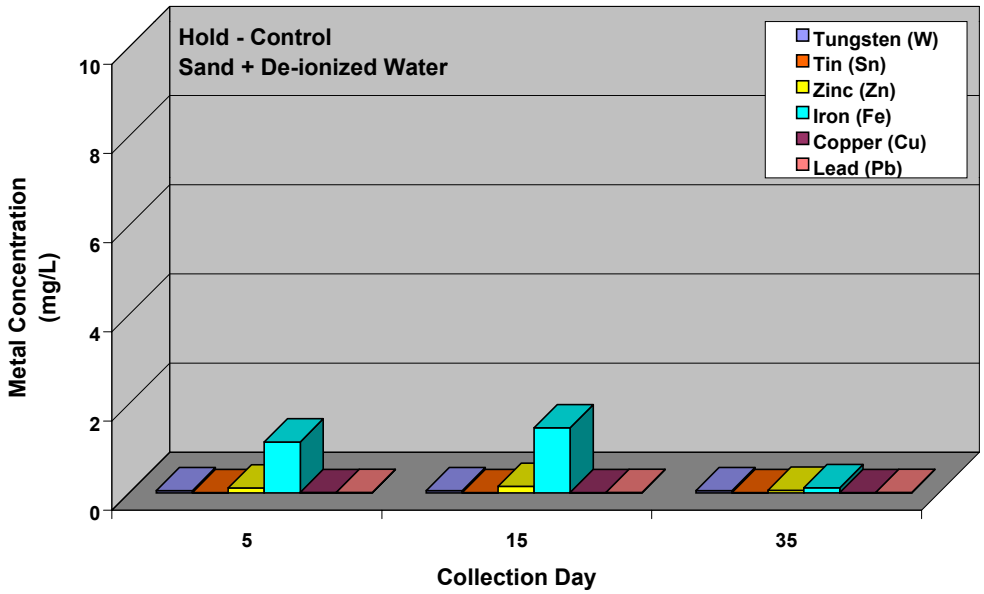

Figure A-9. Metal concentrations in leachate for control sample from the hold test using sand and de-ionized water.

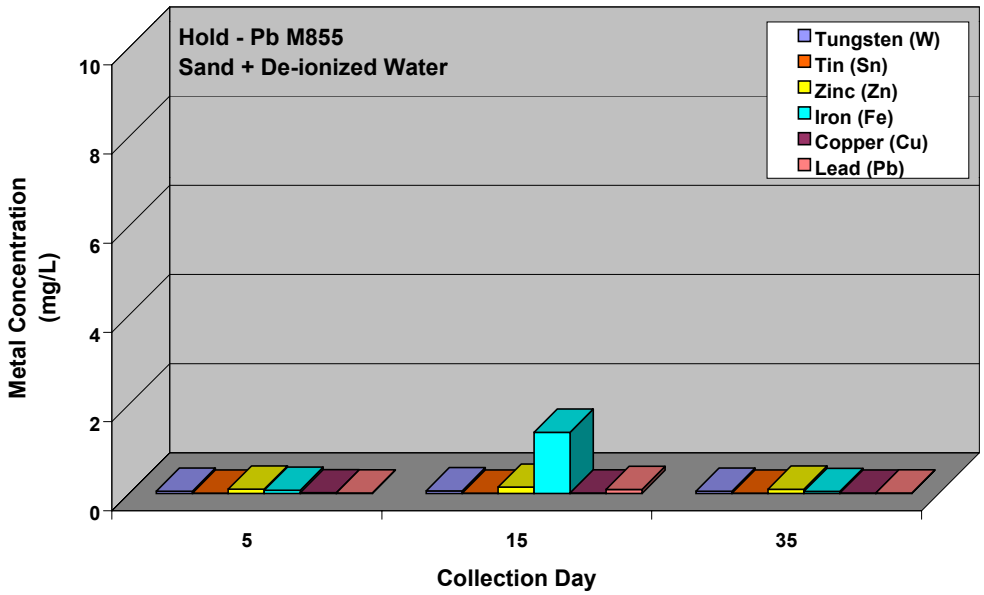

Figure A-10. Metal concentrations in leachate for lead M855 sample from the hold test using sand and de-ionized water.

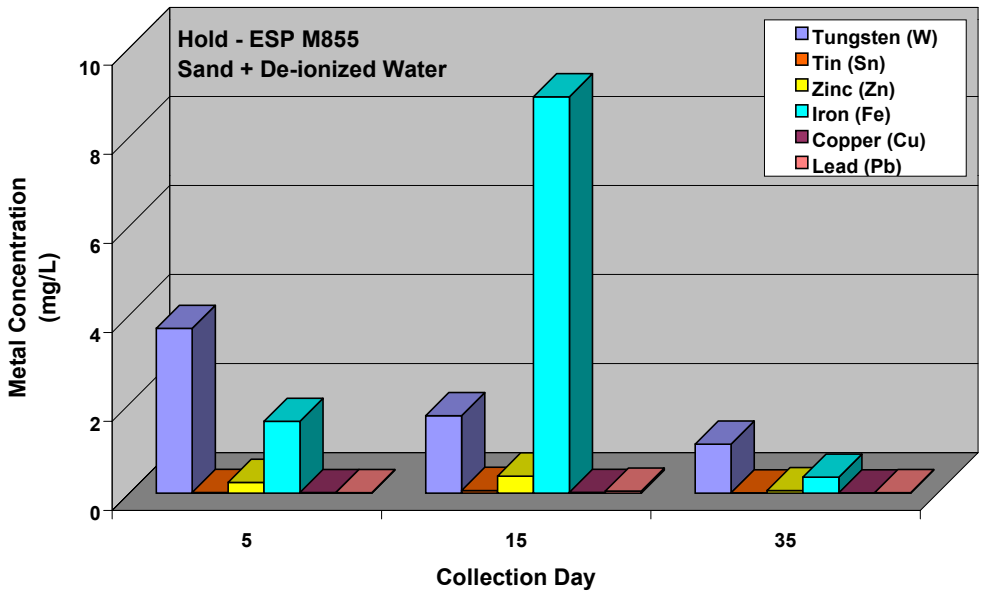

Figure A-11. Metal concentrations in leachate for ESP M855 sample from the hold test using sand and de-ionized water.

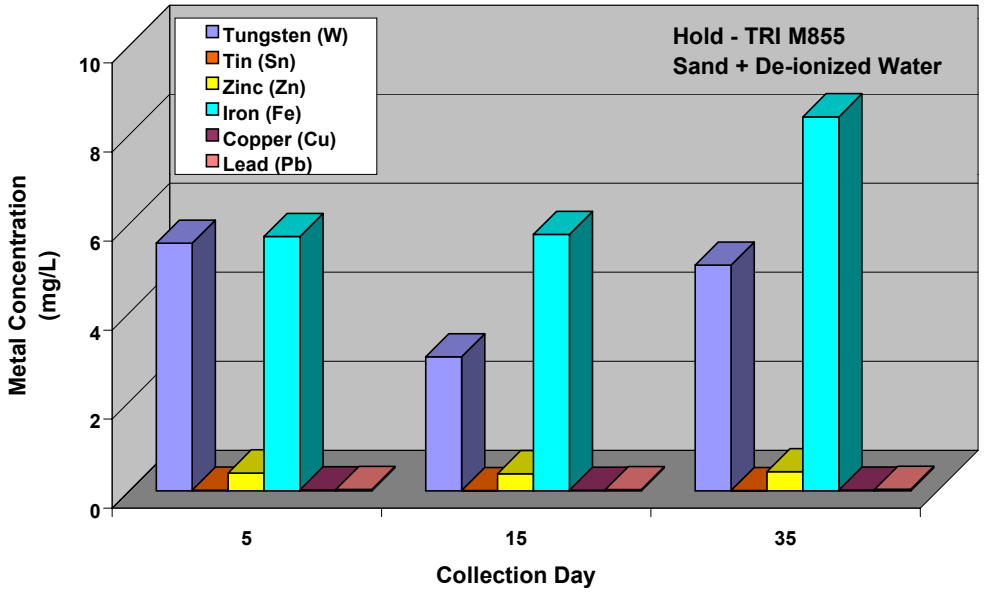

Figure A-12. Metals concentrations in leachate for TRI M855 sample from the hold test using sand and de-ionized water.

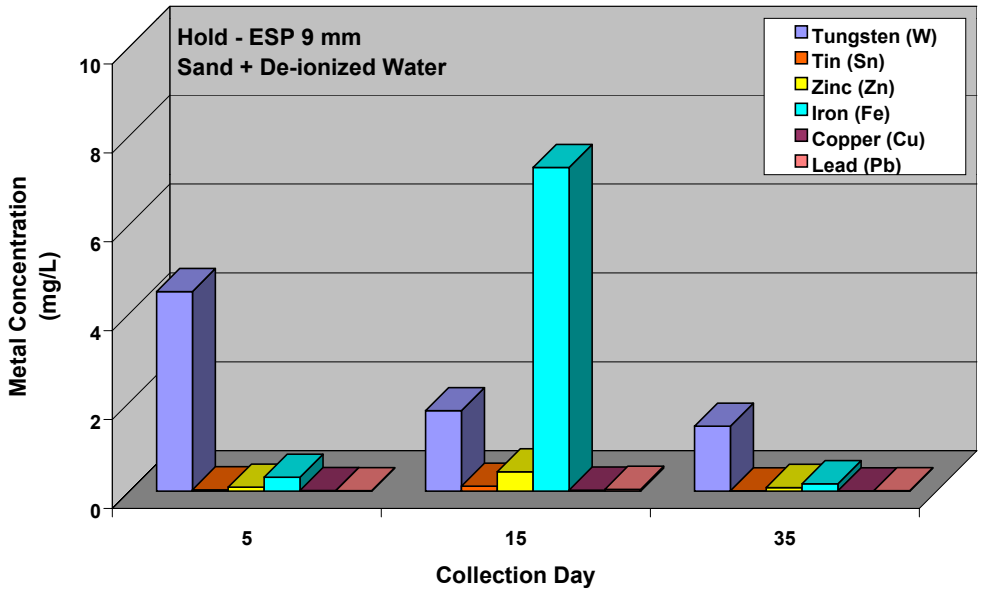

Figure A-11. Metals concentrations in leachate for ESP 9 mm sample from the hold test using sand and de-ionized water.

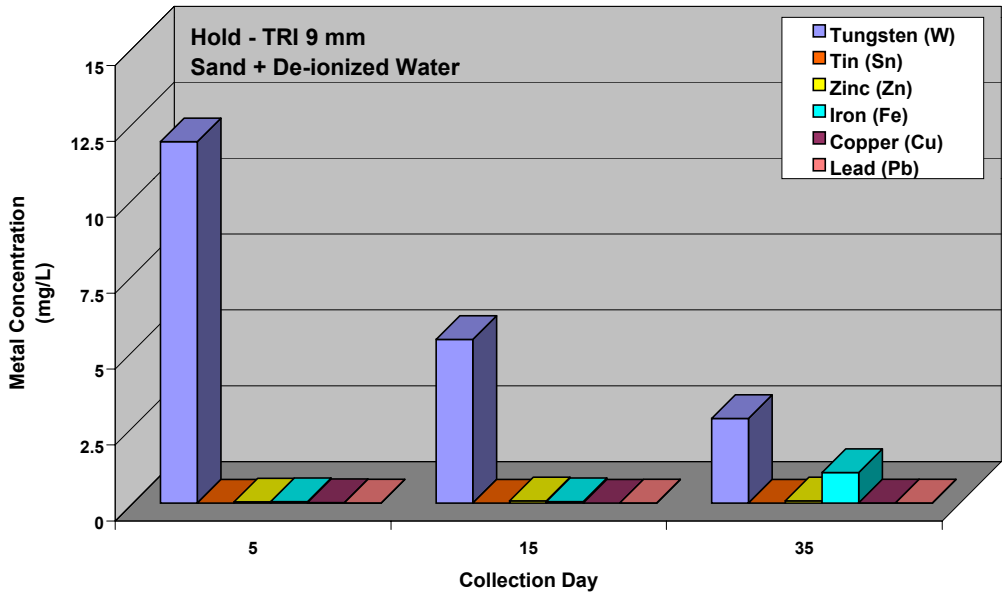

Figure A-12. Metal concentrations in leachate for TRI 9 mm sample from the hold test using sand and de-ionized water.

STANDARD RUN B. Soil with De-Ionized Water

Media: *Soil – Eastern TN top soil*
Solvent: *De-ionized water*
Technique(s): Up-flow and Hold
Bullet types: Lead M855
ESP-1 M855
ESP-1 9 mm FMJ
TRI-1 M855
TRI-1 9 mm FMJ

Summary: No tungsten was found in the leachant. Levels were below the detection limits of instrument (0.05 ppm).

Comments: The sample from Day 16 of the up-flow test for TRI-1 9 mm Ball was lost during handling and shipping for chemical analysis.

Table B-1: Metals Analysis: Up-Flow – De-ionized Water + Soil
(Concentrations in mg/L)

Detection limits

W	Sn	Zn	Fe	Cu	Pb
0.1	0.01	0.005	0.01	0.003	0.01

De-ionized water

W	Sn	Zn	Fe	Cu	Pb
< 0.05	< 0.005	< 0.005	0.0532	< 0.005	< 0.01

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.1	0.0114	0.376	0.0239	0.0054	0.0196
2	< 0.1	< 0.00	0.206	0.0336	0.004	0.0143
4	< 0.1	< 0.01	0.150	0.0418	< 0.003	< 0.01
8	< 0.1	< 0.01	0.0547	0.303	< 0.003	< 0.01
16	< 0.1	< 0.01	0.0126	0.627	0.0048	0.0110
32	< 0.1	< 0.01	0.0054	1.430	0.0047	0.0125

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.1	0.0153	0.400	0.0282	0.0091	0.0263
2	< 0.1	0.0106	0.248	0.0638	0.0058	0.0198
4	< 0.1	0.0106	0.141	0.0691	0.0075	0.0195
8	< 0.1	< 0.01	0.0301	0.587	0.0114	< 0.01
16	< 0.1	< 0.01	0.0094	0.911	0.0072	0.0126
32	< 0.1	< 0.01	0.0065	2.89	0.0045	0.0576

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.1	0.0129	0.180	0.0484	0.0074	0.0323
2	< 0.1	< 0.01	0.276	0.0806	0.0054	0.0223
4	< 0.1	< 0.01	0.0898	0.038	0.0065	0.0192
8	< 0.1	< 0.01	0.0356	0.151	0.0049	0.0105
16	< 0.1	< 0.01	< 0.005	0.146	< 0.003	< 0.01
32	< 0.1	< 0.01	0.0087	0.0702	< 0.003	0.0112

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.1	0.0135	0.400	0.0173	0.0074	0.0268
2	< 0.1	0.0151	0.261	0.0339	0.007	0.0255
4	< 0.1	0.0118	0.169	0.334	0.02	0.0194
8	< 0.1	< 0.01	0.0718	0.612	0.0258	< 0.01
16	nd	nd	nd	nd	nd	nd
32	< 0.1	< 0.01	0.0164	0.841	0.011	0.0137

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.1	< 0.01	0.333	0.0924	0.0076	0.0112
2	< 0.1	< 0.01	0.234	0.153	0.0102	0.0118
4	< 0.1	< 0.01	0.191	0.0169	< 0.003	< 0.01
8	< 0.1	< 0.01	0.100	0.0549	< 0.003	< 0.01
16	< 0.1	0.0122	< 0.005	0.479	0.003	< 0.01
32	< 0.1	< 0.01	0.0325	2.63	0.0045	< 0.01

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.1	< 0.01	0.365	0.0310	< 0.003	0.0180
2	< 0.1	< 0.01	0.285	0.0426	< 0.003	0.0116
4	< 0.1	< 0.01	0.226	0.0199	< 0.003	0.0107
8	< 0.1	< 0.01	0.114	0.1250	< 0.003	< 0.01
16	< 0.1	< 0.01	0.0223	0.0757	< 0.003	< 0.01
32	< 0.1	< 0.01	0.0163	0.0229	< 0.003	< 0.01

Table B-2: Metals Analysis: Hold - Soil and De-ionized Water
(Concentrations in mg/L)

Detection limits

W	Sn	Zn	Fe	Cu	Pb
0.1	0.01	0.005	0.01	0.003	0.01

De-ionized water

W	Sn	Zn	Fe	Cu	Pb
< 0.1	< 0.01	< 0.005	0.0105	0.004	0.0123

Control

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.1	< 0.01	0.416	0.709	0.0144	0.0482
15	< 0.1	< 0.01	0.307	0.906	0.0119	0.0151
35	< 0.1	< 0.01	0.267	0.779	0.0225	0.0135

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.1	< 0.01	0.448	0.169	< 0.003	0.0613
15	< 0.1	< 0.01	0.324	3.73	0.0071	0.0209
35	< 0.1	0.0244	0.373	6.39	0.0154	0.0331

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.1	< 0.01	0.376	0.159	< 0.003	0.0703
15	< 0.1	< 0.01	0.342	0.747	0.0046	0.0288
35	< 0.1	< 0.01	0.443	0.937	0.008	0.0211

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.1	< 0.01	0.385	0.214	< 0.003	0.0863
15	< 0.1	< 0.01	0.349	0.0542	0.0077	0.0247
35	< 0.1	< 0.01	0.395	0.201	0.0051	0.0169

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.1	< 0.01	0.377	0.0651	< 0.003	0.0613
15	< 0.1	< 0.01	0.317	0.0657	0.005	0.0242
35	< 0.1	< 0.01	0.39	0.923	0.0059	0.0187

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.1	< 0.01	0.326	0.0381	< 0.003	0.0701
15	< 0.1	< 0.01	0.441	0.111	0.0092	0.0265
35	< 0.1	< 0.01	0.575	0.791	0.0089	0.0229

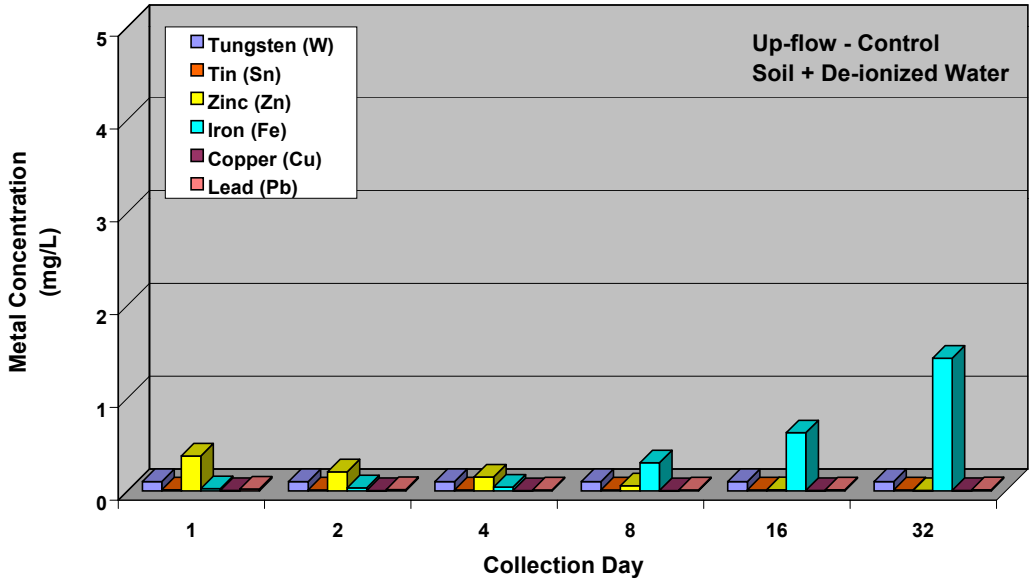

Figure B-1. Metal concentrations in leachate for control sample from the up-flow test using soil and de-ionized water.

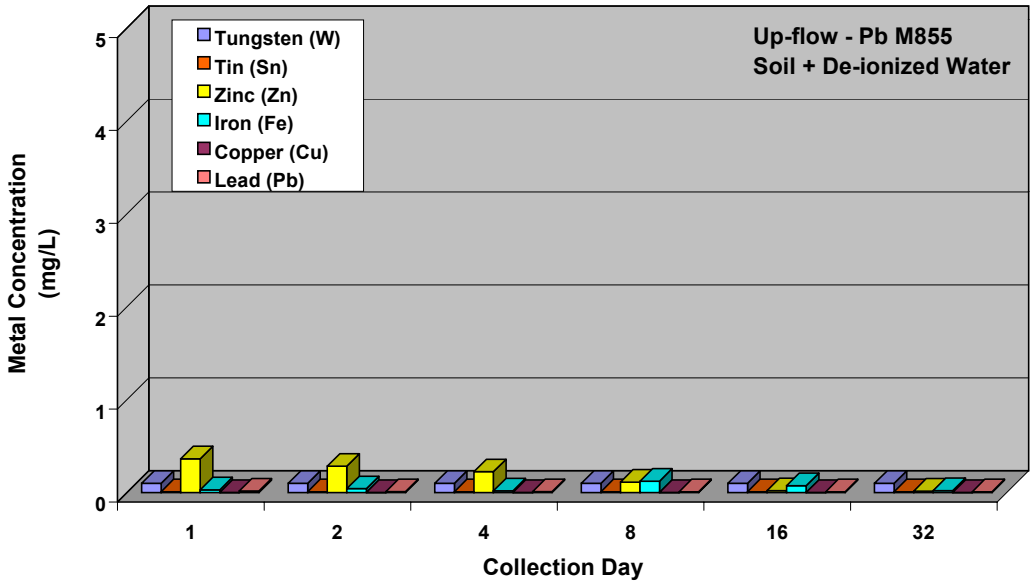

Figure B-2. Metal concentrations in leachate for the lead M855 sample from the up-flow test using soil and de-ionized water.

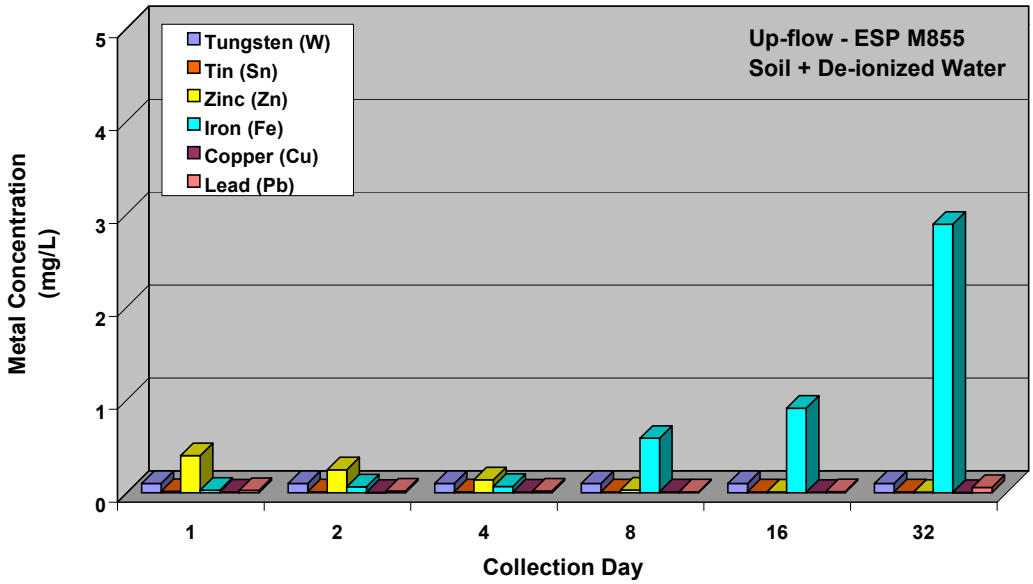

Figure B-3. Metal concentrations in leachate for the ESP M855 sample from the up-flow test using soil and de-ionized water.

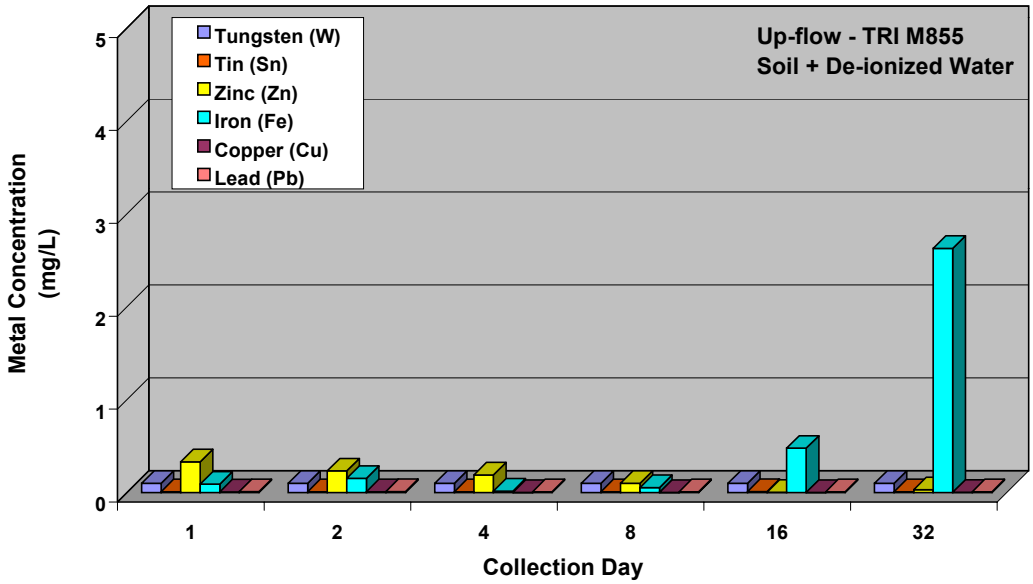

Figure B-4. Metal concentrations in leachate for the TRI M855 sample from the up-flow test using soil and de-ionized water.

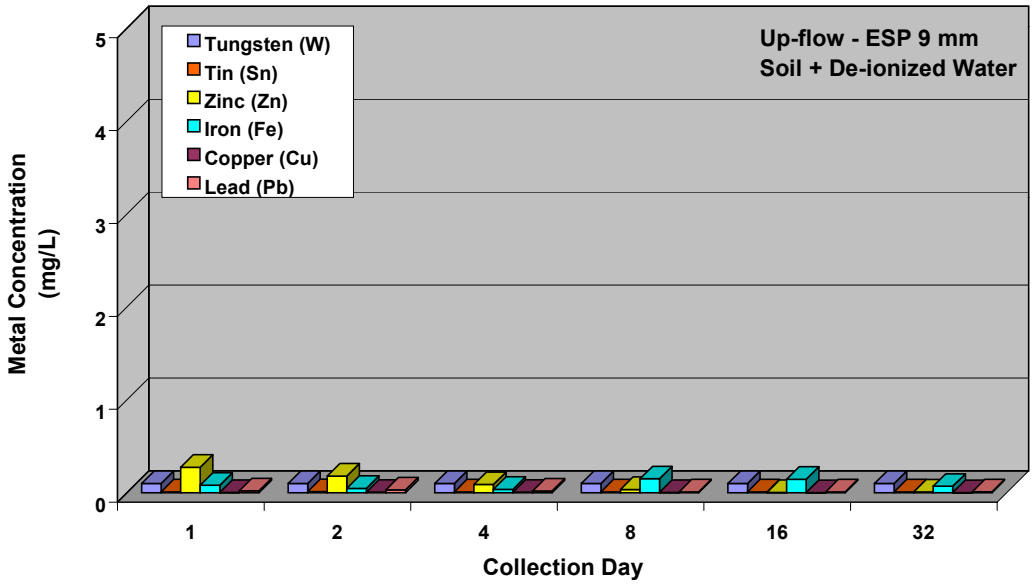

Figure B-5. Metal concentrations in leachate for the ESP 9mm sample from the up-flow test using soil and de-ionized water.

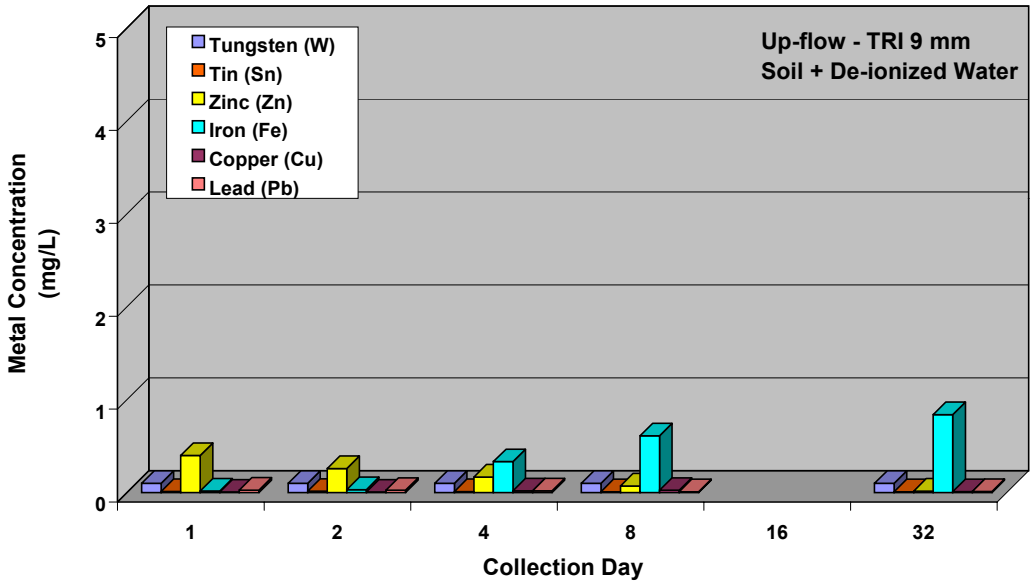

Figure B-6. Metal concentrations in leachate for the TRI 9 mm sample from the up-flow test using soil and de-ionized water.

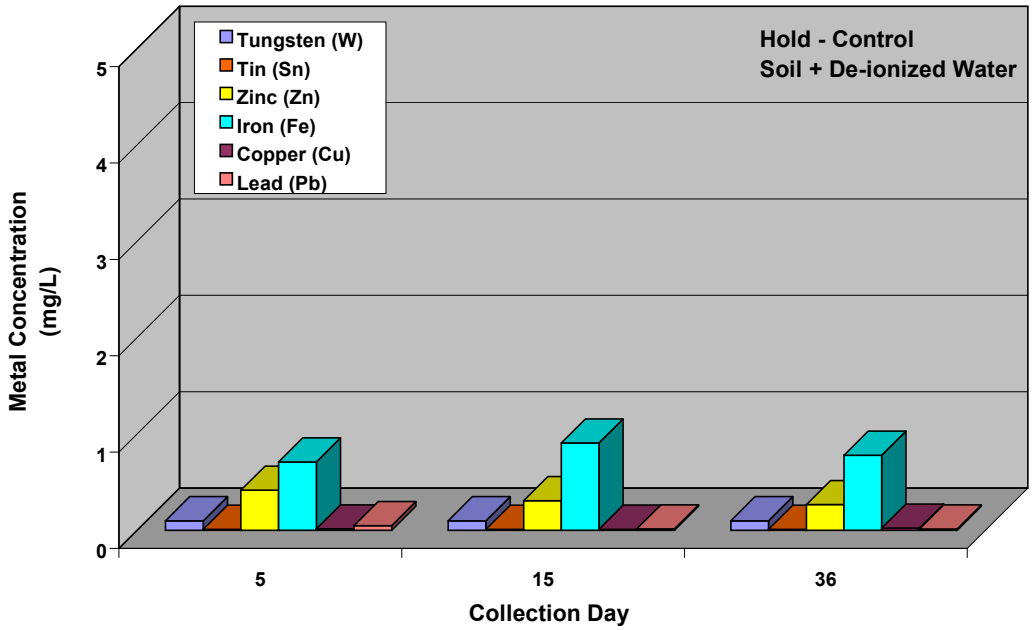

Figure B-7. Metal concentrations in leachate for the control sample from the hold test using soil and de-ionized water.

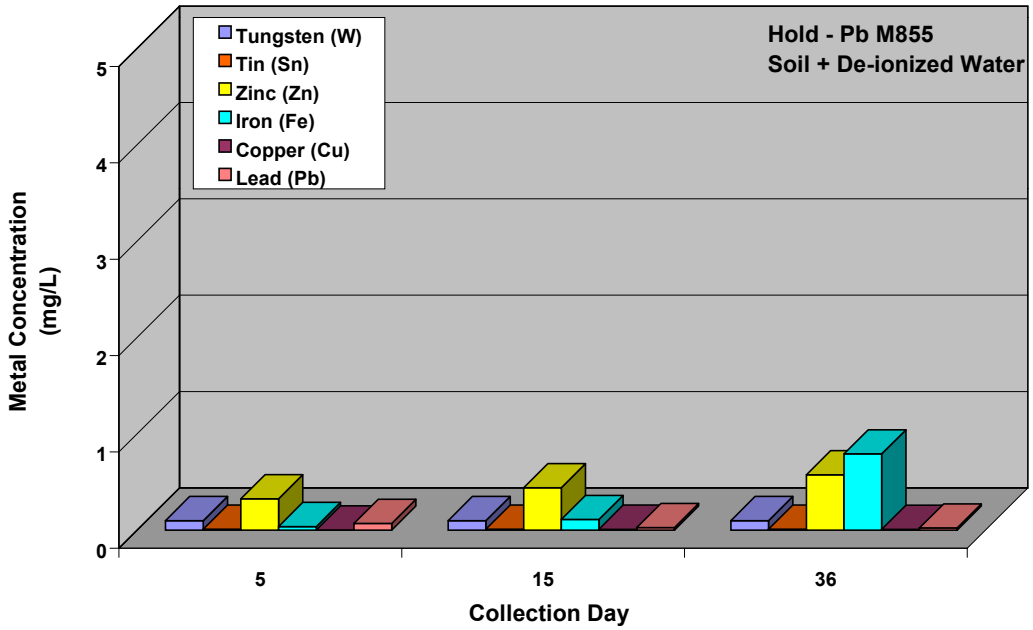

Figure B-8. Metal concentrations in leachate for the lead M855 sample from the hold test using soil and de-ionized water.

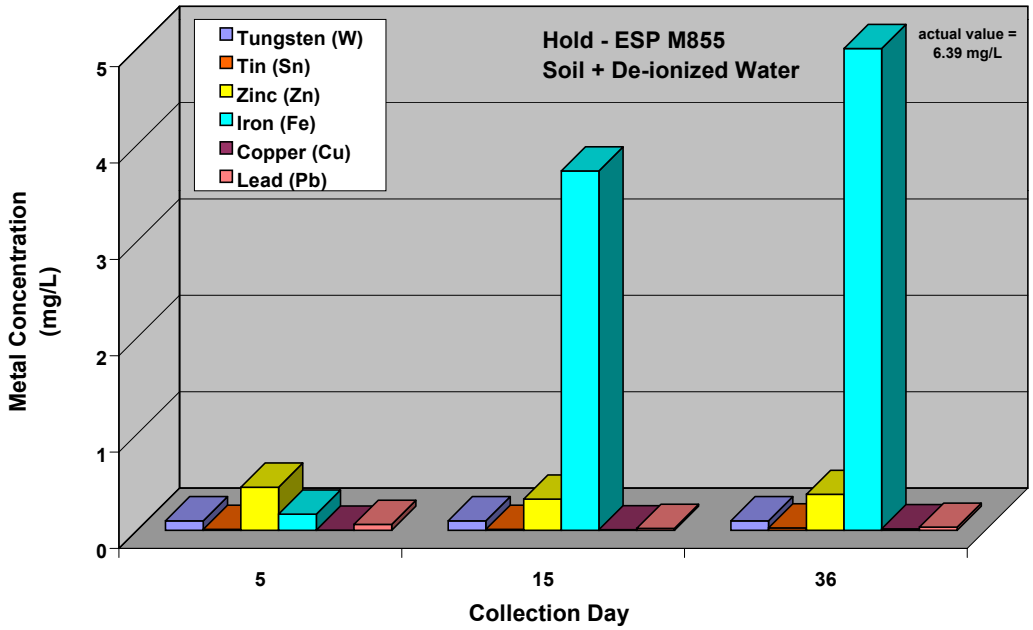

Figure B-9. Metal concentrations in leachate for the ESP M855 sample from the hold test using soil and de-ionized water.

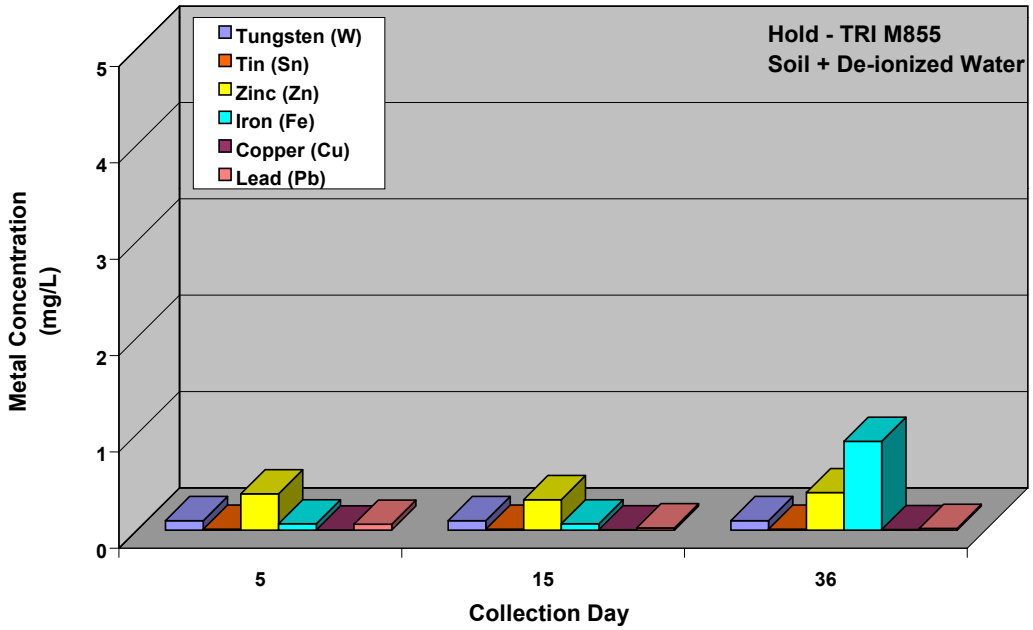

Figure B-10. Metal concentrations in leachate for the TRI M855 sample from the hold test using soil and de-ionized water.

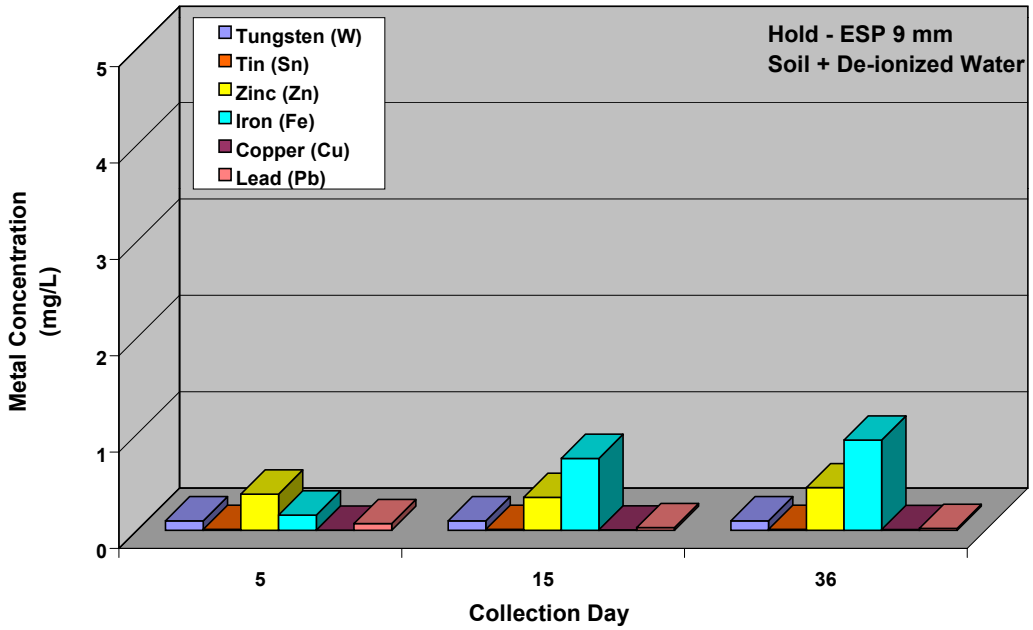

Figure B-11. Metal concentrations in leachate for the ESP 9 mm sample from the hold test using soil and de-ionized water.

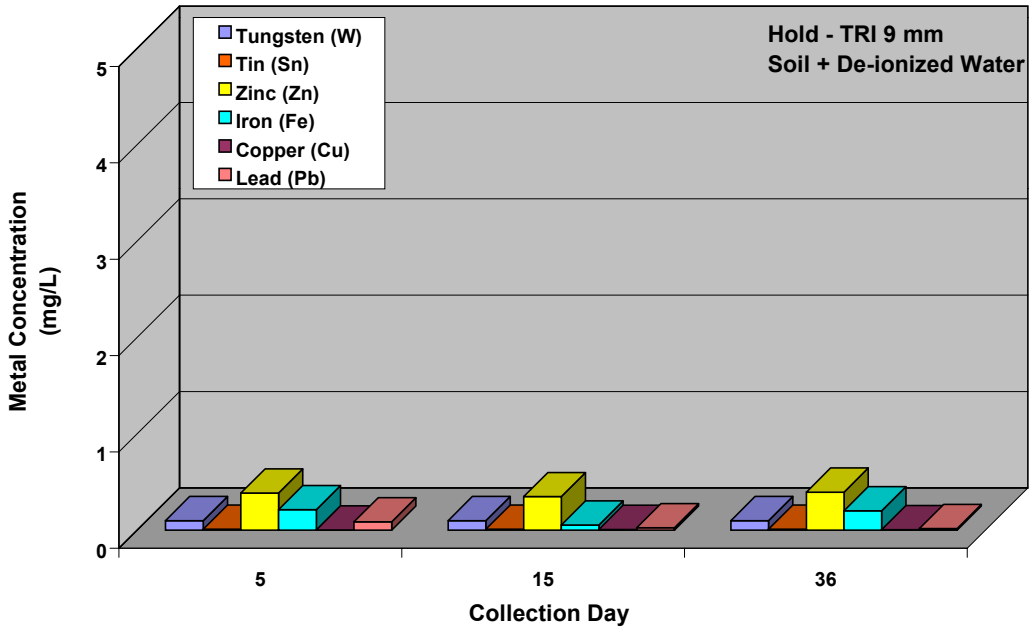

Figure B-12. Metal concentrations in leachate for the TRI 9 mm sample from the hold test using soil and de-ionized water.

STANDARD RUN C. Sand with Simulated Ocean Water

Media: *Sand*
Solvent: *Simulated Ocean Water*
Technique(s): Up-flow and Hold
Bullet types: Lead M855
ESP-1 M855
ESP-1 9 mm FMJ
TRI-1 M855
TRI-1 9 mm FMJ

Summary: Increased tungsten leaching as compared to de-ionized water with definite decrease in concentration with time.

Comments: See A.

Table C-1: Metals Analysis: Up-Flow - Sand + Simulated Ocean Water
(Concentrations in mg/L)

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
1,2,4,8	0.05	0.005	0.005	0.01	0.005	0.05
16,30	0.05	0.02	0.005	0.03	0.02	0.1

Simulated ocean water

W	Sn	Zn	Fe	Cu	Pb
< 0.05	< 0.02	< 0.005	< 0.01	0.0065	< 0.05

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.005	0.0169	0.115	0.005	< 0.05
2	0.05	< 0.005	0.0086	< 0.01	0.0065	< 0.05
4	< 0.05	< 0.005	< 0.005	0.168	< 0.005	< 0.05
8	< 0.05	< 0.005	< 0.005	0.120	< 0.005	< 0.05
16	< 0.05	< 0.02	0.033	0.217	0.045	< 0.1
30	< 0.05	< 0.02	0.025	0.232	0.043	< 0.1

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	7.0	< 0.005	0.0051	0.401	0.0065	< 0.05
2	5.7	< 0.005	< 0.005	0.0106	0.0056	< 0.05
4	3.3	< 0.005	< 0.005	< 0.01	0.0095	< 0.05
8	2.8	< 0.005	< 0.005	< 0.01	0.0059	< 0.05
16	1.1	< 0.02	0.022	0.203	0.036	0.12
30	0.8	< 0.02	0.026	0.209	0.042	0.13

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	6.4	< 0.005	0.0139	0.0115	0.0105	< 0.05
2	6.2	< 0.005	0.0103	0.0258	0.0113	< 0.05
4	4.9	< 0.005	0.0104	< 0.01	0.0118	< 0.05
8	3.7	< 0.005	< 0.005	0.113	0.0082	< 0.05
16	1.5	< 0.02	0.021	0.222	0.043	0.15
30	0.7	< 0.02	0.027	0.219	0.045	0.11

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	66.0	< 0.005	0.0125	0.0107	0.0091	< 0.05
2	30.0	< 0.005	< 0.005	< 0.01	< 0.005	< 0.05
4	27.7	< 0.005	< 0.005	0.0149	< 0.005	< 0.05
8	20.1	< 0.005	< 0.005	< 0.01	< 0.005	< 0.05
16	4.4	< 0.02	0.029	0.214	0.038	< 0.1
30	2.7	< 0.02	0.021	0.200	0.039	< 0.1

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	51.6	< 0.005	0.0084	< 0.01	0.0058	< 0.05
2	34.4	< 0.005	0.0061	< 0.01	0.0067	< 0.05
4	22.0	< 0.005	< 0.005	0.0151	0.0083	< 0.05
8	18.7	< 0.005	< 0.005	0.205	0.0056	< 0.05
16	11.8	< 0.02	0.046	0.219	0.043	< 0.1
30	3.4	< 0.02	0.036	0.169	0.040	< 0.1

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.005	0.0129	0.0188	< 0.005	< 0.05
2	< 0.05	< 0.005	0.0123	0.0744	< 0.005	< 0.05
4	< 0.05	< 0.005	0.0255	0.0717	< 0.005	< 0.05
8	< 0.05	< 0.005	0.01	< 0.01	< 0.005	< 0.05
16	< 0.05	< 0.02	0.027	0.164	0.040	< 0.1
30	< 0.05	< 0.02	0.025	0.162	0.039	< 0.1

Table C-2: Metals Analysis: Hold - Sand + Simulated Ocean Water
(Concentrations in mg/L)

Instant ocean

W	Sn	Zn	Fe	Cu	Pb
< 0.05	< 0.02	< 0.005	< 0.01	0.0091	< 0.05

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
5	0.05	0.005	0.005	0.01	0.005	0.05
20,35	0.05	0.02	0.005	0.03	0.02	0.1

Control

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.02	0.0201	< 0.01	0.0154	< 0.05
15	< 0.05	< 0.02	0.061	0.19	0.046	< 0.1
35	< 0.05	< 0.02	0.068	0.185	0.042	< 0.1

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	2.71	< 0.02	0.0273	< 0.01	0.0136	< 0.05
15	1.64	< 0.02	0.057	0.181	0.047	< 0.1
35	1.27	< 0.02	0.06	0.178	0.047	< 0.1

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	9.15	< 0.02	0.0114	0.0776	0.018	< 0.05
15	4.86	< 0.02	0.037	0.225	0.046	< 0.1
35	2.15	< 0.02	0.057	0.189	0.04	< 0.1

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	45.9	< 0.02	0.0378	< 0.01	0.0117	< 0.05
15	25.9	< 0.02	0.065	0.168	0.045	< 0.1
35	8.83	< 0.02	0.069	0.169	0.045	< 0.1

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	36.1	< 0.02	0.0385	< 0.01	0.0119	< 0.05
15	19.6	< 0.02	0.085	0.188	0.036	< 0.1
35	15.6	< 0.02	0.086	0.184	0.044	< 0.1

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.02	0.0249	< 0.01	0.0137	< 0.05
15	< 0.05	< 0.02	0.044	0.184	0.043	< 0.1
35	< 0.05	< 0.02	0.056	0.211	0.044	< 0.1

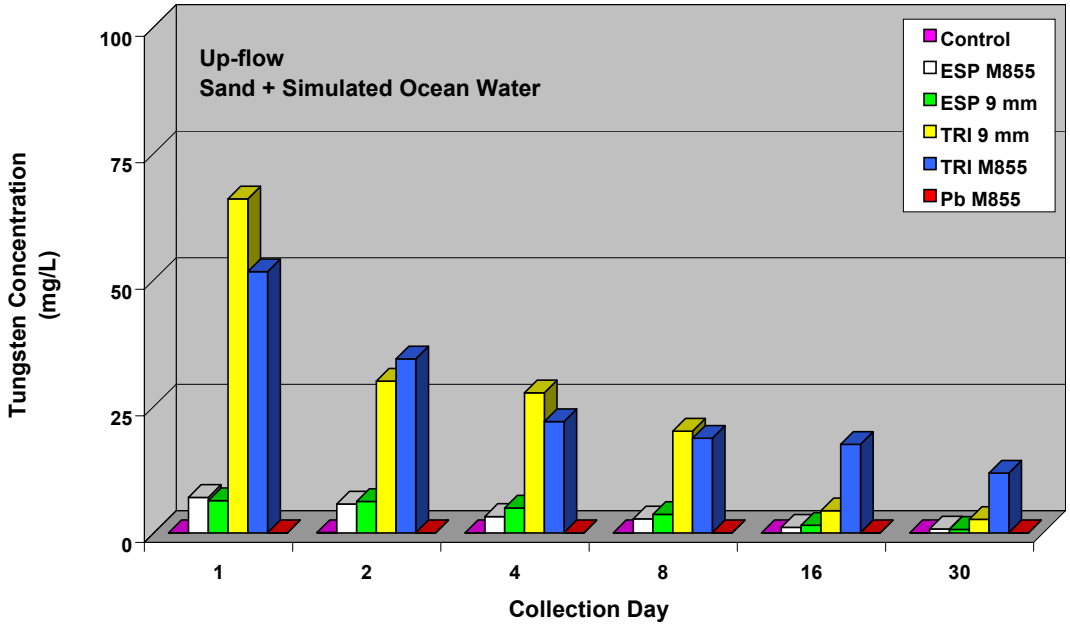

Figure C-1. Tungsten concentration in leachate for up-flow test using sand and ocean water.

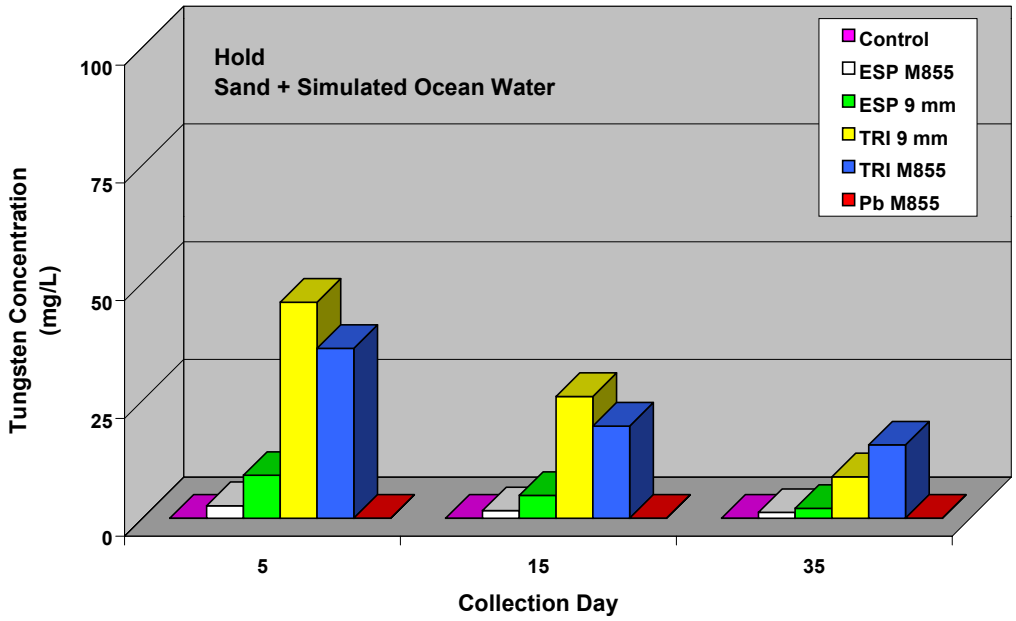

Figure C-2. Tungsten concentration in leachate for hold test using sand and ocean water

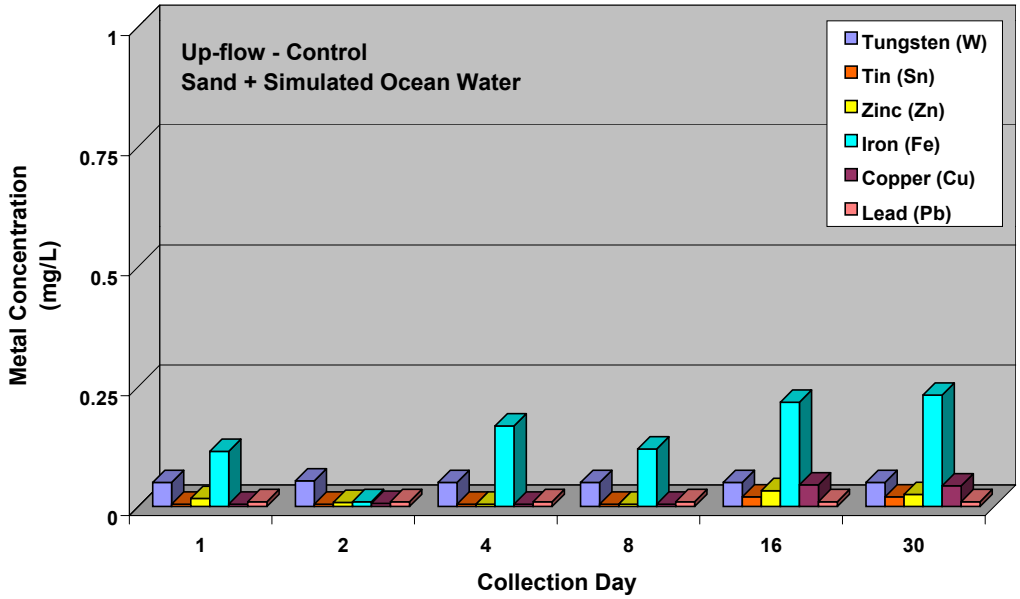

Figure C-3. Metal concentrations in leachate for control sample from the up-flow test using sand and ocean water.

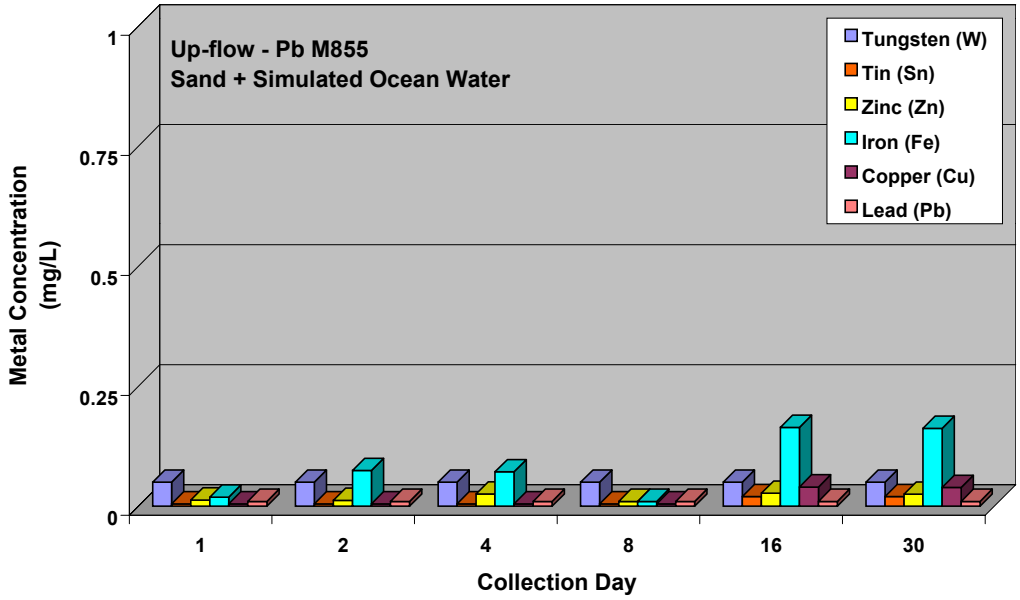

Figure C-4. Metal concentrations in leachate for lead M855 sample from the up-flow test using sand and ocean water.

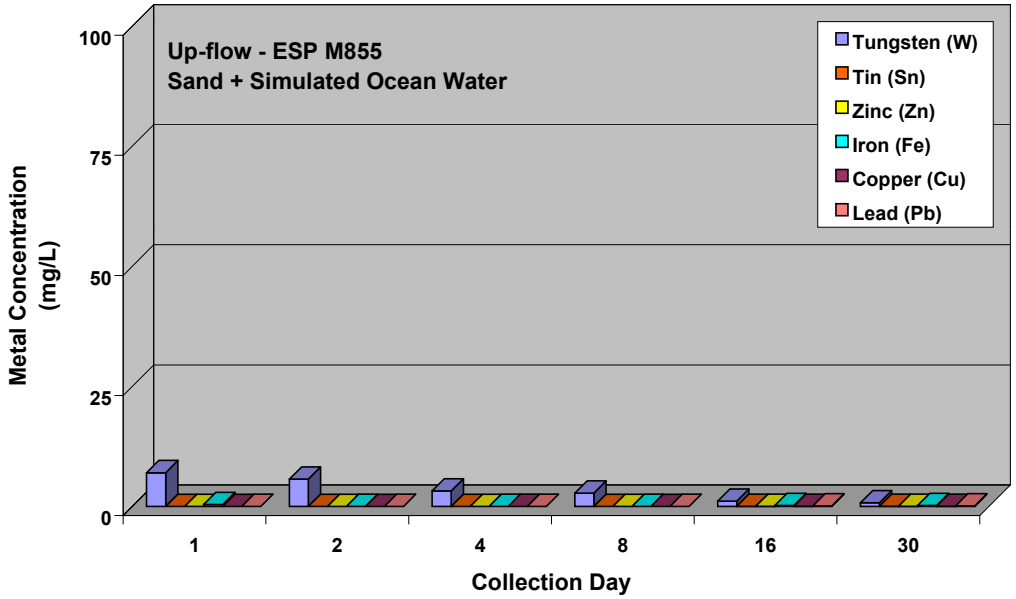

Figure C-5. Metal concentrations in leachate for ESP M855 sample from the up-flow test using sand and ocean water.

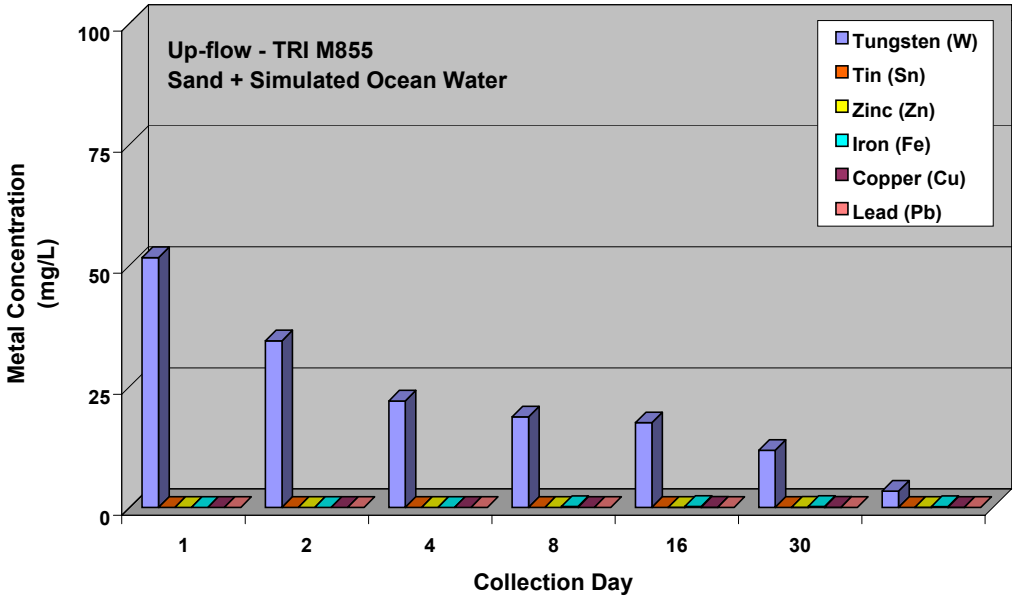

Figure C-6. Metal concentrations in leachate for TRI M855 sample from the up-flow test using sand and ocean water.

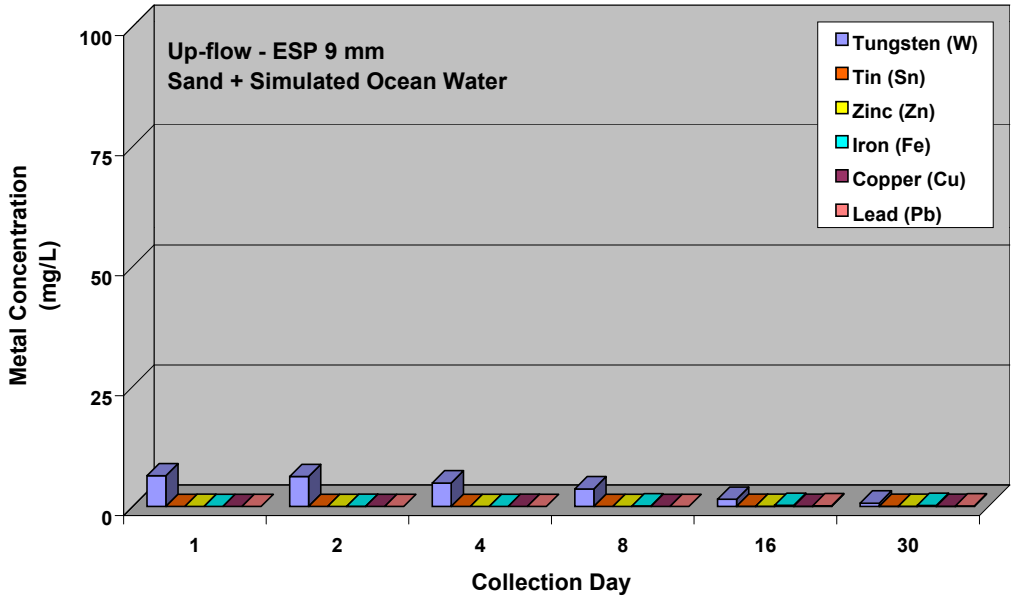

Figure C-7. Metal concentrations in leachate for ESP 9 mm sample from the up-flow test using sand and ocean water.

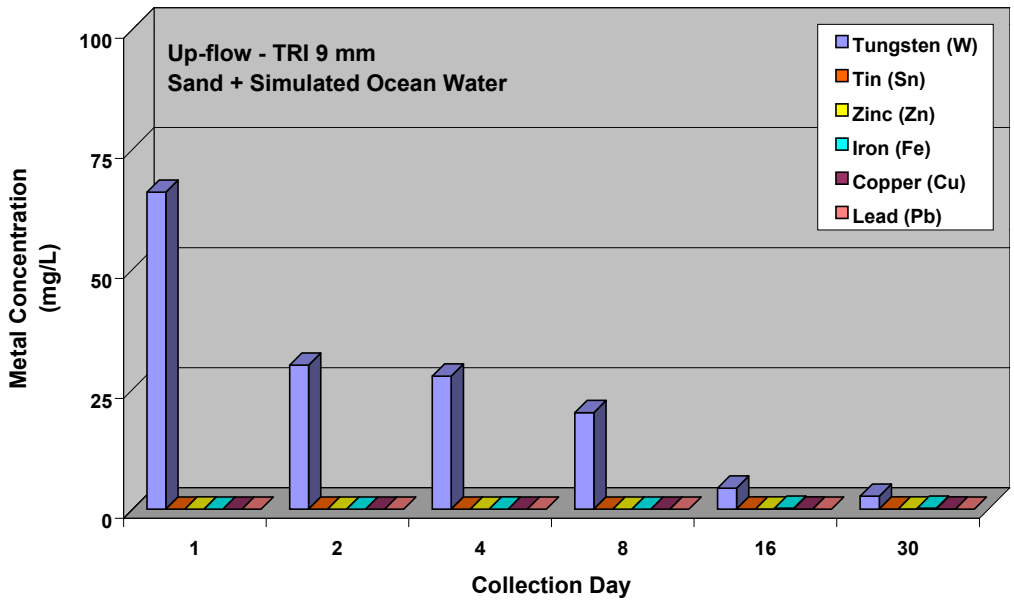

Figure C-8. Metal concentrations in leachate for TRI 9 mm sample from the up-flow test using sand and ocean water.

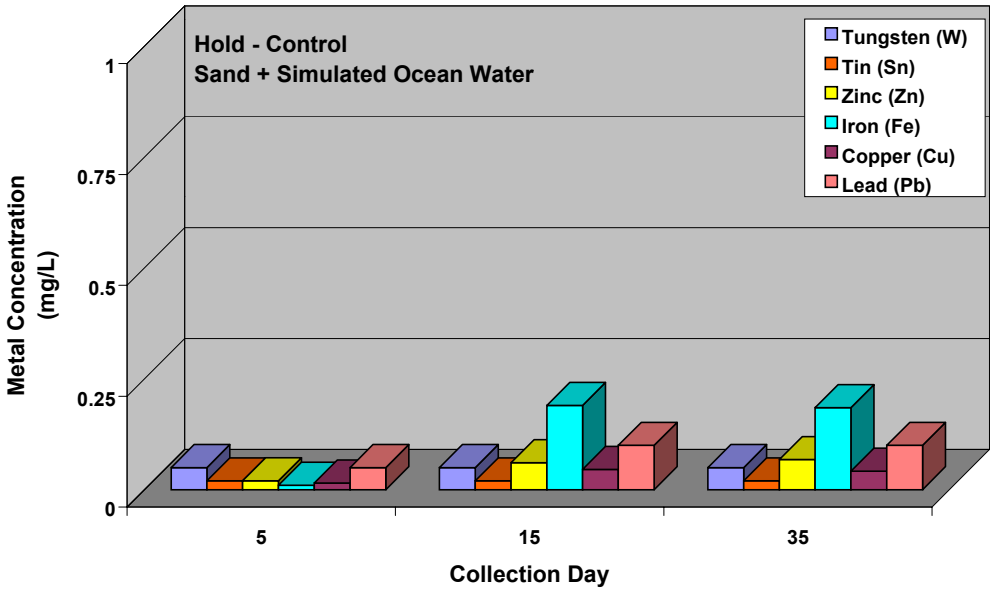

Figure C-9. Metal concentrations in leachate for control sample from the hold test using sand and ocean water.

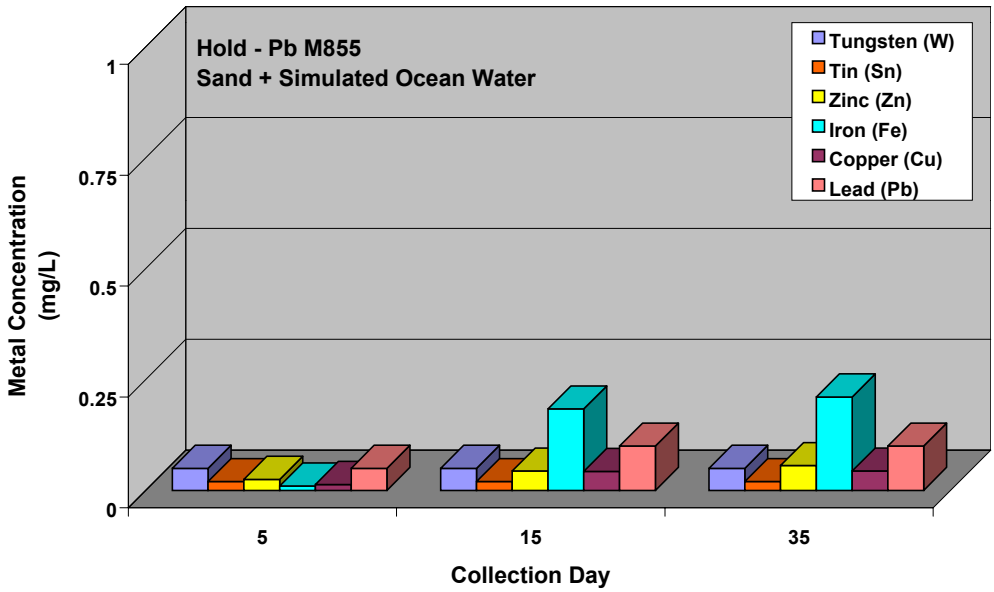

Figure C-10. Metal concentrations in leachate for lead M855 sample from the hold test using sand and ocean water

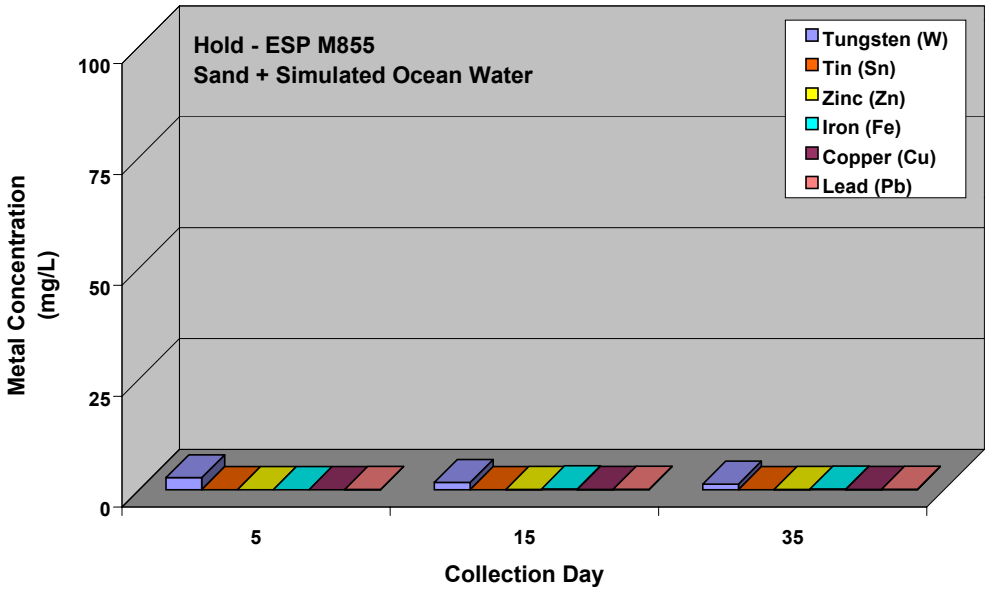

Figure C-11. Metal concentrations in leachate for ESP M855 sample from the hold test using sand and ocean water

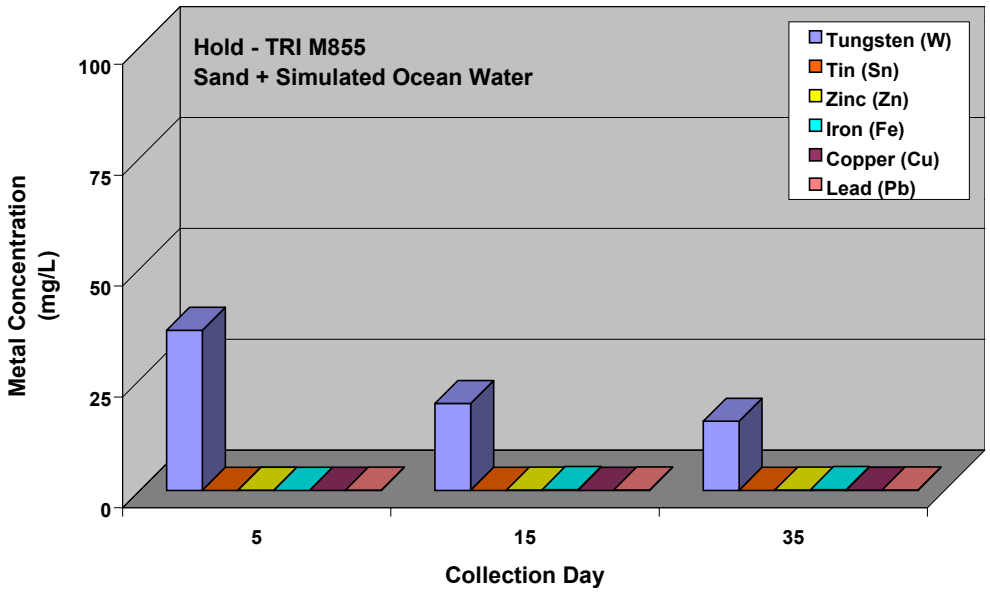

Figure C-12. Metal concentrations in leachate for TRI M855 sample from the hold test using sand and ocean water

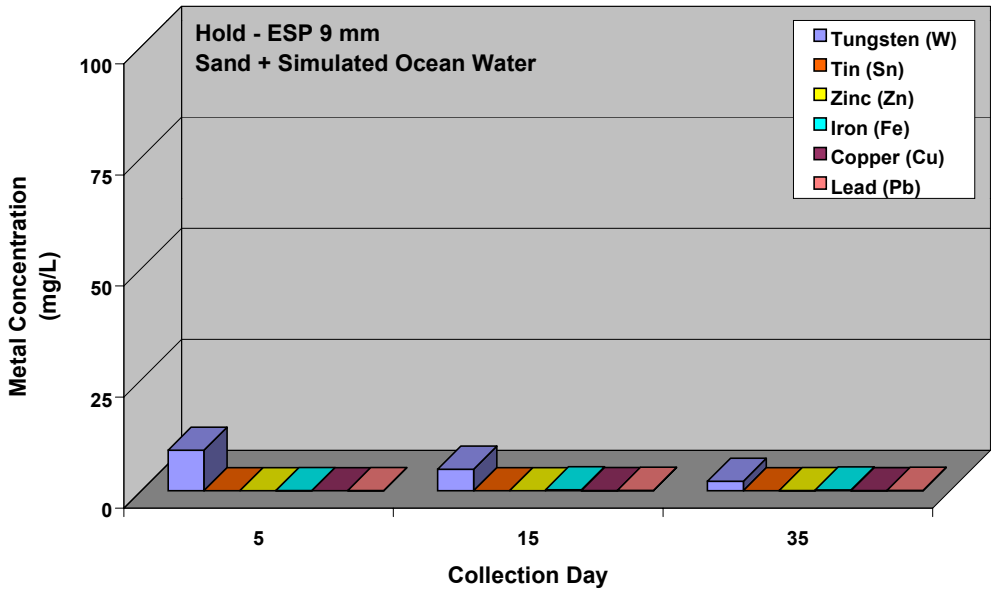

Figure C-13. Metal concentrations in leachate for ESP 9 mm sample from the hold test using sand and ocean water

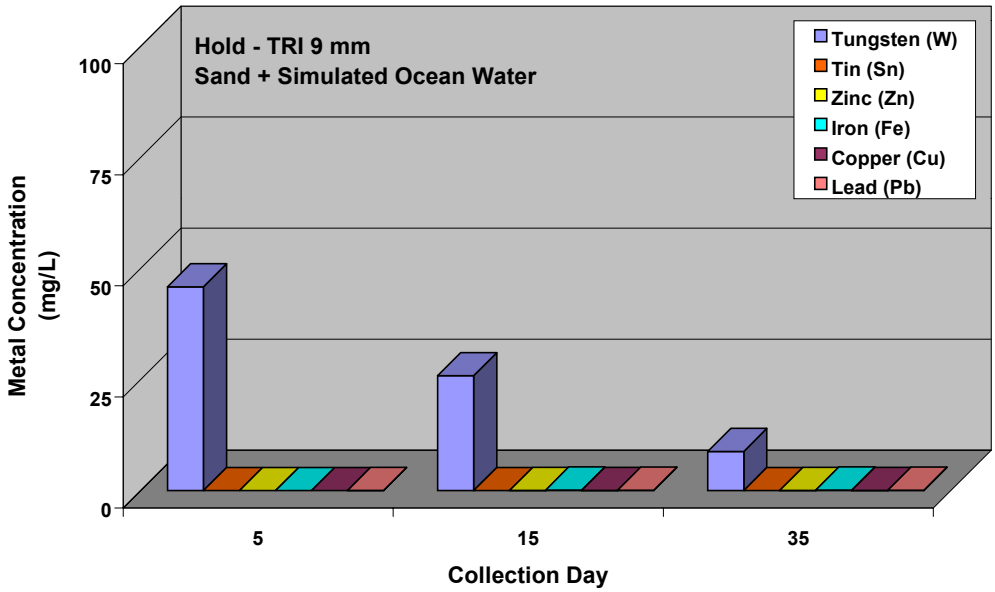

Figure C-14. Metal concentrations in leachate for TRI 9 mm sample from the hold test using sand and ocean water

STANDARD RUN D. Sand with Simulated Rain Water

Media: *Sand*
Solvent: *Rain water*
Technique(s): Up-flow and Hold
Bullet types: Lead M855
ESP-1 M855
ESP-1 9 mm FMJ
TRI-1 M855
TRI-1 9 mm FMJ

Summary: Increased tungsten leaching as compared to de-ionized water with definite decrease in concentration with time

Comments: See A.

Table D-1: Metals Analysis: Up-Flow - Sand + Rain Water
(Concentrations in mg/L)

Detection limits

	W	Sn	Zn	Fe	Cu	Pb
	0.05	0.05	0.005	0.03	0.02	0.01

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.05	0.028	< 0.03	< 0.02	< 0.01
2	< 0.05	< 0.05	0.018	< 0.03	< 0.02	< 0.01
4	< 0.05	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
8	< 0.05	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
16	< 0.05	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
32	< 0.05	< 0.05	0.0099	< 0.03	< 0.02	< 0.01

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	13.0	< 0.05	0.015	< 0.03	< 0.02	< 0.01
2	10.0	< 0.05	0.013	< 0.03	< 0.02	< 0.01
4	5.3	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
8	6.2	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
16	9.2	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
32	2.4	< 0.05	0.025	0.0355	< 0.02	0.0291

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	18.0	< 0.05	0.019	< 0.03	< 0.02	< 0.01
2	13.0	< 0.05	0.014	< 0.03	< 0.02	< 0.01
4	6.4	< 0.05	0.01	< 0.03	< 0.02	< 0.01
8	7.0	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
16	9.2	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
32	2.2	< 0.05	0.0248	0.0181	< 0.02	0.0328

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	48.0	< 0.05	0.02	< 0.03	< 0.02	< 0.01
2	20.0	< 0.05	0.016	< 0.03	< 0.02	< 0.01
4	8.6	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
8	10.0	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
16	15.0	< 0.05	0.011	< 0.03	< 0.02	< 0.01
32	1.9	< 0.05	0.0226	0.0244	< 0.02	0.032

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	50.0	< 0.05	0.02	< 0.03	< 0.02	< 0.01
2	23.0	< 0.05	0.016	< 0.03	< 0.02	< 0.01
4	10.0	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
8	12.0	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
16	15.0	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
32	1.9	< 0.05	0.0361	0.0323	< 0.02	0.035

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.05	0.015	< 0.03	< 0.02	< 0.01
2	< 0.05	< 0.05	0.015	< 0.03	< 0.02	< 0.01
4	< 0.05	< 0.05	0.011	< 0.03	< 0.02	< 0.01
8	< 0.05	< 0.05	< 0.005	< 0.03	< 0.02	< 0.01
16	< 0.05	< 0.05	0.013	< 0.03	< 0.02	< 0.01
32	< 0.05	< 0.05	0.0352	0.0319	< 0.02	0.0358

Table D-2: Metals Analysis: Hold - Sand + Simulated Rain Water
(Concentrations in mg/L)

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
5,15	5	0.05	0.005	0.03	0.02	0.1
35	0.05	0.02	0.005	0.03	0.02	0.01

Control

Day	W	Sn	Zn	Fe	Cu	Pb
5	5	0.05	0.046	0.03	0.02	0.1
15	5	0.05	0.031	0.03	0.02	0.1
35	0.05	0.0221	0.129	0.0452	0.02	0.0444

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	12	< 0.05	0.043	< 0.03	< 0.02	< 0.1
15	5.7	< 0.05	0.017	< 0.03	< 0.02	< 0.1
35	2.2	0.0213	0.0872	0.0574	< 0.02	0.0154

ESP-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	18	< 0.05	0.04	< 0.03	< 0.02	< 0.1
15	13	< 0.05	0.023	< 0.03	< 0.02	< 0.1
35	5.01	< 0.02	0.0491	0.061	< 0.02	0.0177

TRI-1 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
5	36	< 0.05	0.043	< 0.03	< 0.02	< 0.1
15	33	< 0.05	0.026	< 0.03	< 0.02	< 0.1
35	7.3	< 0.02	0.079	0.0942	< 0.02	0.0206

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	38	< 0.05	0.034	< 0.03	< 0.02	< 0.1
15	35	< 0.05	0.065	< 0.03	< 0.02	< 0.1
35	2.41	< 0.02	0.0585	0.0547	< 0.02	0.021

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.037	< 0.03	< 0.02	< 0.1
15	< 5	< 0.05	0.04	< 0.03	< 0.02	< 0.1
35	0.061	< 0.02	0.129	0.0506	< 0.02	0.0164

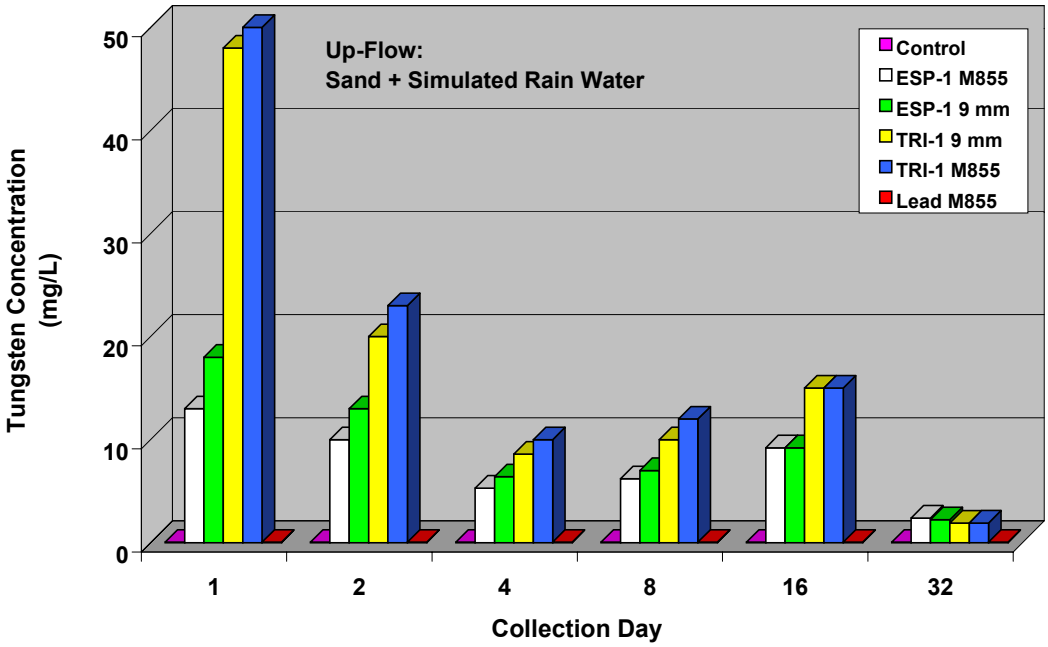

Figure D-1. Tungsten concentration in leachate for up-flow test using sand and rain water.

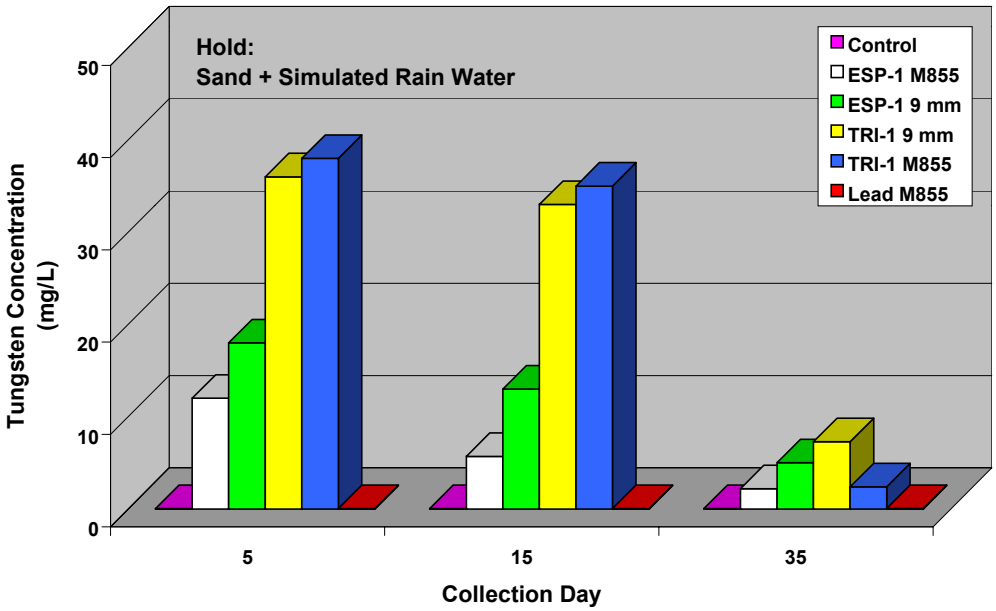

Figure D-2. Tungsten concentration in leachate for hold test using sand and rain water

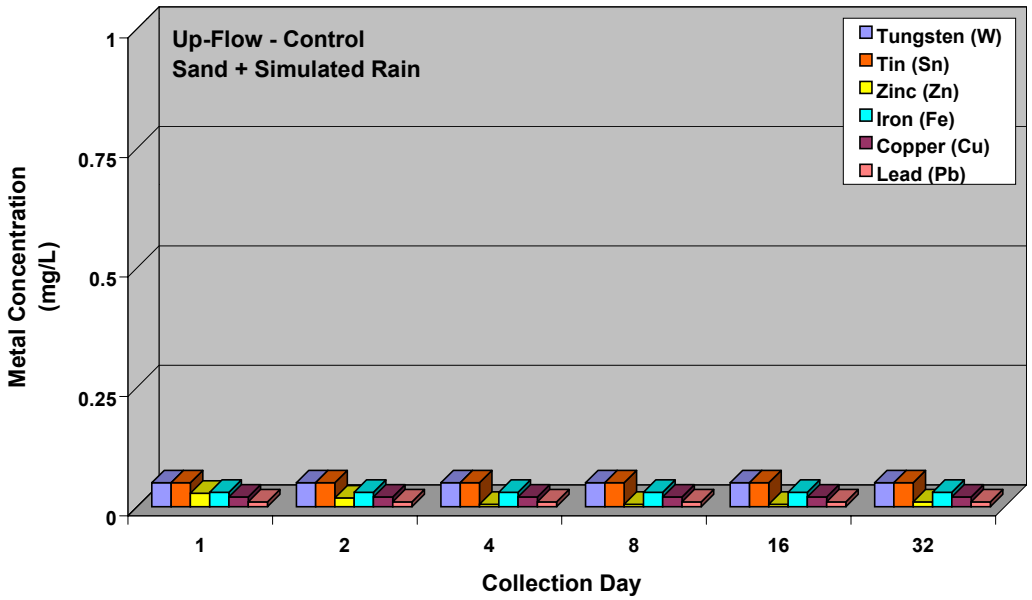

Figure D-3. Metal concentrations in leachate for control sample from the up-flow test using sand and rain water.

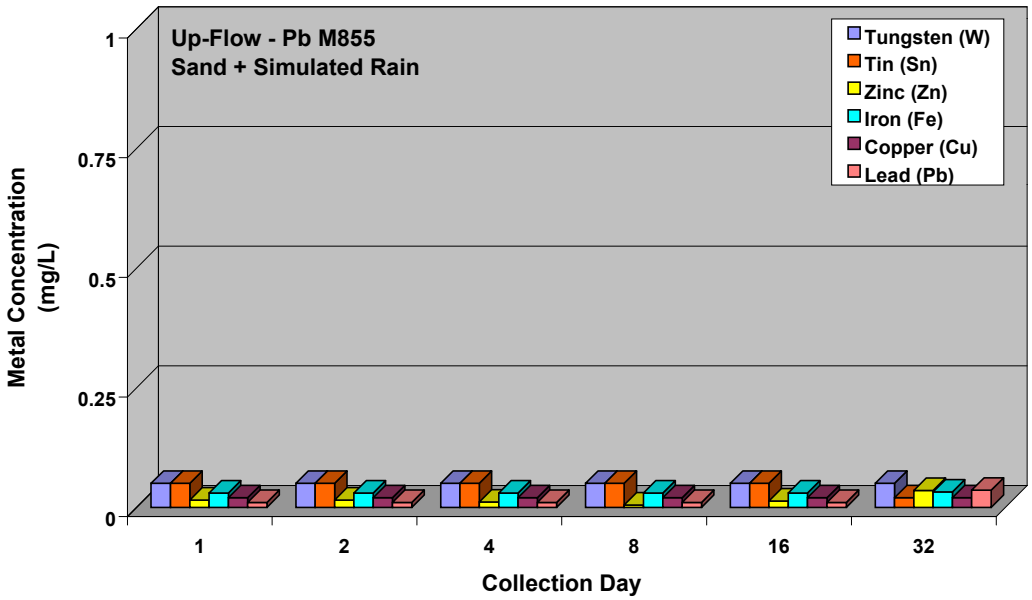

Figure D-4. Metal concentrations in leachate for lead M855 sample from the up-flow test using sand and rain water.

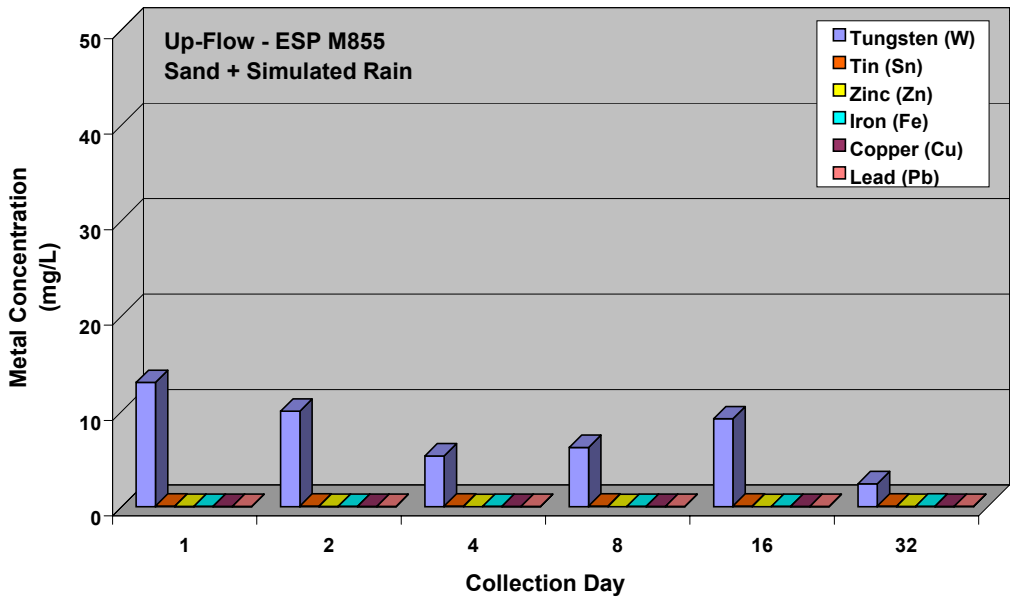

Figure D-5. Metal concentrations in leachate for ESP M855 sample from the up-flow test using sand and rain water.

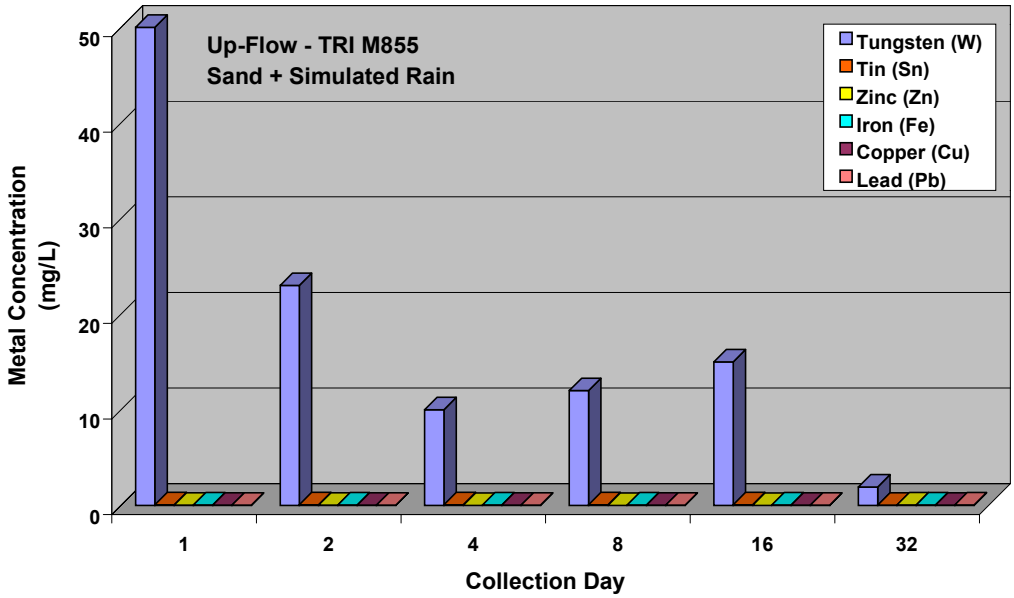

Figure D-6. Metal concentrations in leachate for TRI M855 sample from the up-flow test using sand and rain water

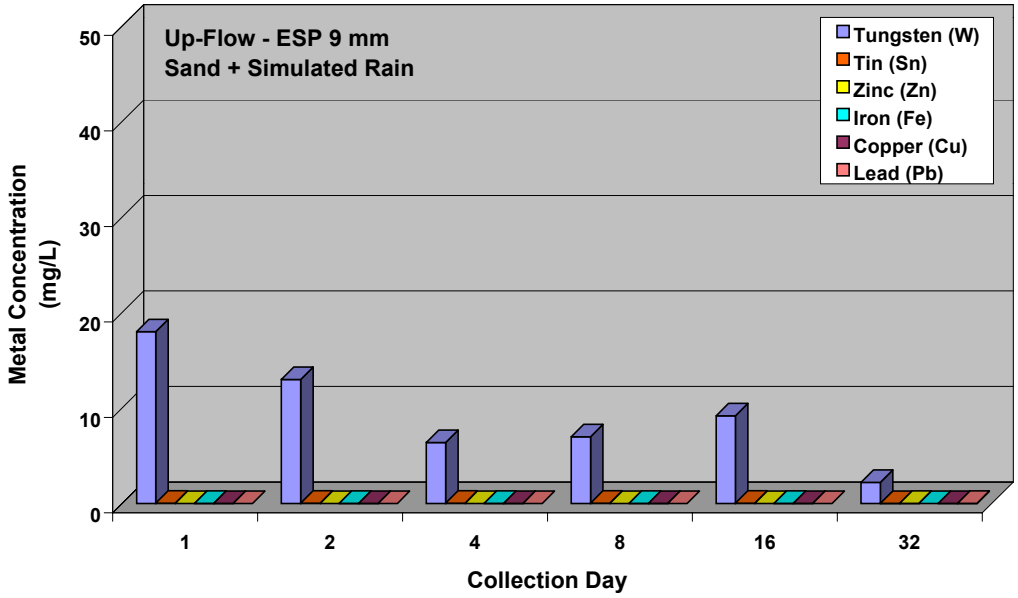

Figure D-7. Metal concentrations in leachate for ESP 9 mm sample from the up-flow test using sand and rain water.

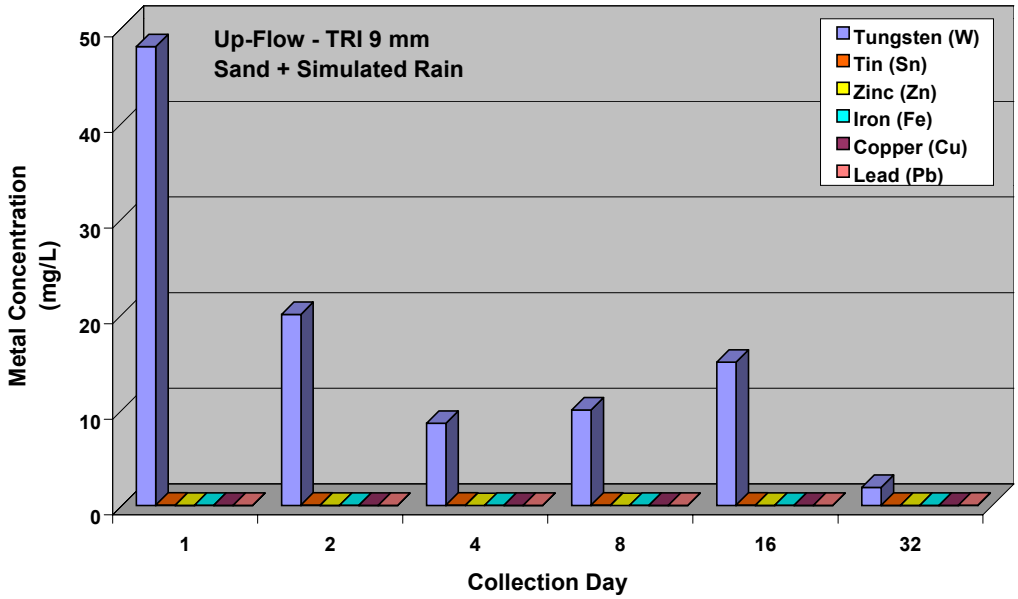

Figure D-8. Metal concentrations in leachate for TRI 9 mm sample from the up-flow test using sand and rain water.

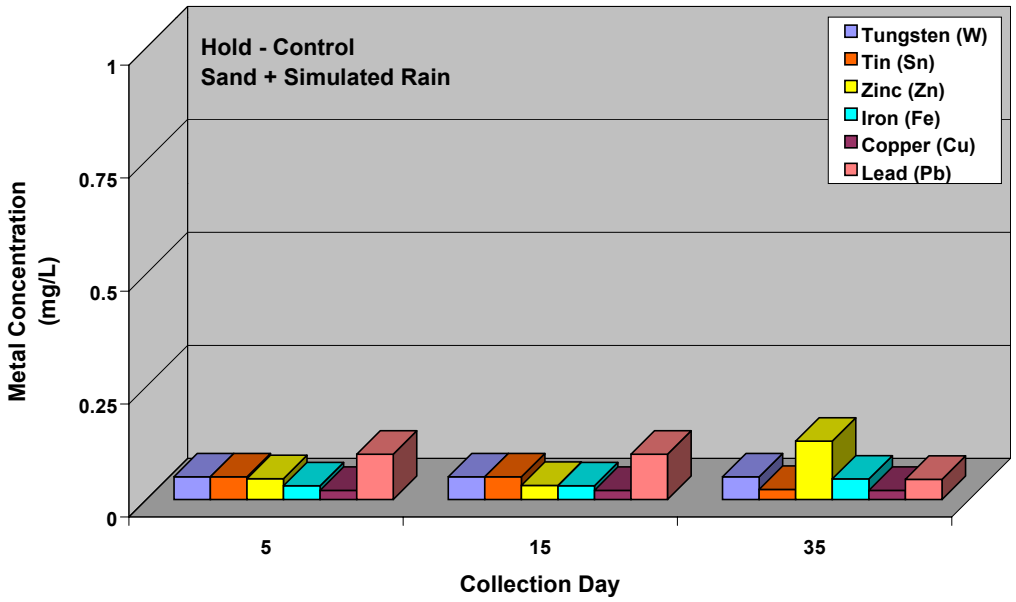

Figure D-9. Metal concentrations in leachate for control sample from the hold test using sand and rain water.

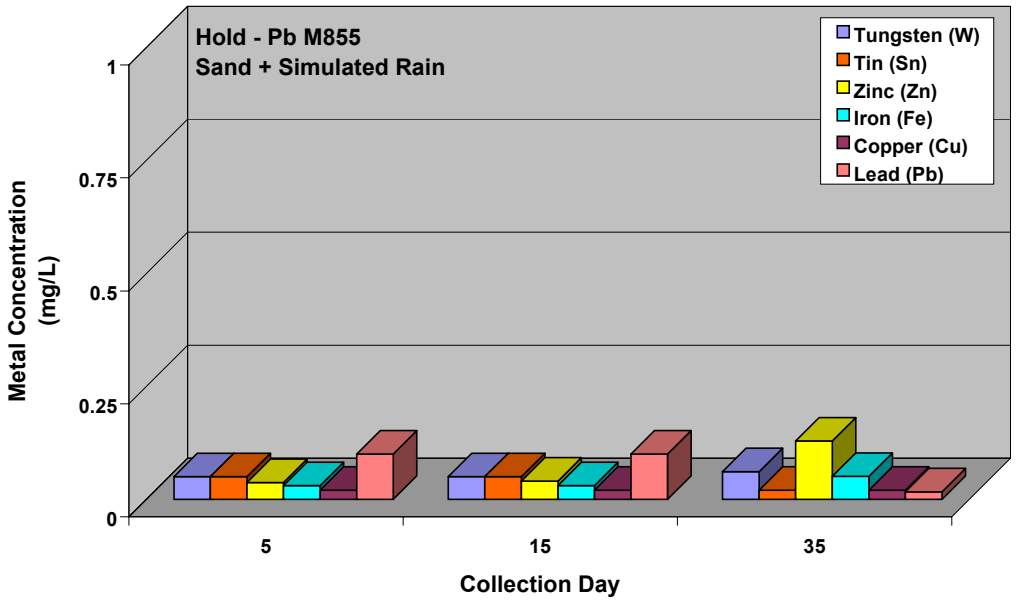

Figure D-10. Metal concentrations in leachate for lead M855 sample from the hold test using sand and rain water.

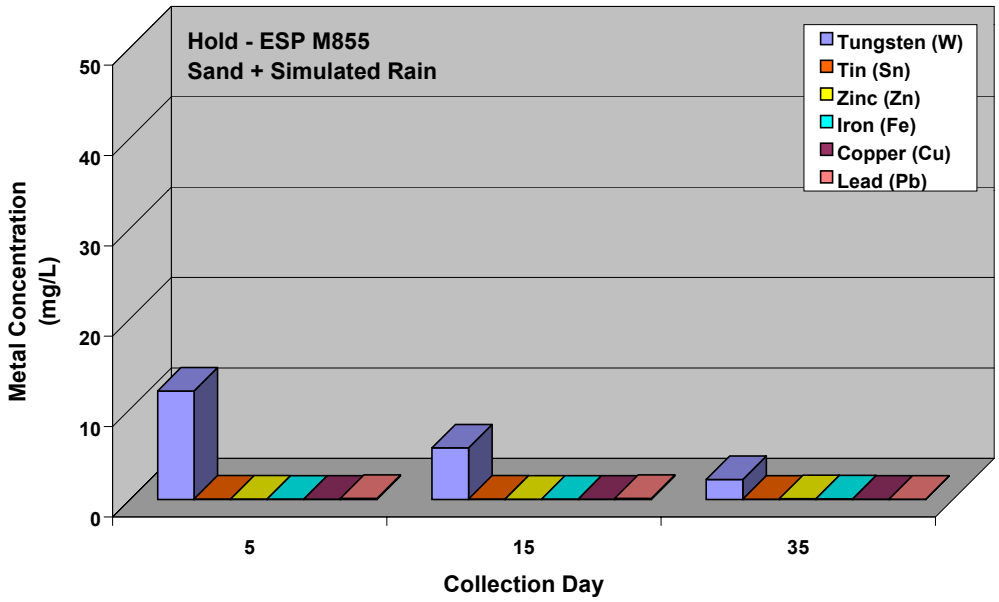

Figure D-11. Metal concentrations in leachate for ESP M855 sample from the hold test using sand and rain water.

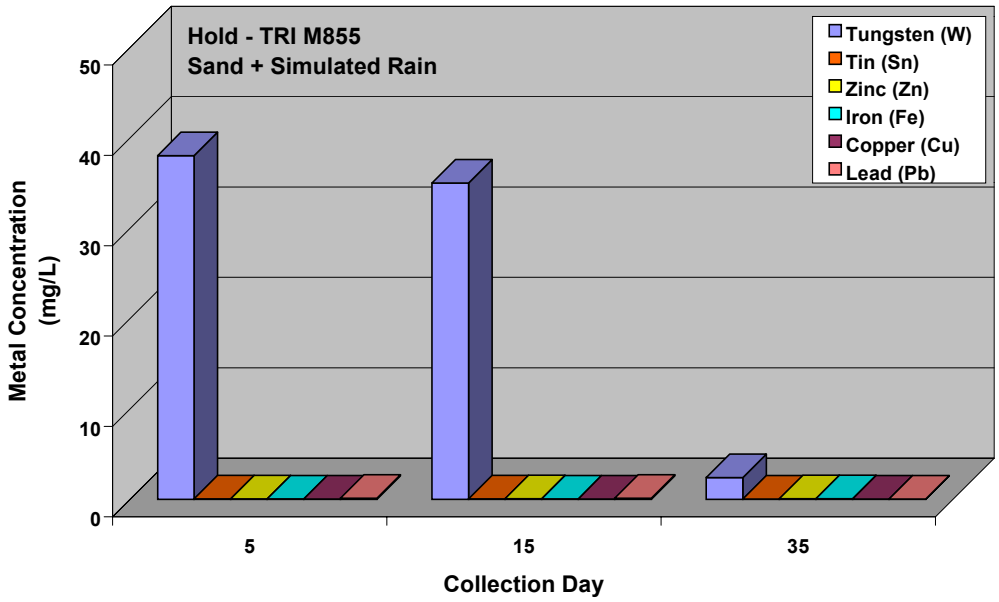

Figure D-12. Metal concentrations in leachate for TRI M855 sample from the hold test using sand and rain water.

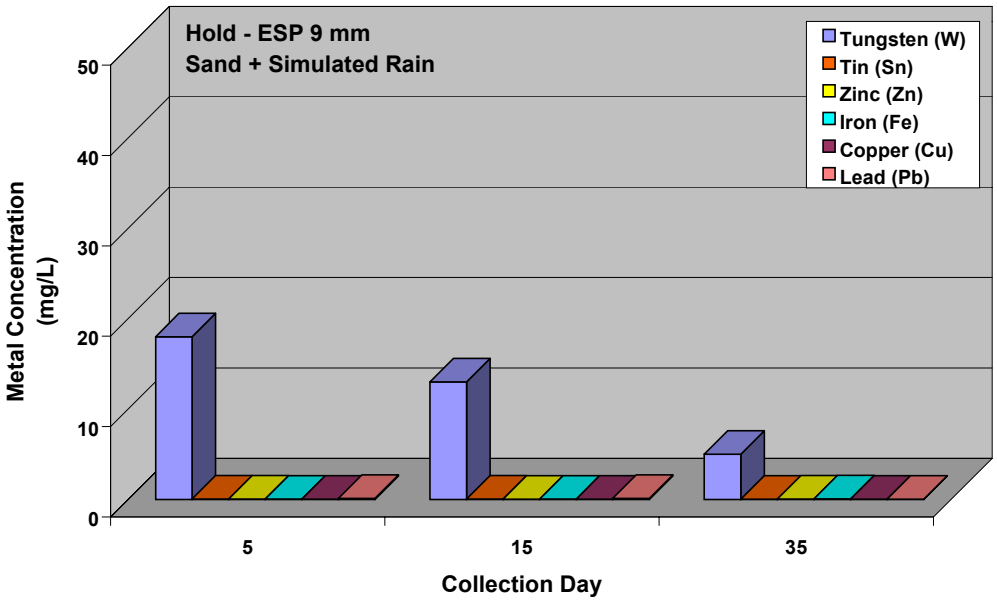

Figure D-13. Metal concentrations in leachate for ESP 9 mm sample from the hold test using sand and rain water.

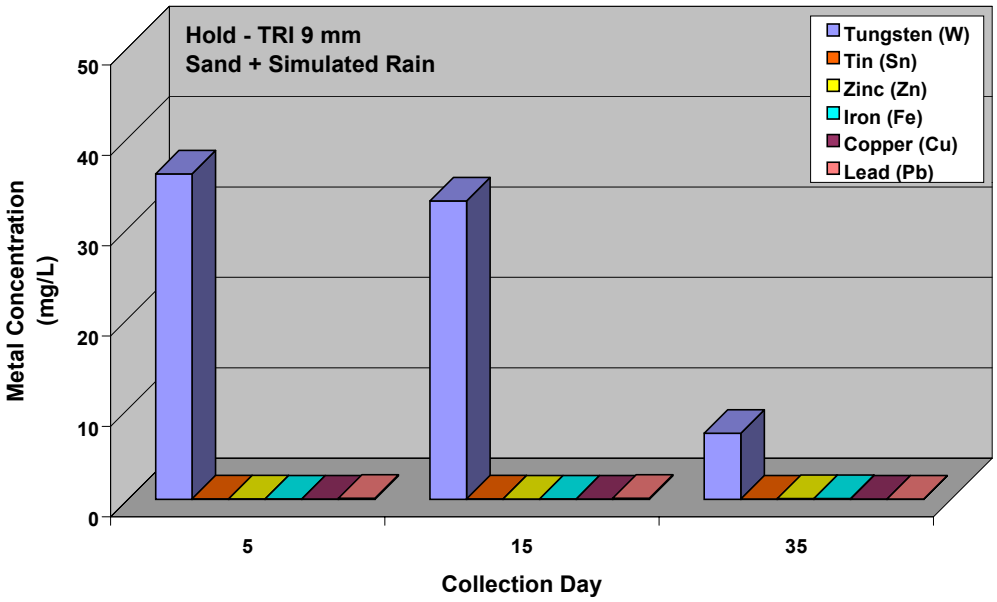

Figure D-14. Metal concentrations in leachate for TRI 9 mm sample from the hold test using sand and rain water.

STANDARD RUN E. Soil with Simulated Rain Water

Media: *Soil – Eastern TN top soil*
Solvent: *Simulated rain water*
Technique(s): Up-flow and Hold
Bullet types: Lead M855
ESP-1 M855
ESP-1 9 mm FMJ
TRI-1 M855
TRI-1 9 mm FMJ

Summary: No tungsten found in leachant. Levels below detection limits of instrument (5 ppm*).

Table E-1: Metals Analysis: Up-Flow - Soil + Simulated Rain Water
(Concentration in mg/L)

Detection limits

W	Sn	Zn	Fe	Cu	Pb
5	0.05	0.005	0.03	0.02	0.01

Simulated acid rain

W	Sn	Zn	Fe	Cu	Pb
< 5	< 0.05	0.037	< 0.03	< 0.02	< 0.01

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 5	< 0.05	0.322	0.074	< 0.02	< 0.01
2	< 5	< 0.05	0.058	< 0.03	< 0.02	< 0.01
4	< 5	< 0.05	0.019	0.454	< 0.02	< 0.01
8	< 5	< 0.05	0.048	0.906	< 0.02	< 0.01
16	< 5	< 0.05	0.025	1.35	< 0.02	< 0.01
32	< 5	< 0.05	0.006	5.85	< 0.02	< 0.01

ESP M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 5	< 0.05	0.364	0.03	< 0.02	< 0.01
2	< 5	< 0.05	0.134	0.058	< 0.02	< 0.01
4	< 5	< 0.05	0.022	0.229	< 0.02	< 0.01
8	< 5	< 0.05	0.040	1.57	< 0.02	< 0.01
16	< 5	< 0.05	0.031	2.26	< 0.02	< 0.01
32	< 5	< 0.05	0.114	5.35	< 0.02	< 0.01

ESP 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 5	< 0.05	0.524	0.112	< 0.02	< 0.01
2	< 5	< 0.05	0.232	0.045	< 0.02	< 0.01
4	< 5	< 0.05	0.032	0.212	< 0.02	< 0.01
8	< 5	< 0.05	0.046	0.418	< 0.02	< 0.01
16	< 5	< 0.05	0.023	0.716	< 0.02	< 0.01
32	< 5	< 0.05	0.012	1.54	< 0.02	< 0.01

TRI 9mm Ball

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 5	< 0.05	0.422	0.03	< 0.02	< 0.01
2	< 5	< 0.05	0.205	0.099	< 0.02	< 0.01
4	< 5	< 0.05	0.039	0.086	< 0.02	< 0.01
8	< 5	< 0.05	0.076	0.499	< 0.02	< 0.01
16						
32	< 5	< 0.05	< 0.005	1.38	< 0.02	< 0.01

TRI M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 5	< 0.05	0.641	0.081	< 0.02	< 0.01
2	< 5	< 0.05	0.244	0.059	< 0.02	< 0.01
4						
8	< 5	< 0.05	0.018	0.307	< 0.02	< 0.01
16	< 5	< 0.05	0.06	1.11	< 0.02	< 0.01
32	< 5	< 0.05	0.008	1.21	< 0.02	< 0.01

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 5	< 0.05	0.359	0.034	< 0.02	< 0.01
2	< 5	< 0.05	0.158	< 0.03	< 0.02	< 0.01
4	< 5	< 0.05	0.026	0.131	< 0.02	< 0.01
8	< 5	< 0.05	0.033	0.098	< 0.02	< 0.01
16	< 5	< 0.05	0.026	0.669	< 0.02	< 0.01
32	< 5	< 0.05	0.022	1.50	< 0.02	< 0.01

Table E-2: Metals Analysis: Hold - Soil + Simulated Rain Water
(Concentration in mg/L)

Detection limits

W	Sn	Zn	Fe	Cu	Pb
5	0.05	0.005	0.03	0.02	0.1

Solvent - simulated acid rain

W	Sn	Zn	Fe	Cu	Pb
< 5	< 0.05	0.031	0.044	< 0.02	< 0.1

Control

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.42	0.063	< 0.02	< 0.1
15	< 5	< 0.05	0.424	0.035	< 0.02	< 0.1
35	< 5	< 0.05	0.409	0.281	< 0.02	0.05

ESP M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.468	0.072	< 0.02	< 0.1
15	< 5	< 0.05	0.529	0.072	< 0.02	< 0.1
35	< 5	< 0.05	0.627	0.814	< 0.02	< 0.1

ESP 9 mm

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.482	0.047	< 0.02	< 0.1
15	< 5	< 0.05	0.47	0.154	< 0.02	0.08
35	< 5	< 0.05	0.501	0.648	< 0.02	< 0.1

TRI 9 mm

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.45	0.074	< 0.02	< 0.1
15	< 5	< 0.05	0.438	0.426	< 0.02	< 0.1
35	< 5	< 0.05	0.462	4.57	< 0.02	< 0.1

TRI M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.444	0.056	< 0.02	< 0.1
15	< 5	< 0.05	0.313	0.321	< 0.02	< 0.1
35	< 5	< 0.05	0.301	3.29	< 0.02	< 0.1

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 5	< 0.05	0.589	0.136	< 0.02	< 0.1
15	< 5	< 0.05	0.404	0.133	< 0.02	0.07
35	< 5	< 0.05	0.434	6.42	< 0.02	< 0.1

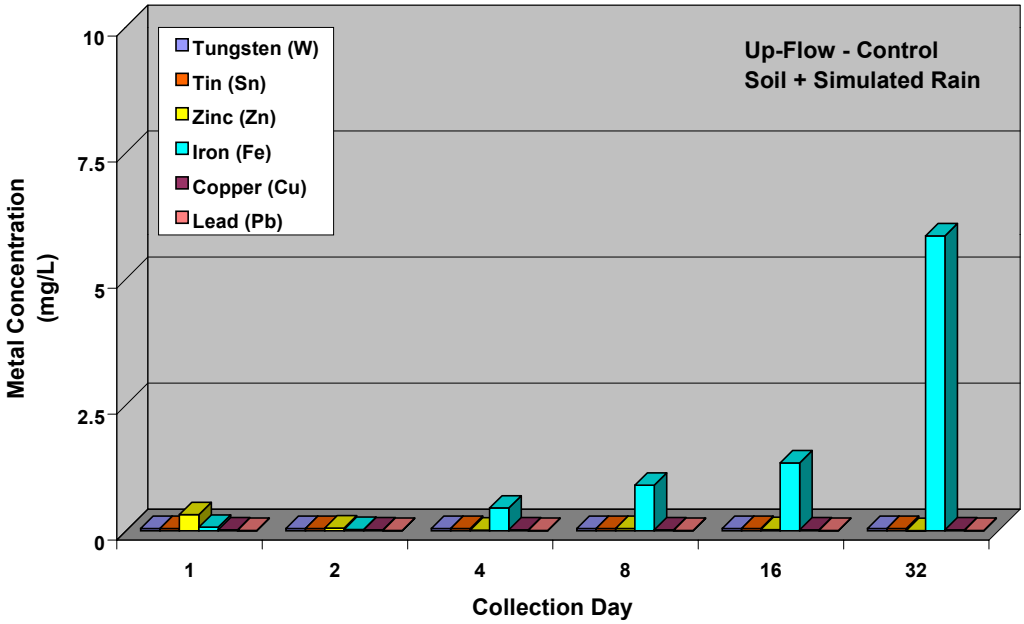

Figure E-1. Metal concentrations in leachate for control sample from the up-flow test using soil and rain water.

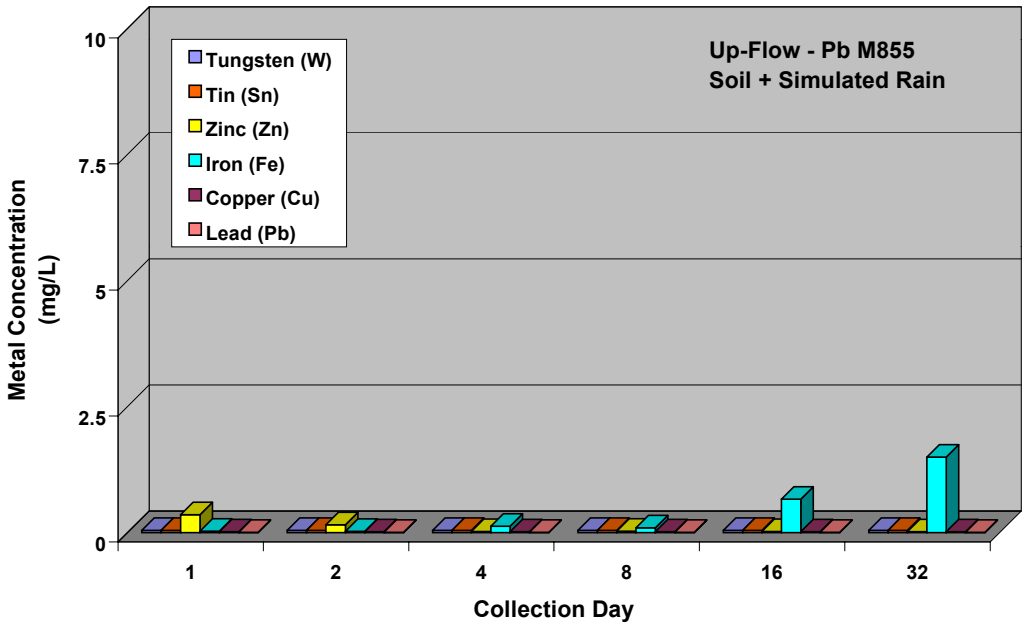

Figure E-2. Metal concentrations in leachate for lead M855 sample from the up-flow test using soil and rain water.

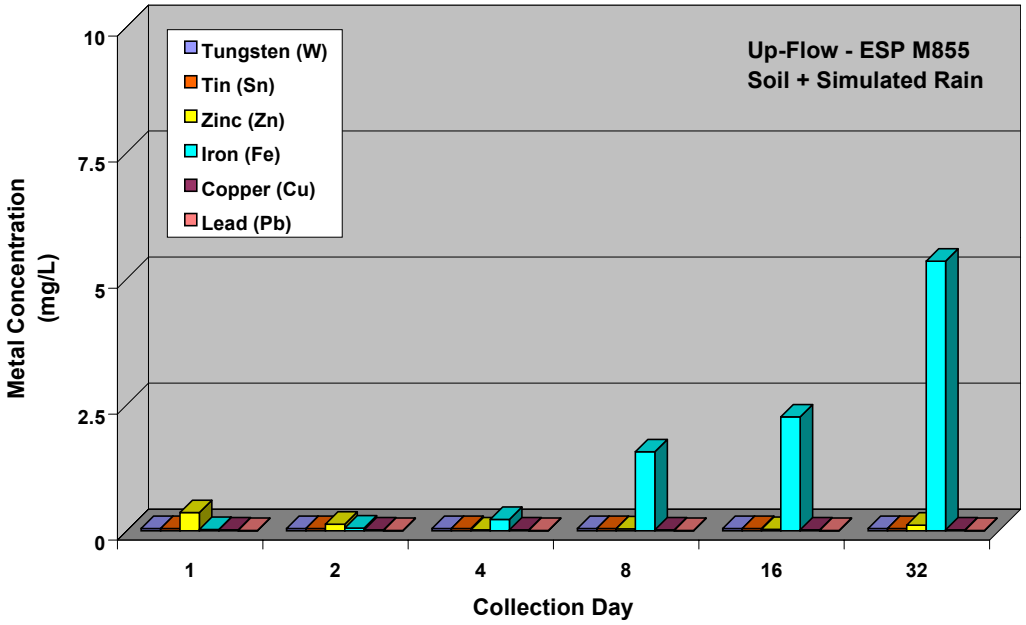

Figure E-3. Metal concentrations in leachate for ESP M855 sample from the up-flow test using soil and rain water.

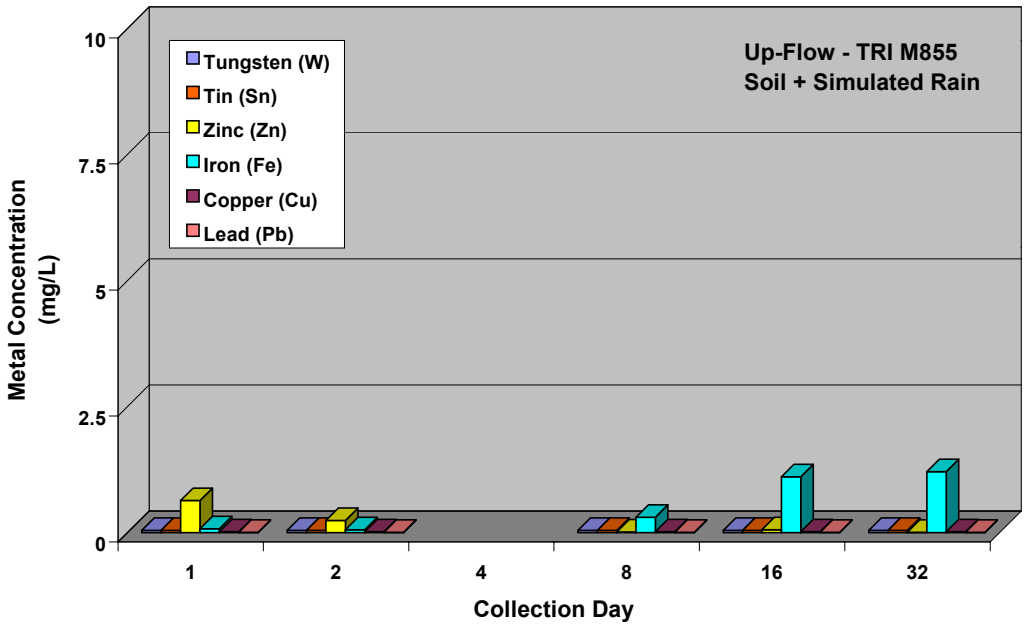

Figure E-4. Metal concentrations in leachate for TRI M855 sample from the up-flow test using soil and rain water

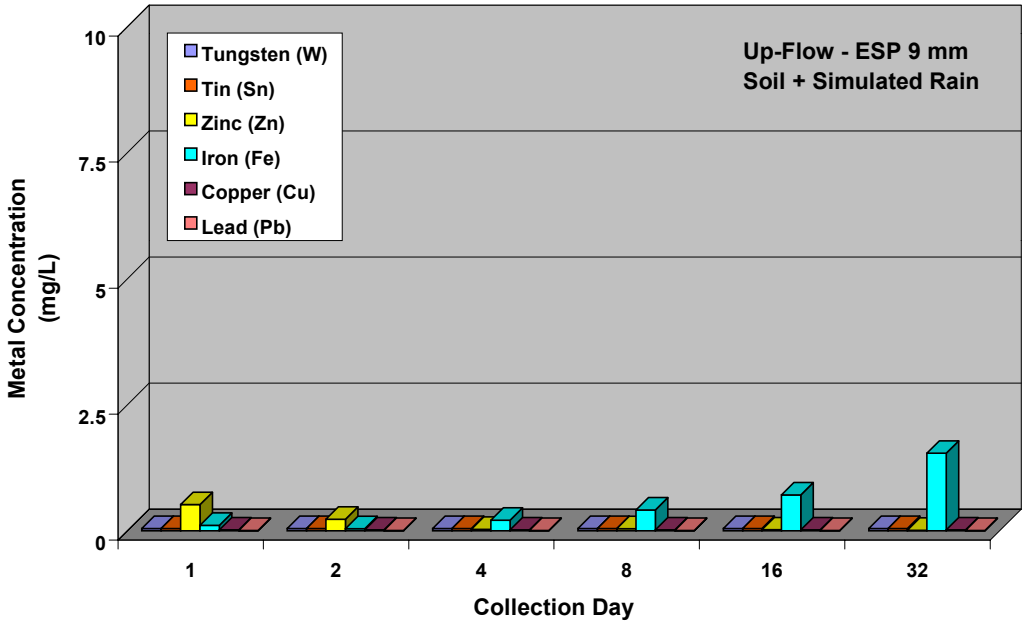

Figure E-5. Metal concentrations in leachate for ESP 9 mm sample from the up-flow test using soil and rain water.

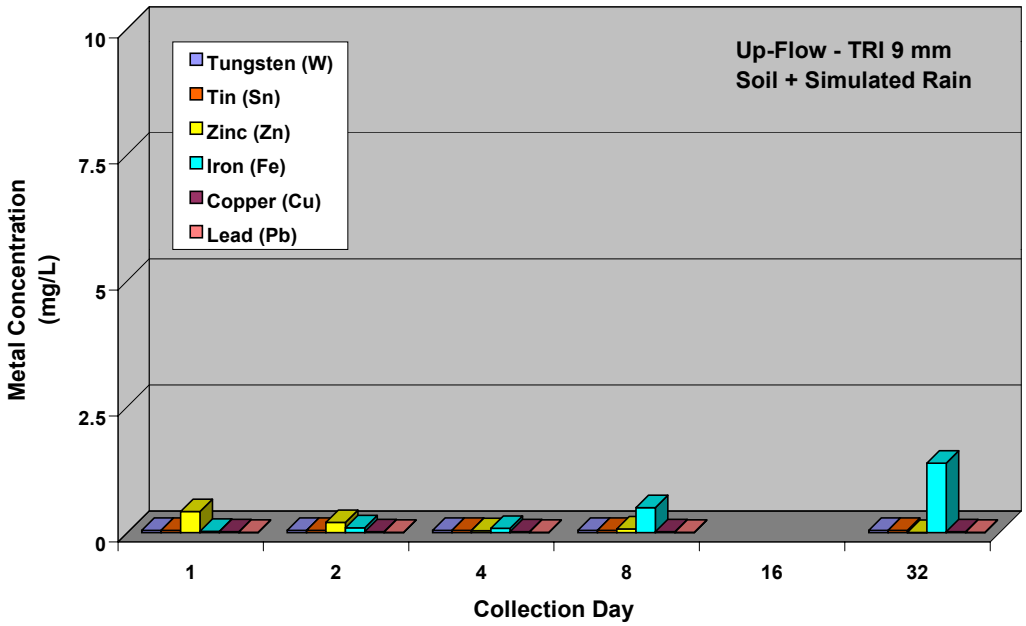

Figure E-6. Metal concentrations in leachate for TRI 9 mm sample from the up-flow test using soil and rain water.

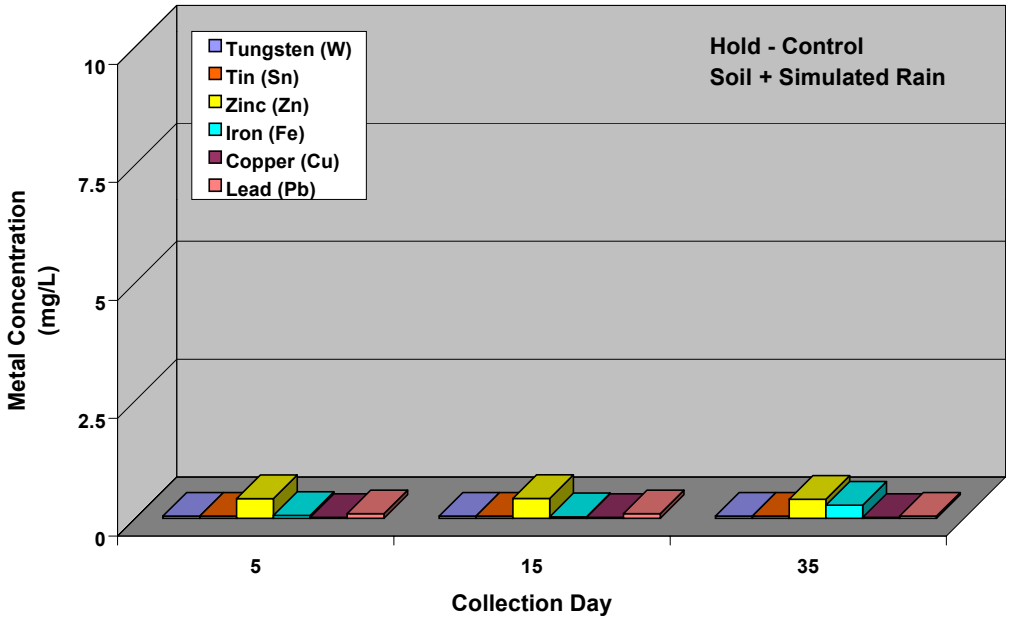

Figure E-7. Metal concentrations in leachate for control sample from the hold test using soil and rain water.

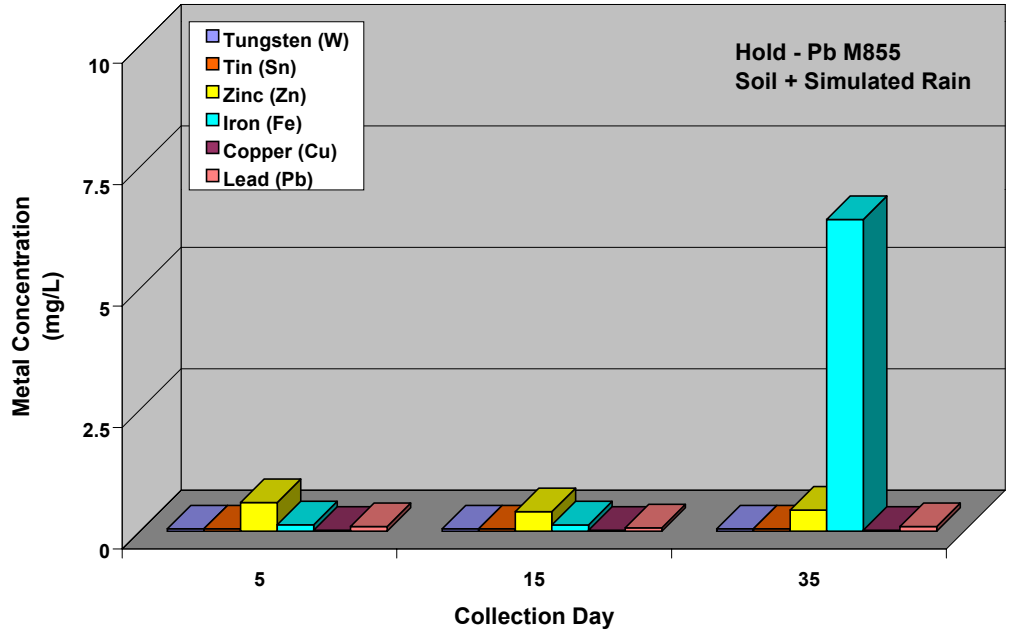

Figure E-8. Metal concentrations in leachate for lead M855 sample from the hold test using soil and rain water.

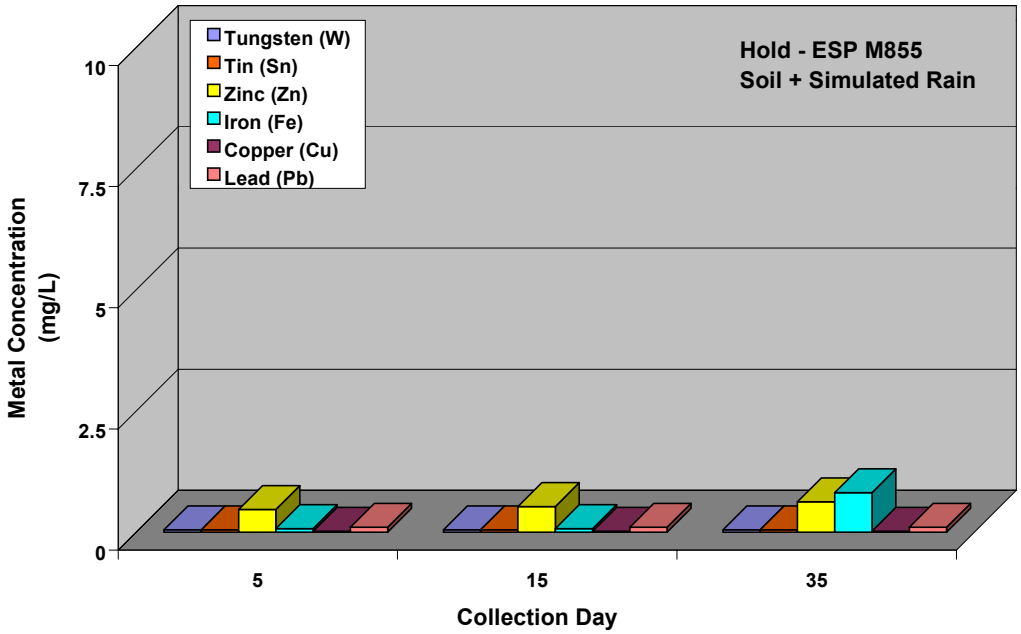

Figure E-9. Metal concentrations in leachate for ESP M855 sample from the hold test using soil and rain water.

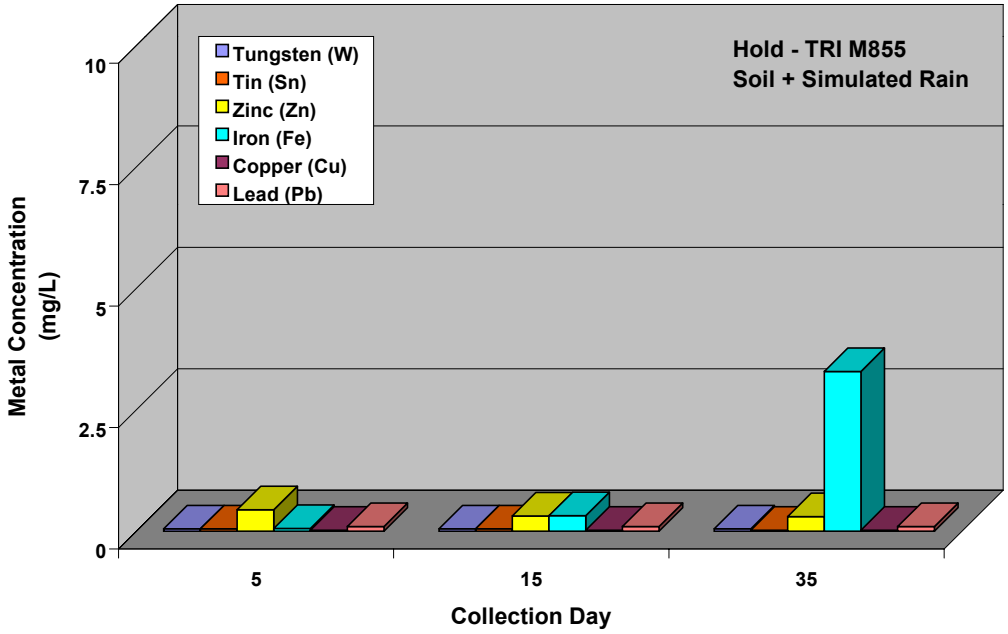

Figure E-10. Metal concentrations in leachate for TRI M855 sample from the hold test using soil and rain water.

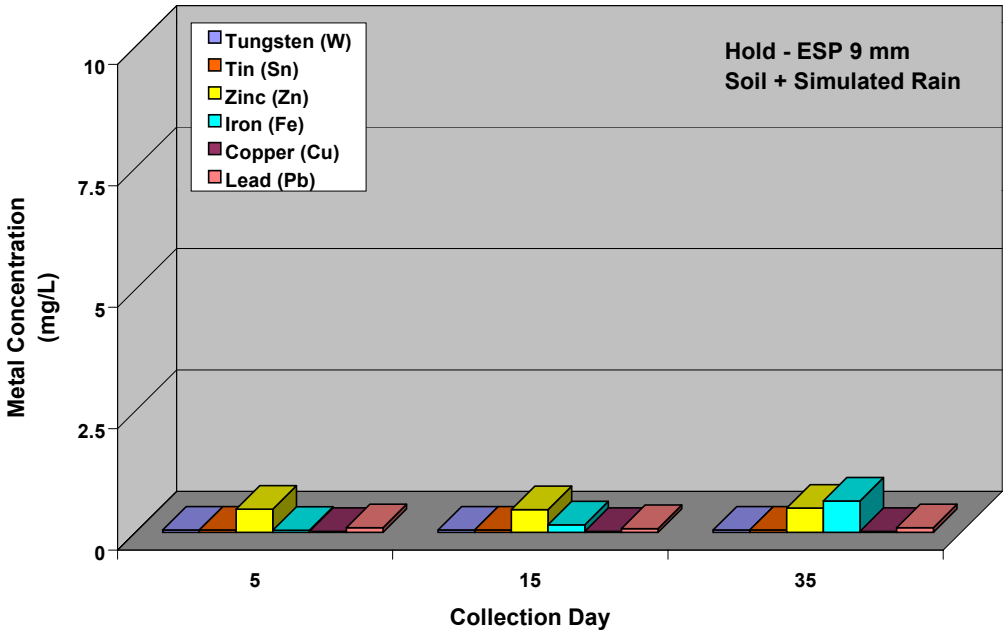

Figure E-11. Metal concentrations in leachate for ESP 9 mm sample from the hold test using soil and rain water.

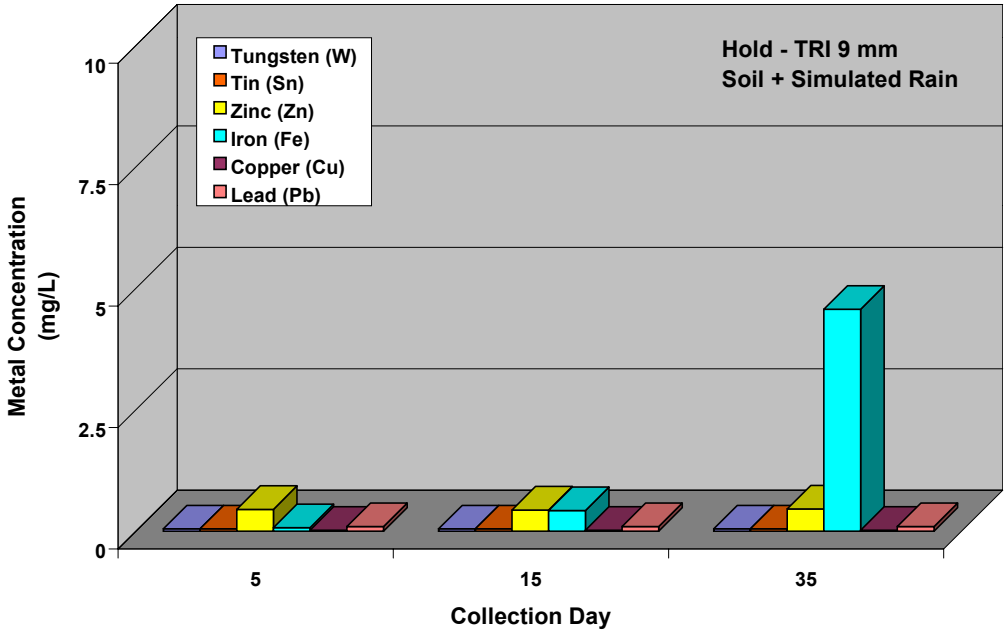

Figure E-12. Metal concentrations in leachate for TRI 9 mm sample from the hold test using soil and rain water.

SPECIAL RUN G. Mixed Materials

Media: *Sand*
Solvent: *Rain water*
Technique(s): Hold
Bullet types: Lead M855
ESP-1 M855
TRI-1 M855
Lead M855 + ESP-1 M855
Lead M855 + TRI-1 M855

Summary: No apparent differences in tungsten or lead concentrations for mixed materials.
*Detection limits of instrument were lower for more recent runs (0.01 ppm for W and Pb).

Media: *Soil*
Solvent: *Rain water*
Technique(s): Hold
Bullet types: Lead M855
ESP-1 M855
TRI-1 M855
Lead M855 + ESP-1 M855
Lead M855 + TRI-1 M855

Summary: No apparent differences in tungsten or lead concentrations for mixed materials. Detection limits of instrument were lower for more recent runs (0.01 ppm for W and Pb).

Table G-1. Metals Analysis: Sand + Simulated Rain Water

Lead mixtures Hold Technique

(Concentrations in mg/L)

Detection limits

	W	Sn	Zn	Fe	Cu	Pb
	0.05	0.05	0.005	0.01	0.02	0.02
	0.01	0.05	0.005	0.01	0.02	0.02

Control (Sand Only)

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.0748	0.8	< 0.02	0.0239
15	< 0.05	< 0.05	0.0972	0.209	< 0.02	< 0.02
35	0.02	< 0.05	0.0254	0.1	< 0.02	< 0.02

Lead M855 + TRI M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	5.56	< 0.05	0.235	1.66	0.0272	0.0381
15	4.52	< 0.05	0.0833	0.0515	< 0.02	< 0.02
35	3.51	< 0.05	0.0865	0.084	< 0.02	< 0.02

Lead M855 + ESP M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	2.81	< 0.05	0.186	2.13	< 0.02	< 0.02
15	2.91	< 0.05	0.0433	0.161	< 0.02	< 0.02
35	2.69	< 0.05	0.0205	0.0708	< 0.02	< 0.02

ESP M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	12	< 0.05	0.043	< 0.03	< 0.02	< 0.1
15	5.7	< 0.05	0.017	< 0.03	< 0.02	< 0.1
35	2.2	0.0213	0.0872	0.0574	< 0.02	0.0154

TRI M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	38	< 0.05	0.034	< 0.03	< 0.02	< 0.1
15	35	< 0.05	0.065	< 0.03	< 0.02	< 0.1
35	2.41	< 0.02	0.0585	0.0547	< 0.02	0.021

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.037	< 0.03	< 0.02	< 0.1
15	< 0.05	< 0.05	0.04	< 0.03	< 0.02	< 0.1
35	0.061	< 0.02	0.129	0.0506	< 0.02	0.0164

Control (Soil Only)

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.294	0.133	< 0.02	0.0374
15	< 0.05	< 0.05	0.36	0.0646	< 0.02	0.0549
35	0.029	< 0.05	0.242	0.822	< 0.02	0.1

Lead M855 + TRI M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	0.13	< 0.05	0.386	0.0463	< 0.02	0.0206
15	< 0.05	< 0.05	0.293	0.154	0.0201	0.0282
35	0.037	< 0.05	0.164	12.3	< 0.02	< 0.02

Lead M855 + ESP M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.208	0.417	0.0207	0.0463
15	< 0.05	< 0.05	0.211	0.516	0.0302	0.0462
35	< 0.01	< 0.05	0.126	12.4	< 0.02	0.0257

ESP M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.468	0.072	< 0.02	< 0.1
15	< 0.05	< 0.05	0.529	0.072	< 0.02	< 0.1
35	< 5	< 0.05	0.627	0.814	< 0.02	< 0.1

TRI M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.444	0.056	< 0.02	< 0.1
15	< 0.05	< 0.05	0.313	0.321	< 0.02	< 0.1
35	< 0.05	< 0.05	0.301	3.29	< 0.02	< 0.1

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.589	0.136	< 0.02	< 0.1
15	< 0.05	< 0.05	0.404	0.133	< 0.02	0.07
35	< 0.05	< 0.05	0.434	6.42	< 0.02	< 0.1

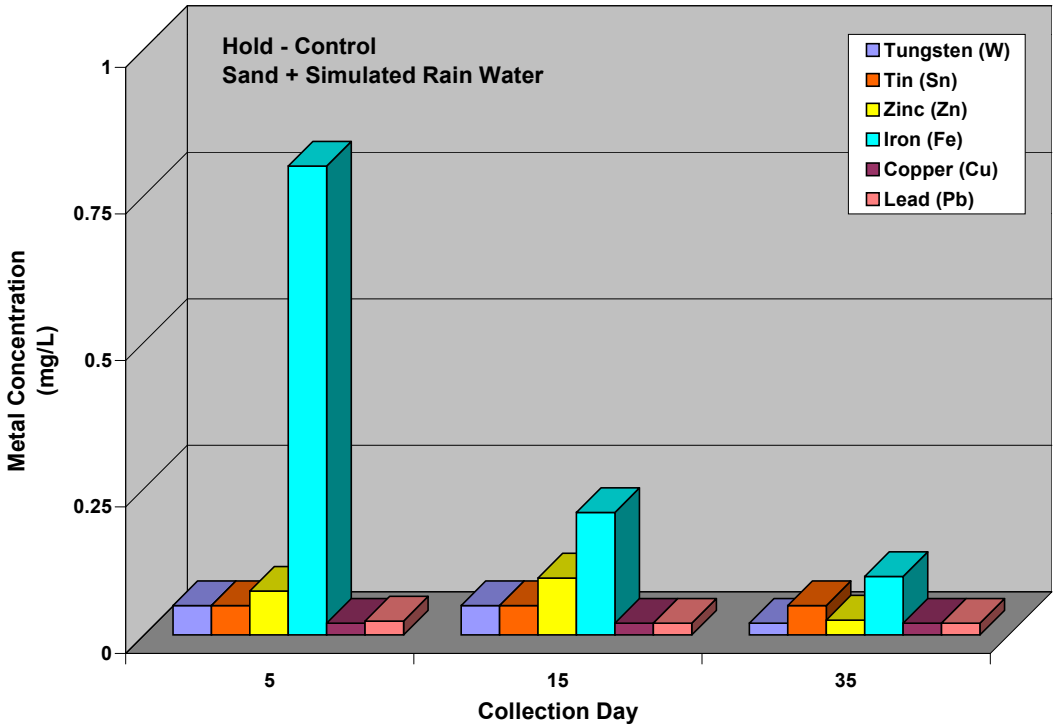

Figure G-1. Metal concentrations in leachate for control sample from the hold test using sand and rain water.

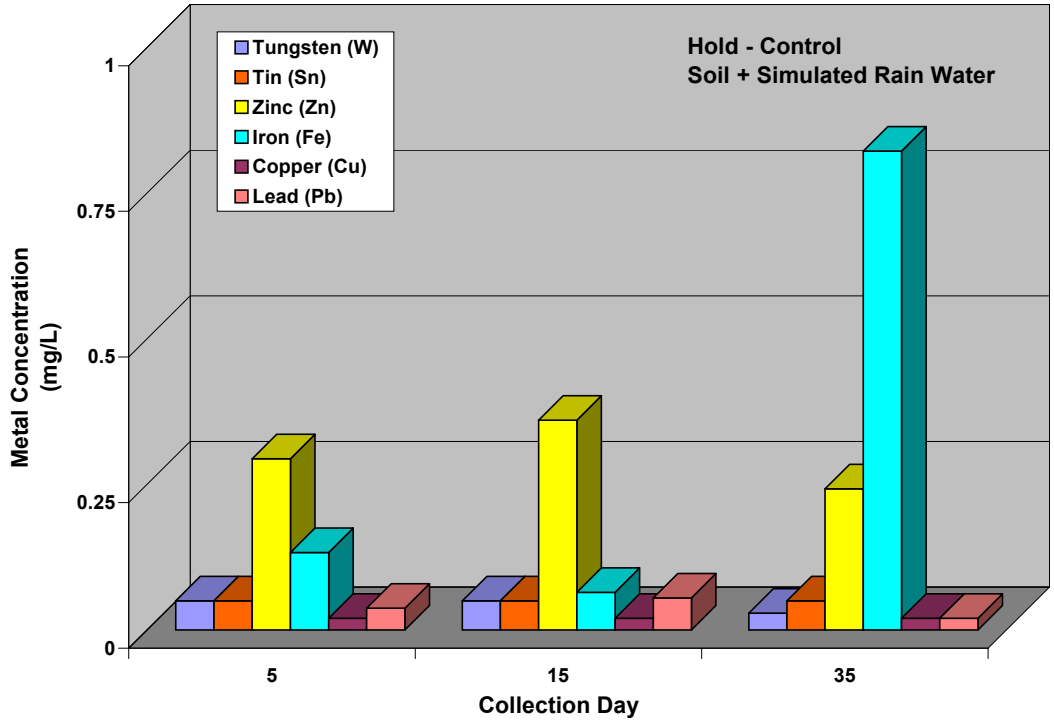

Figure G-2. Metal concentrations in leachate for control sample from the hold test using soil and rain water.

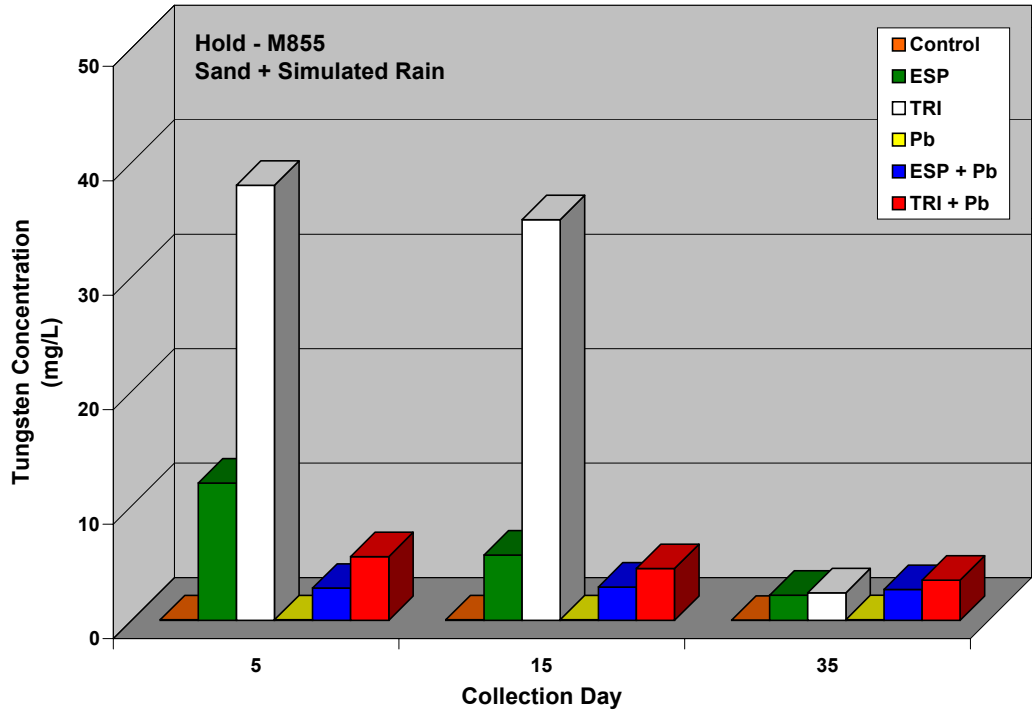

Figure G-3. Tungsten concentrations in leachate for mixed materials sample from the hold test using sand and rain water.

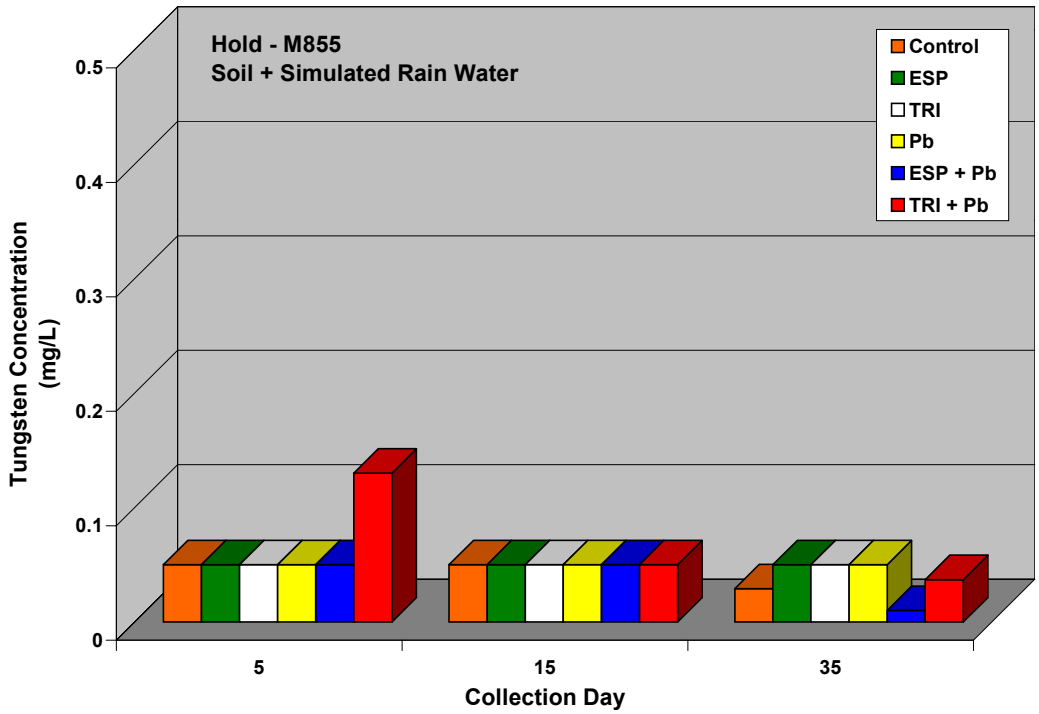

Figure G-4. Tungsten concentrations in leachate for mixed materials sample from the hold test using soil and rain water.

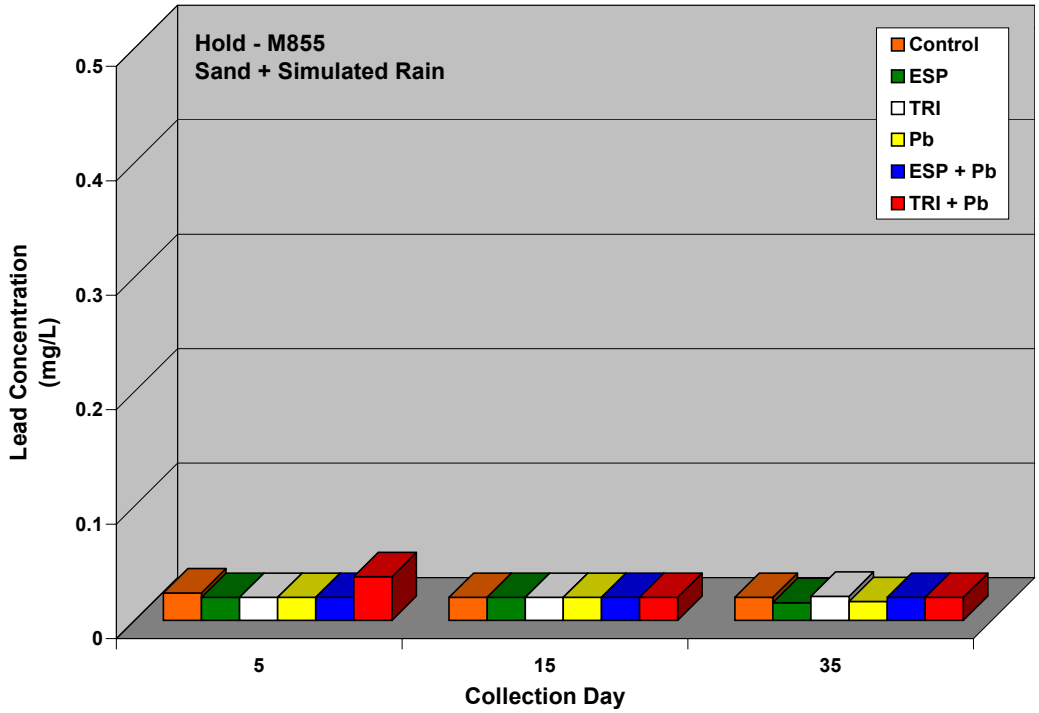

Figure G-5. Lead concentrations in leachate for mixed materials sample from the hold test using sand and rain water.

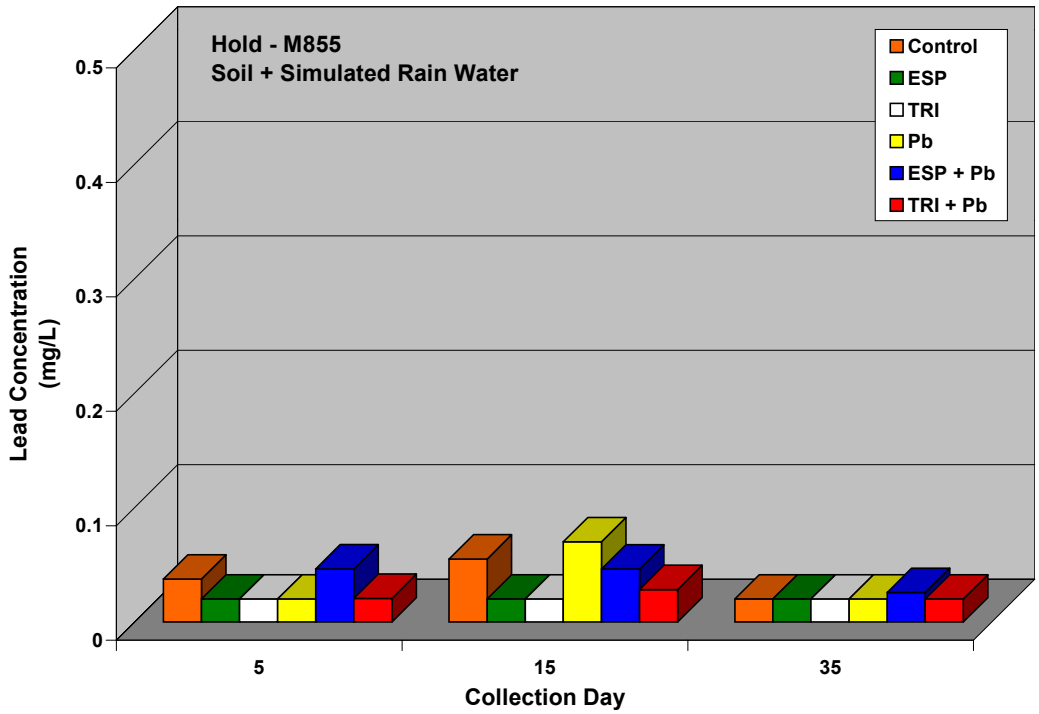

Figure G-6. Lead concentrations in leachate for mixed materials sample from the hold test using soil and rain water.

SPECIAL RUN H. Precision Study

Media: *Sand and Soil (separate runs, not mixtures)*
Solvent: *Rain water*
Technique(s): Up-Flow and Hold
Bullet types: ESP-1 M855
TRI-1 M855

Summary:

Tungsten was not leached from columns containing "contaminated" soil mixtures thus there was no data to display.

The up-flow results, average concentrations and standard deviations, for the ESP-containing columns demonstrated good reproducibility for the technique (Table H-1 and Figure H-1). The variability in the data for 32-day samples was much greater than for all other samples. The metals concentrations were measured using a different instrument thus the integrity of the data is suspect.

The hold results, average concentrations and standard deviations, for the ESP-containing columns demonstrated good reproducibility for the technique (Table H-1 and Figure H-1). The variability in the data for 32-day samples was much greater than for all other samples. The metals concentrations were measured using a different instrument thus the integrity of the data is suspect.

The up-flow and hold results, average concentrations and standard deviations, for the TRI-containing columns did not demonstrate as good reproducibility as for the ESP materials (Tables H-1 and H-2, and Figure H-2 and H-4). The variability in the data for all samples was much greater. As noted for the earlier studies using sand as the media (A, C and D), small particles of tungsten (< 0.7 microns) were washed from the column into the leachant. A fraction of the tungsten in the TRI material is smaller than 1 micron in diameter (see Section .

Table H-1. Metals Analysis: Up-flow - Sand + Simulated Rain Water
Precision study
[Concentrations in mg/L]

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
1,2,4,8,16	0.05	0.05	0.005	0.01	0.01	0.02
32	0.05	0.05	0.01	0.03	0.02	0.02

Column A. ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	36.6	< 0.05	0.0237	< 0.03	< 0.02	0.0264
2	19.3	< 0.05	< 0.01	< 0.03	< 0.02	0.0203
4	17	< 0.05	< 0.01	< 0.03	< 0.02	0.0214
8	18.3	< 0.05	0.0111	< 0.03	< 0.02	< 0.02
16	17.1	< 0.05	< 0.01	0.036	< 0.02	< 0.02
32	33.4	< 0.05	0.067	0.0232	< 0.01	0.21

Column B. ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	29.2	< 0.05	0.0274	< 0.03	< 0.02	0.0263
2	17.6	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
4	16.7	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
8	18	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
16	15.5	< 0.05	< 0.01	0.035	< 0.02	< 0.02
32	12.6	0.0968	0.628	0.508	< 0.01	0.233

Column C. ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	30.7	< 0.05	0.0374	< 0.03	< 0.02	0.0272
2	17.8	< 0.05	0.0109	< 0.03	< 0.02	< 0.02
4	17.4	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
8	19.7	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
16	18.9	< 0.05	< 0.01	0.034	< 0.02	< 0.02
32	39.7	< 0.05	0.0505	0.0476	< 0.01	0.258

Average: ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	32.2	< 0.05	0.03	< 0.03	< 0.02	0.0266
2	18.2	< 0.05	< 0.01	< 0.03	< 0.02	< 0.021
4	17.0	< 0.05	< 0.01	< 0.03	< 0.02	< 0.021
8	18.7	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
16	17.2	< 0.05	< 0.01	0.04	< 0.02	< 0.02
32	28.6	< 0.05	0.25	0.19	< 0.01	0.2337

Standard Deviation: ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	3.9	0.0	0.01	0.00	0.0	0.0005
2	0.9	0.0	0.00	0.00	0.0	0.0002
4	0.4	0.0	0.00	0.00	0.0	0.0008
8	0.9	0.0	0.00	0.00	0.0	0.0000
16	1.7	0.0	0.00	0.00	0.0	0.0000
32	14.2	0.0	0.33	0.27	0.0	0.0240

Column D. TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	17	< 0.05	0.0554	< 0.03	< 0.02	0.0241
2	22.7	< 0.05	< 0.01	< 0.03	< 0.02	0.0217
4	3.37	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
8	12.4	< 0.05	0.0151	< 0.03	< 0.02	< 0.02
16	24.4	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
32	64.1	< 0.05	0.0843	< 0.01	< 0.01	0.207

Column E. TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	32.3	< 0.05	0.0202	< 0.03	< 0.02	0.0268
2	14.2	< 0.05	0.0115	< 0.03	< 0.02	< 0.02
4	11.9	< 0.05	0.012	< 0.03	< 0.02	< 0.02
8	11.1	< 0.05	0.0106	< 0.03	< 0.02	< 0.02
16	8.85	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
32	30.2	< 0.05	0.0624	< 0.01	< 0.01	0.247

Column F. TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	28.8	< 0.05	0.0151	< 0.03	< 0.02	0.0255
2	14.7	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
4	10.4	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
8	9.12	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
16	8.13	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
32	33	< 0.05	0.0315	< 0.01	< 0.01	0.284

Average: TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	26.0	< 0.05	0.03	< 0.03	< 0.02	0.0255
2	17.2	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
4	8.6	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
8	10.9	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
16	13.8	< 0.05	< 0.01	< 0.03	< 0.02	< 0.02
32	42.4	< 0.05	0.06	< 0.01	< 0.01	0.2460

Standard Deviation: TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	8.0	0.00	0.02	0.00	0.00	0.0014
2	4.8	0.00	0.00	0.00	0.00	0.0000
4	4.6	0.00	0.00	0.00	0.00	0.0000
8	1.7	0.00	0.00	0.00	0.00	0.0000
16	9.2	0.00	0.00	0.00	0.00	0.0000
32	18.8	0.00	0.03	0.00	0.00	0.0385

Table H-2. Metals Analysis: Hold - Sand + Simulated Rain Water

Precision Study

(Concentrations in mg/L)

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
5,15	0.05	0.05	0.005	0.01	0.01	0.02
35	0.05	0.05	0.01	0.03	0.02	0.02

Column A. ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	56.6	< 0.05	0.039	< 0.03	< 0.02	0.0376
15	45	< 0.05	0.0474	< 0.03	< 0.02	0.0332
35	15.4	< 0.05	0.0084	0.0341	< 0.01	0.0595

Column B. ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	37	< 0.05	0.0356	< 0.03	< 0.02	0.0273
15	17	< 0.05	0.0347	< 0.03	< 0.02	0.02
35	15.6	< 0.05	0.0073	0.0287	< 0.01	0.0699

Column C. ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	49.7	< 0.05	0.0321	< 0.03	< 0.02	0.0339
15	41.3	< 0.05	0.0324	< 0.03	< 0.02	0.0293
35	18	< 0.05	0.0084	0.0295	< 0.01	0.0722

Average: ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	47.8	< 0.05	0.0356	< 0.03	< 0.02	0.0329
15	34.4	< 0.05	0.0382	< 0.03	< 0.02	0.0275
35	16.3	< 0.05	0.0080	< 0.03	< 0.01	0.0672

Standard Deviation

Day	W	Sn	Zn	Fe	Cu	Pb
5	9.9	0	0.0035	0	0	0.0052
15	15.2	0	0.0081	0	0	0.0068
35	1.4	0	0.0006	0.003	0	0.0068

Column D. TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	127	< 0.05	0.0617	< 0.03	< 0.02	0.0748
15	101	< 0.05	0.0423	< 0.03	< 0.02	0.0565
35	10.3	< 0.05	0.0055	0.01	< 0.01	0.0722

Column E. TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	26.6	< 0.05	0.0331	< 0.03	< 0.02	0.0239
15	20.4	< 0.05	0.0442	< 0.03	0.0213	0.02
35	8.53	< 0.05	0.0067	0.0191	< 0.01	0.0683

Column F. TRI-1C M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	41.2	< 0.05	0.0265	< 0.03	< 0.02	0.0334
15	49.5	< 0.05	0.0311	< 0.03	< 0.02	0.0321
35	7.61	< 0.05	0.005	0.0243	< 0.01	0.0494

Average: TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	64.9	< 0.05	0.0404	< 0.03	< 0.02	0.0440
15	57.0	< 0.05	0.0392	< 0.03	< 0.02	0.0362
35	8.8	< 0.05	0.0057	0.02	< 0.01	0.0633

Standard Deviation

Day	W	Sn	Zn	Fe	Cu	Pb
5	54.2	0	0.0187	0	0	0.0271
15	40.8	0	0.0071	0	0	0.0186
35	1.4	0	0.0009	0.007	0	0.0122

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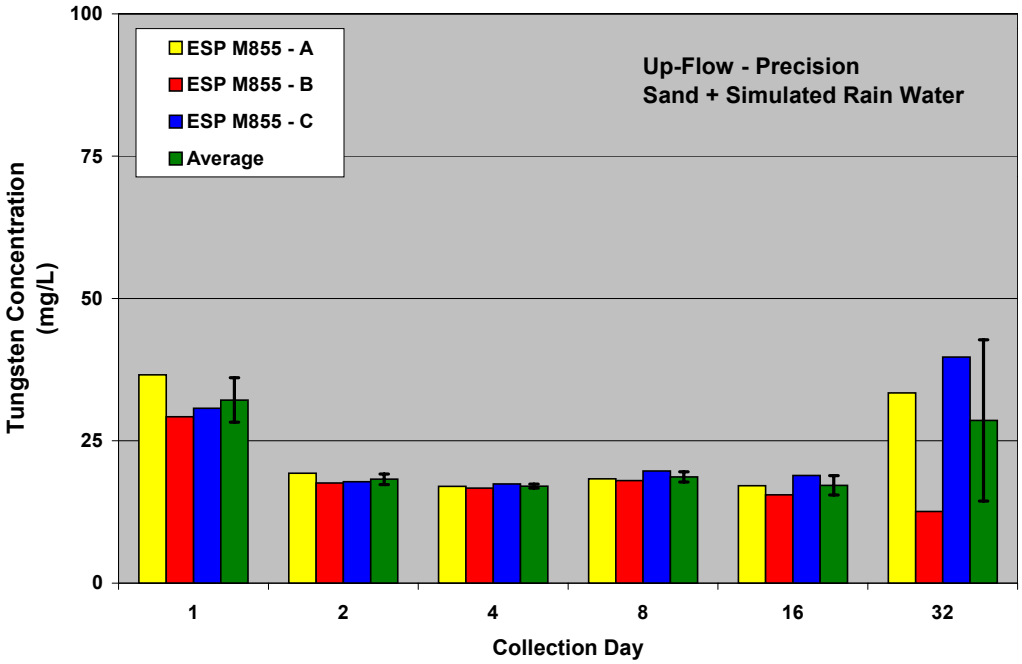

Figure H-1. Tungsten concentrations in leachate for ESP M855 sample from the precision up-flow test using sand and rain water.

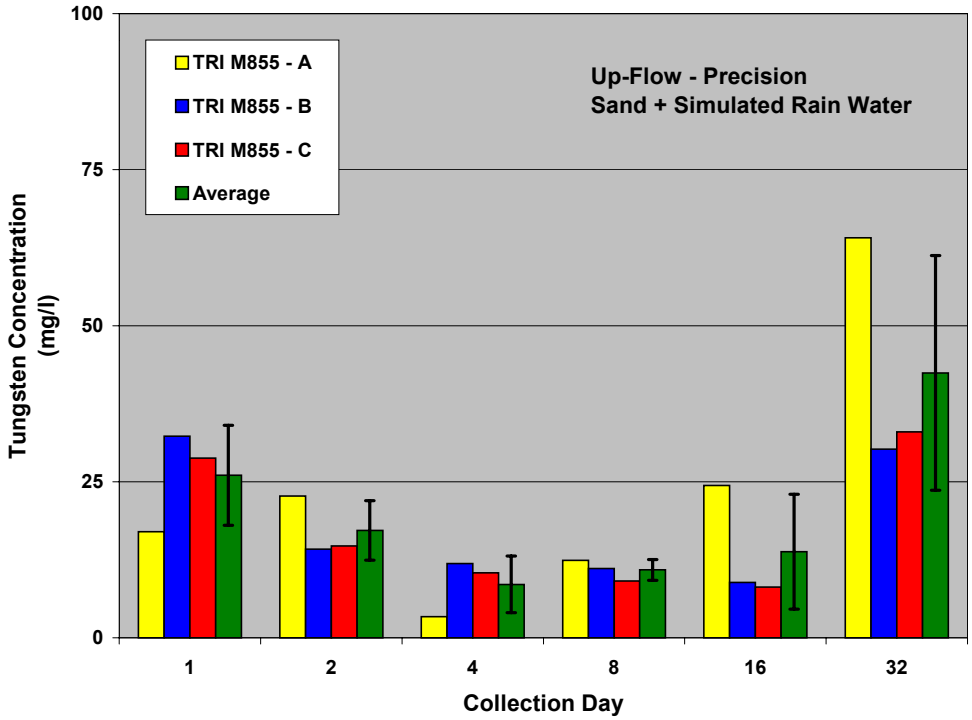

Figure H-2. Tungsten concentrations in leachate for TRI M855 sample from the precision up-flow test using sand and rain water.

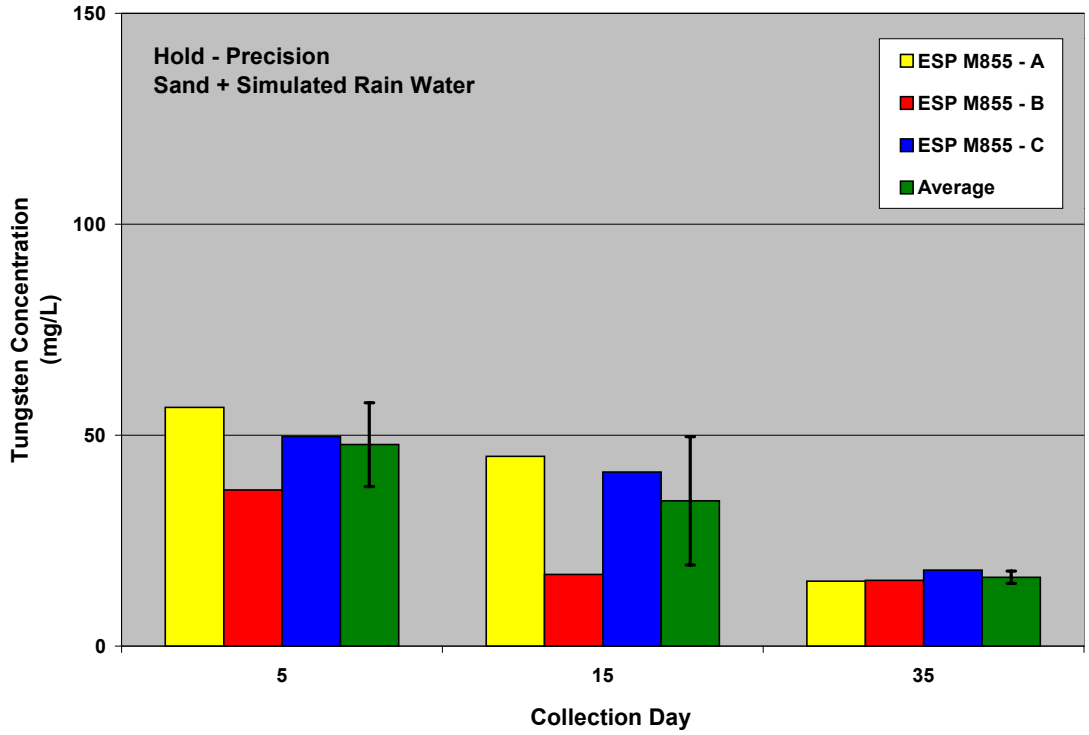

Figure H-3. Tungsten concentrations in leachate for ESP M855 sample from the precision hold test using sand and rain water.

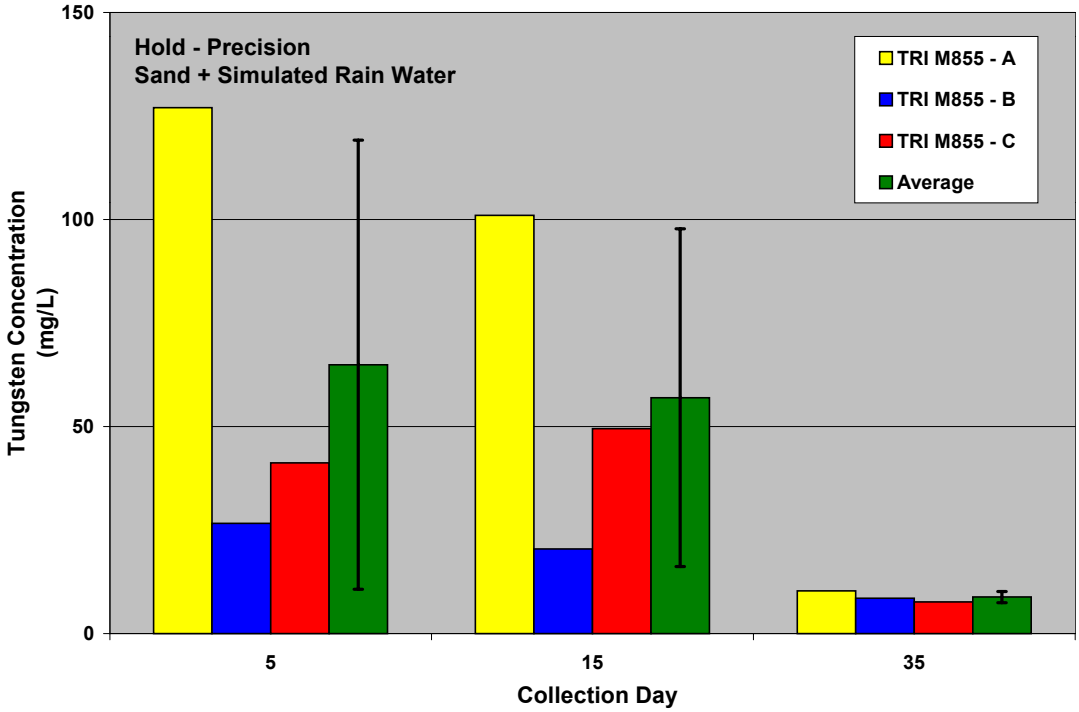

Figure H-4. Tungsten concentrations in leachate for TRI M855 sample from the precision hold test using sand and rain water.

SPECIAL RUN J. Bullets Fired Into Sand

Media: *Sand*
Solvent: *Rain water*
Technique(s): Up-Flow and Hold
Bullet types: Lead M855
ESP-1 M855
TRI-1 M855

Summary: Results similar to those for simulated materials. See D.

Table J-1. Metals Analysis: Up-Flow - Sand + Simulated Rain Water
As-fired projectiles
(Concentrations in mg/L)

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
1,2,4,8	0.05	0.05	0.005	0.01	0.02	0.02
16,32	0.03	0.02	0.01	0.02	0.01	0.05

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.05	0.028	< 0.01	< 0.02	< 0.02
2	< 0.05	< 0.05	0.018	< 0.01	< 0.02	< 0.02
4	< 0.05	< 0.05	0.005	< 0.01	< 0.02	< 0.02
8	< 0.05	< 0.05	0.005	< 0.01	< 0.02	< 0.02
16	< 0.05	< 0.02	0.005	< 0.02	< 0.01	< 0.05
32	< 0.05	< 0.02	0.0099	< 0.02	< 0.01	< 0.05

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	33	< 0.05	0.0424	0.0591	0.0535	0.0895
2	13.6	< 0.05	0.0192	0.0513	0.0282	0.0537
4	7.65	< 0.05	0.0192	0.0572	0.0338	0.0717
8	8.74	< 0.05	0.005	< 0.01	0.0114	0.0317
16	8.07	< 0.02	< 0.01	< 0.02	0.0136	< 0.05
32	5.53	< 0.02	< 0.01	< 0.02	< 0.01	< 0.05

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.05	0.458	0.831	0.309	0.132
2	105	< 0.05	0.177	0.354	0.134	0.102
4	4.45	< 0.05	0.0845	0.153	0.0673	0.0903
8	6.17	< 0.05	0.007	0.0237	0.0194	0.0383
16	18.9	< 0.02	< 0.01	< 0.02	0.0144	< 0.05
32	11.3	< 0.02	< 0.01	< 0.02	< 0.01	< 0.05

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	0.574	< 0.05	0.0223	< 0.01	0.041	0.125
2	0.259	< 0.05	0.0118	< 0.01	0.0237	0.0768
4	0.218	< 0.05	0.0135	0.0297	0.0664	0.108
8	0.216	< 0.05	0.005	0.0199	< 0.01	0.0527
16	0.132	< 0.02	< 0.01	< 0.02	0.0118	< 0.05
32	0.082	< 0.02	< 0.01	< 0.02	< 0.01	< 0.05

Table J-2. Metals Analysis: Hold - Sand + Simulated Rain Water
As-fired projectiles
(Concentrations in mg/L)

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
5,15	0.05	0.05	0.005	0.01	0.02	0.02
35	0.03	0.02	0.01	0.02	0.01	0.05

Control

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.046	< 0.01	< 0.02	< 0.02
15	< 0.05	< 0.05	0.031	< 0.01	< 0.02	< 0.02
35	< 0.03	0.0221	0.129	0.0452	< 0.01	0.0444

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	64.5	0.21	0.185	0.218	0.159	0.186
15	24.9	0.0745	0.075	0.0938	0.0962	0.177
35	9.16	0.0495	0.0592	0.0872	0.096	< 0.05

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	3.76	< 0.05	0.131	0.0541	0.0146	0.099
15	1.98	< 0.05	0.0216	0.0153	0.0211	0.122
35	90.3	0.0345	0.0832	0.769	0.0665	0.0657

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	0.0987	0.524	5.78	0.113	0.231
15	< 0.05	< 0.05	0.123	1.39	0.0518	0.171
35	2.74	0.0226	0.0207	< 0.02	0.0276	< 0.05

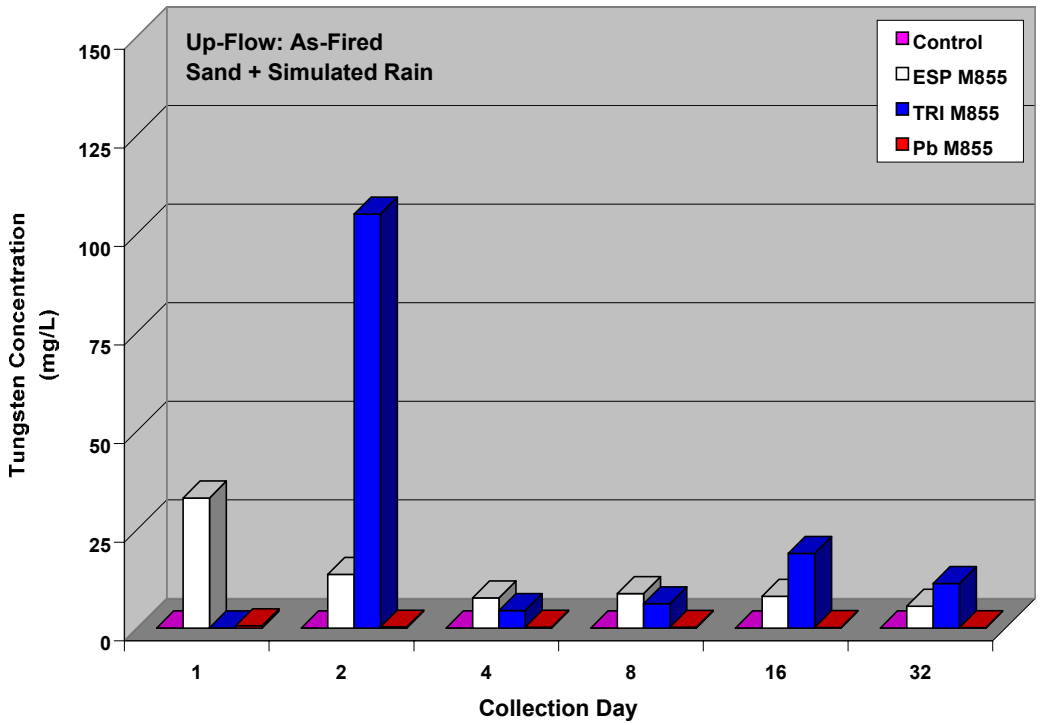

Figure J-1. Tungsten concentration in leachate for up-flow test using sand and rain water.

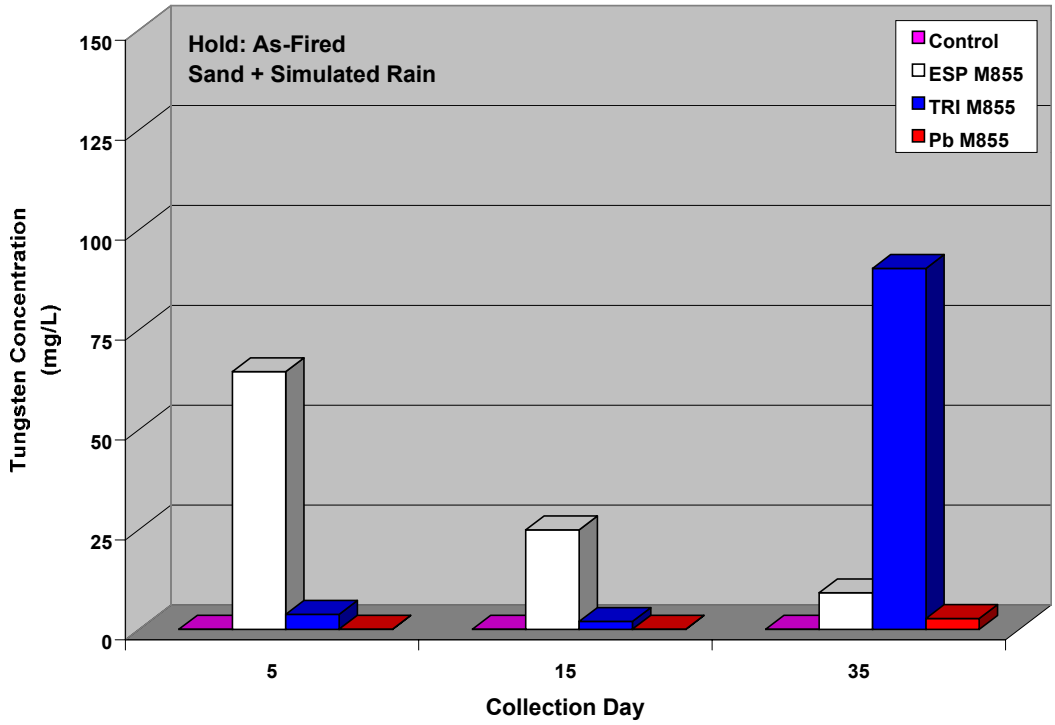

Figure J-2. Tungsten concentration in leachate for hold test using sand and rain water

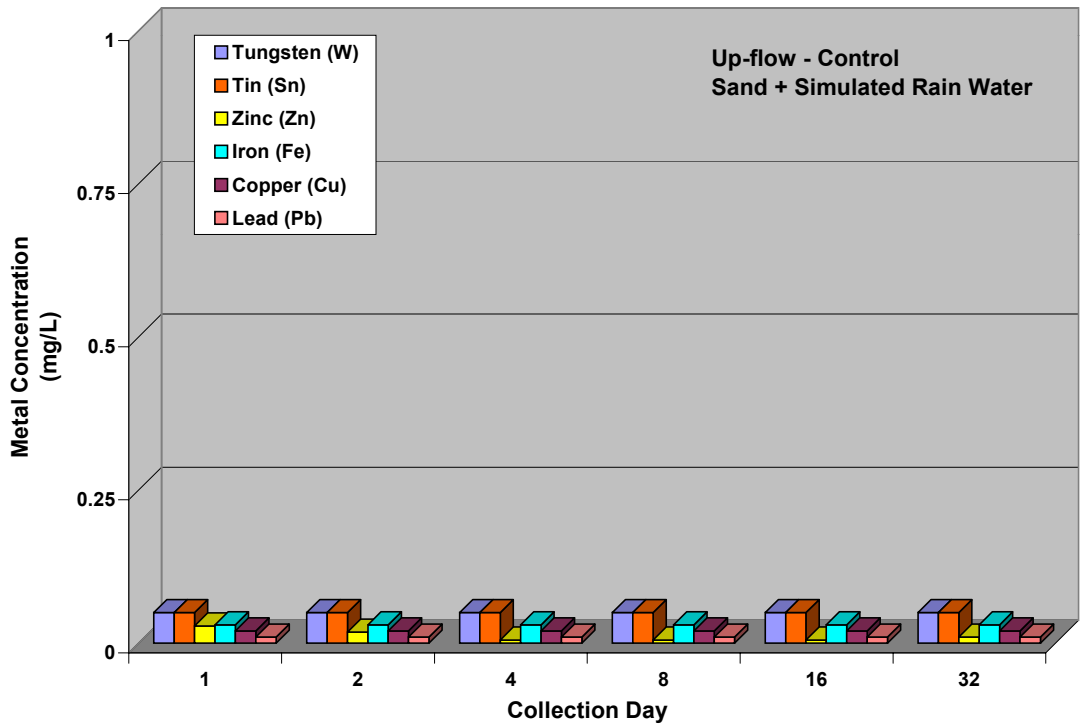

Figure J-3. Metal concentrations in leachate for control sample from the up-flow test using sand and rain water.

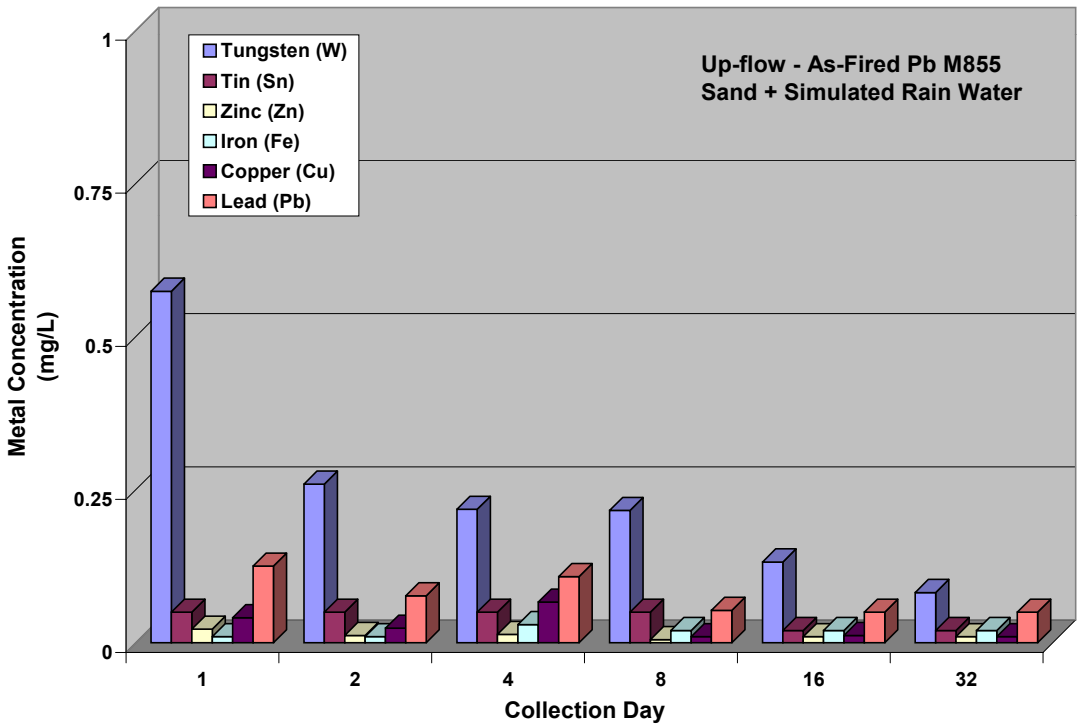

Figure J-4. Metal concentrations in leachate for lead M855 sample from the up-flow test using sand and rain water.

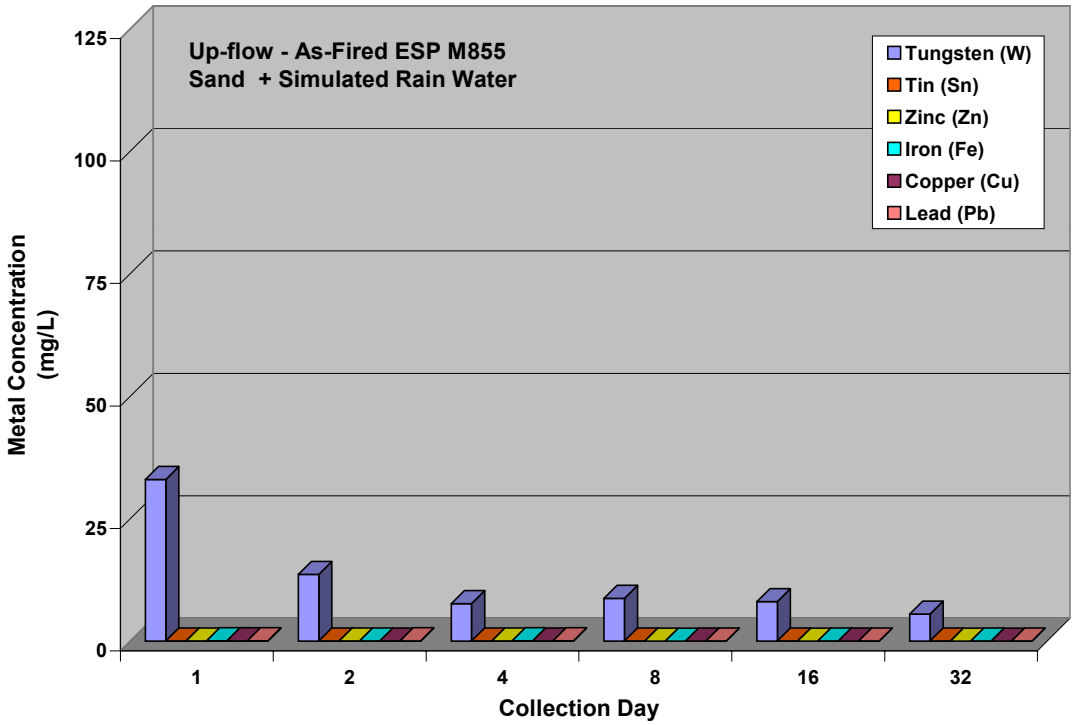

Figure J-5. Metal concentrations in leachate for ESP M855 sample from the up-flow test using sand and rain water.

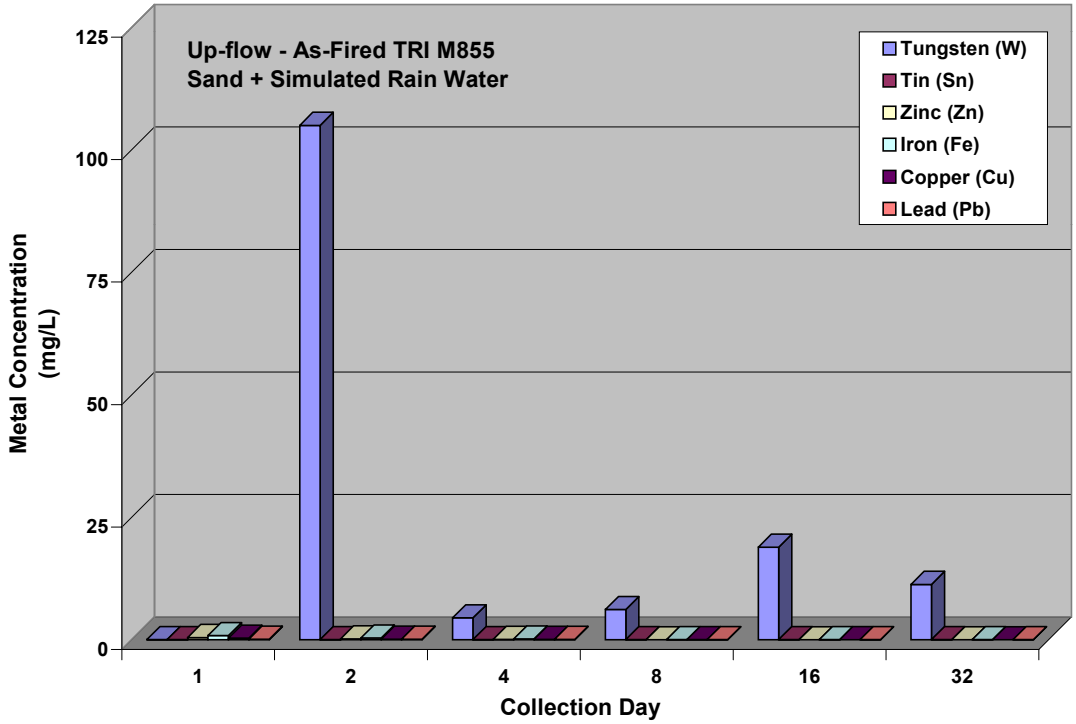

Figure J-6. Metal concentrations in leachate for TRI M855 sample from the up-flow test using sand and rain water

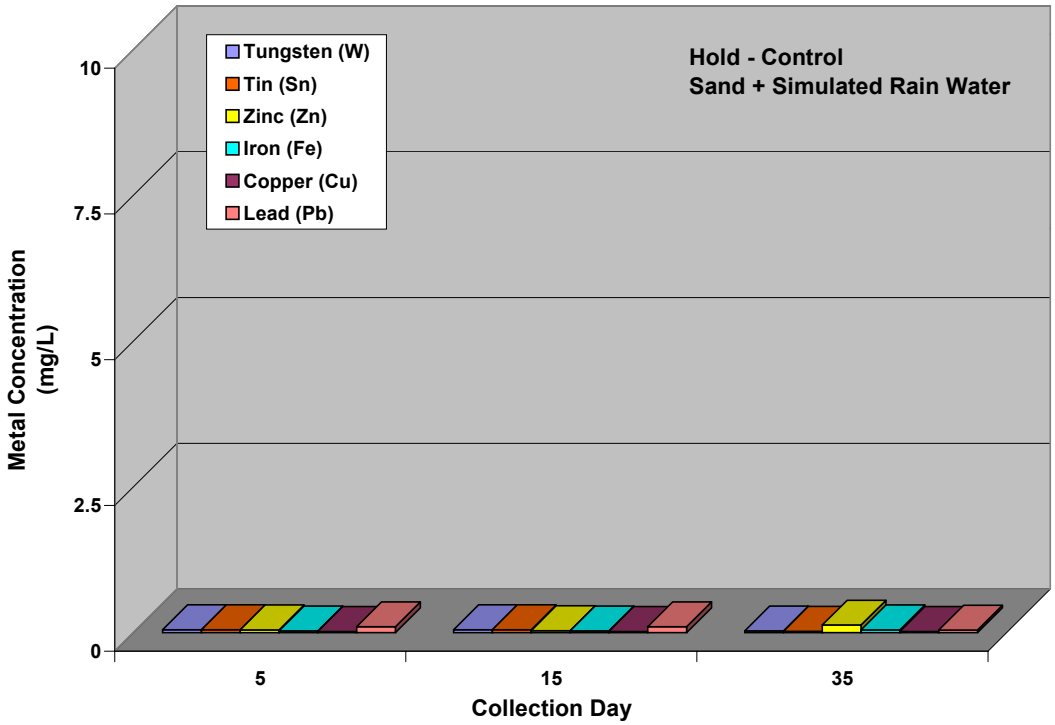

Figure J-7. Metal concentrations in leachate for the control sample from the hold test using sand and rain water.

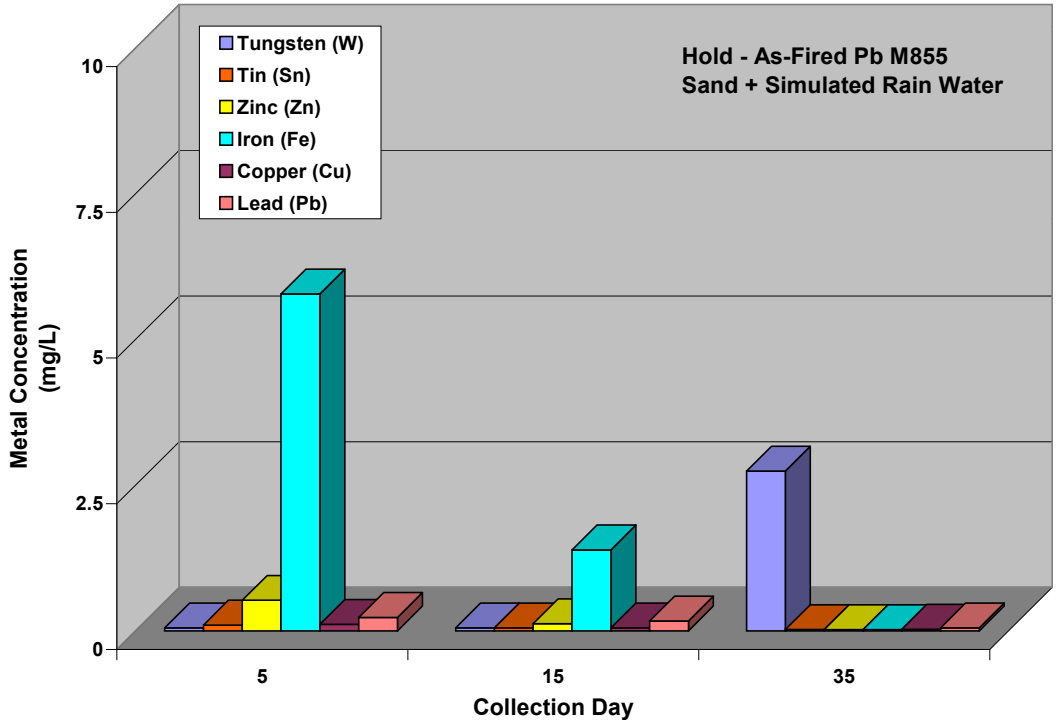

Figure J-8. Metal concentrations in leachate for lead M855 mm sample from the hold test using sand and rain water.

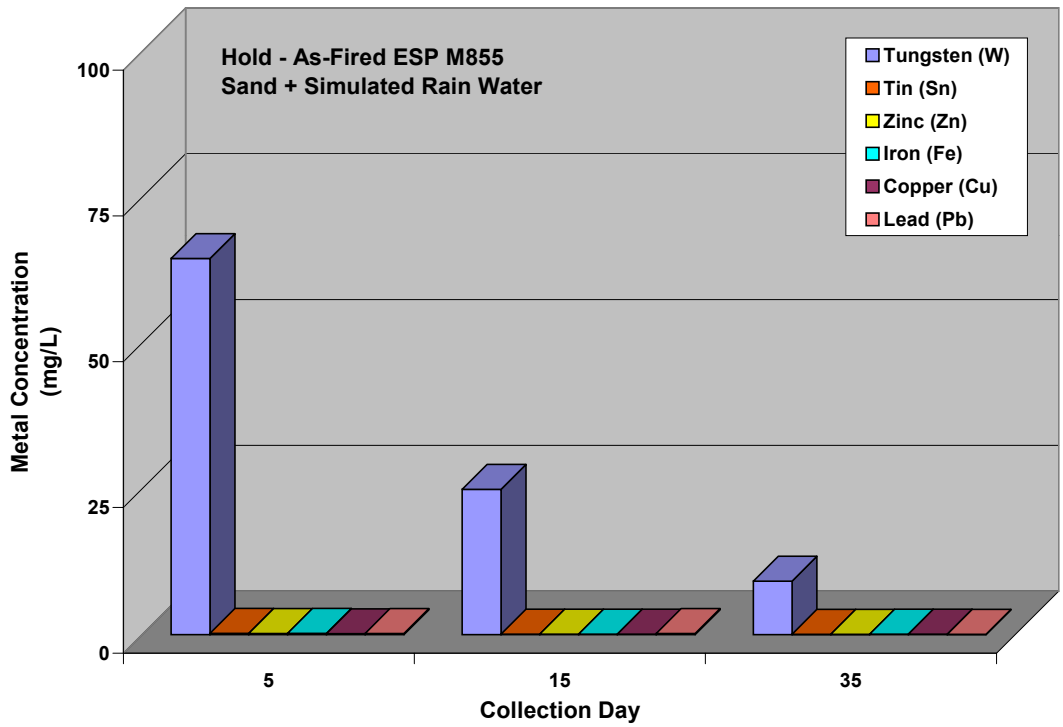

Figure J-9. Metal concentrations in leachate for the ESP M855 sample from the hold test using sand and rain water.

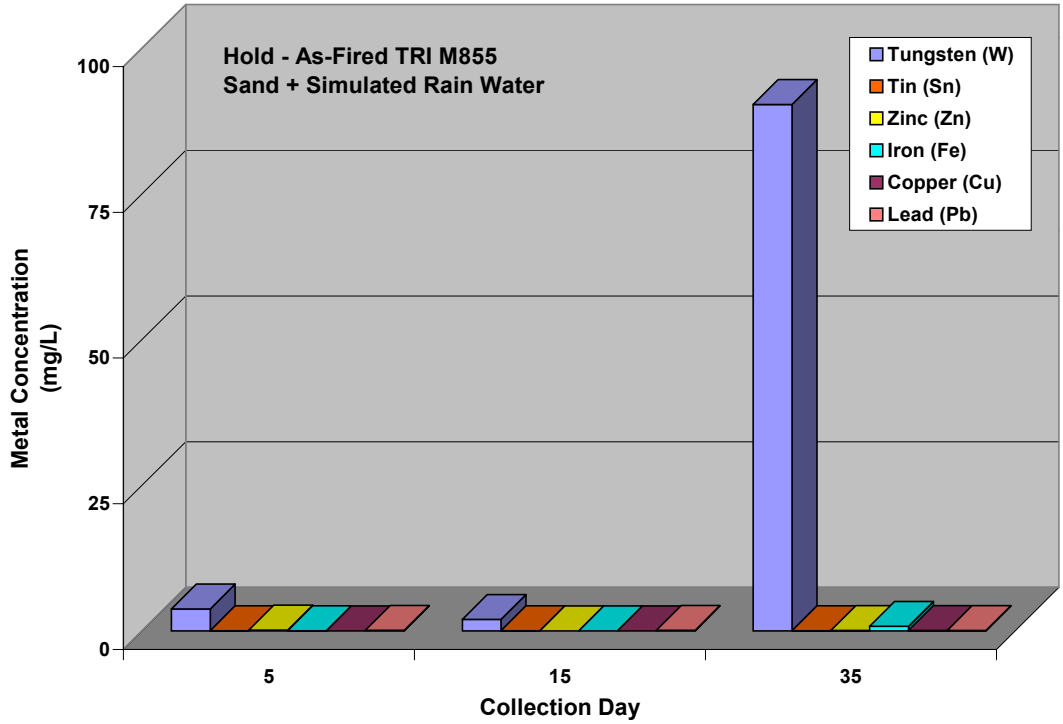

Figure J-10. Metal concentrations in leachate for the TRI M855 sample from the hold test using sand and rain water.

SPECIAL RUN K. Bullets Fired Into Soil

Media: *Soil*
Solvent: *Rain water*
Technique(s): Up-Flow and Hold
Bullet types: Lead M855
ESP-1 M855
TRI-1 M855

Summary: Results same as simulated materials. See Section E.

Table K-1. Metals Analysis: Up-Flow - Soil + Simulated Rain Water
As-fired projectiles
(Concentrations in mg/L)

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
1,2,4,8	0.05	0.05	0.005	0.01	0.02	0.02
16,32	0.03	0.02	0.01	0.02	0.01	0.05

Control

Day	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.05	0.322	0.074	< 0.02	< 0.02
2	< 0.05	< 0.05	0.058	0.030	< 0.02	< 0.02
4	< 0.05	< 0.05	0.019	0.454	< 0.02	< 0.02
8	< 0.05	< 0.05	0.048	0.906	< 0.02	< 0.02
16	< 0.03	< 0.02	0.025	1.35	< 0.01	< 0.05
32	< 0.03	< 0.02	0.006	5.85	< 0.01	< 0.05

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	0.247	< 0.05	0.1260	0.041	0.0915	0.0438
2	0.510	< 0.05	0.0392	2.99	0.1100	0.0593
4	< 0.05	< 0.05	0.1250	11.7	0.3540	0.0766
8	< 0.05	< 0.05	0.0407	2.82	0.1240	0.0338
16	0.274	< 0.02	0.0330	1.71	0.0854	< 0.05
32	0.146	< 0.02	0.0328	2.72	0.0714	< 0.05

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	1.00	< 0.05	0.1670	0.102	0.0661	0.0326
2	5.37	< 0.05	0.0293	0.217	0.0293	0.0200
4	5.09	< 0.05	0.0257	0.107	0.0258	0.0191
8	3.11	< 0.05	0.0193	0.498	0.0328	0.0140
16	2.56	< 0.02	0.0677	4.54	0.1590	< 0.05
32	0.67	< 0.02	0.0473	3.55	0.1110	< 0.05

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	0.0539	< 0.05	0.3080	0.0171	0.0598	0.0599
2	< 0.05	< 0.05	0.0638	0.9710	0.0717	0.1180
4	< 0.05	< 0.05	0.0887	0.6390	0.2610	0.5830
8	< 0.05	< 0.05	0.0364	0.3280	0.0304	0.0355
16	< 0.03	< 0.02	0.0527	3.24	0.1040	0.1870
32	< 0.03	< 0.02	0.0192	4.07	0.1090	0.1300

Table K-2. Metals Analysis: Hold - Soil + Simulated Rain Water
As-fired projectiles
Concentrations in mg/L

Detection limits

Day	W	Sn	Zn	Fe	Cu	Pb
5,20	0.05	0.05	0.005	0.01	0.02	0.02
35	0.03	0.02	0.01	0.02	0.01	0.05

Empty (control)

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.42	0.063	< 0.02	< 0.02
15	< 0.05	< 0.05	0.424	0.035	< 0.02	< 0.02
35	< 0.03	< 0.02	0.409	0.281	< 0.01	< 0.05

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.942	0.583	0.124	0.0632
15	0.15	< 0.05	0.651	0.056	0.0599	0.0967
35	0.11	0.0213	0.543	0.269	0.0426	< 0.05

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	21	< 0.05	0.877	0.727	0.194	0.0645
15	14.4	< 0.05	0.676	0.712	0.134	0.0984
35	18	0.0255	0.404	1.21	0.101	< 0.05

Lead M855

Day	W	Sn	Zn	Fe	Cu	Pb
5	< 0.05	< 0.05	0.325	0.175	0.0442	0.148
15	< 0.05	< 0.05	0.22	0.115	0.0385	0.122
35	0.103	0.0231	0.174	0.0756	0.0407	< 0.05

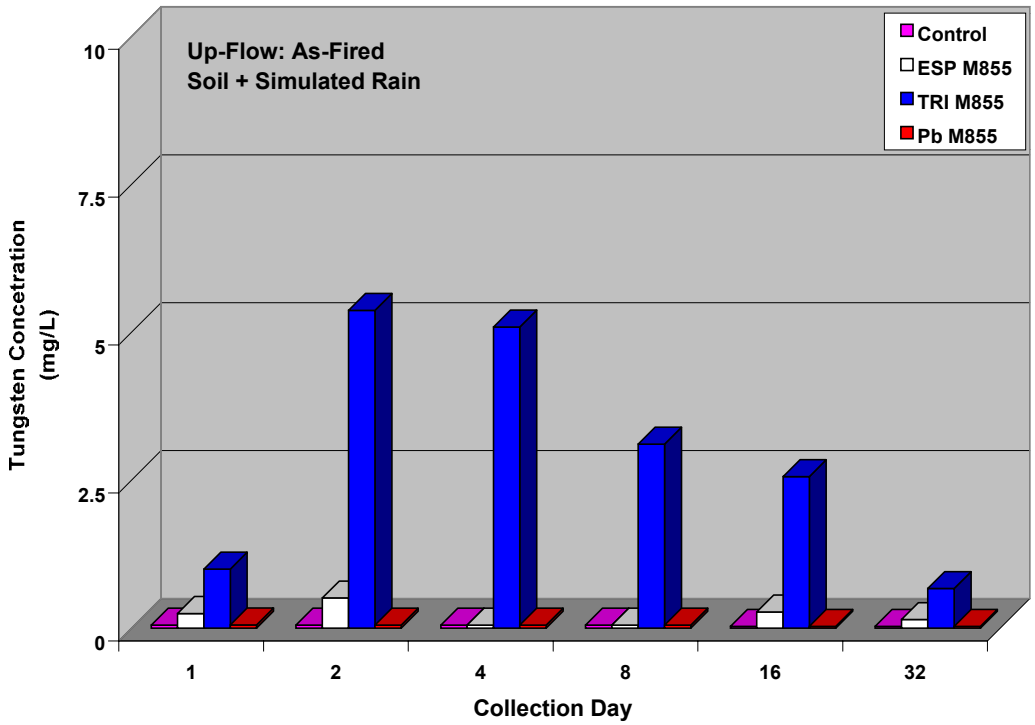

Figure K-1. Tungsten concentration in leachate for up-flow test using soil and rain water.

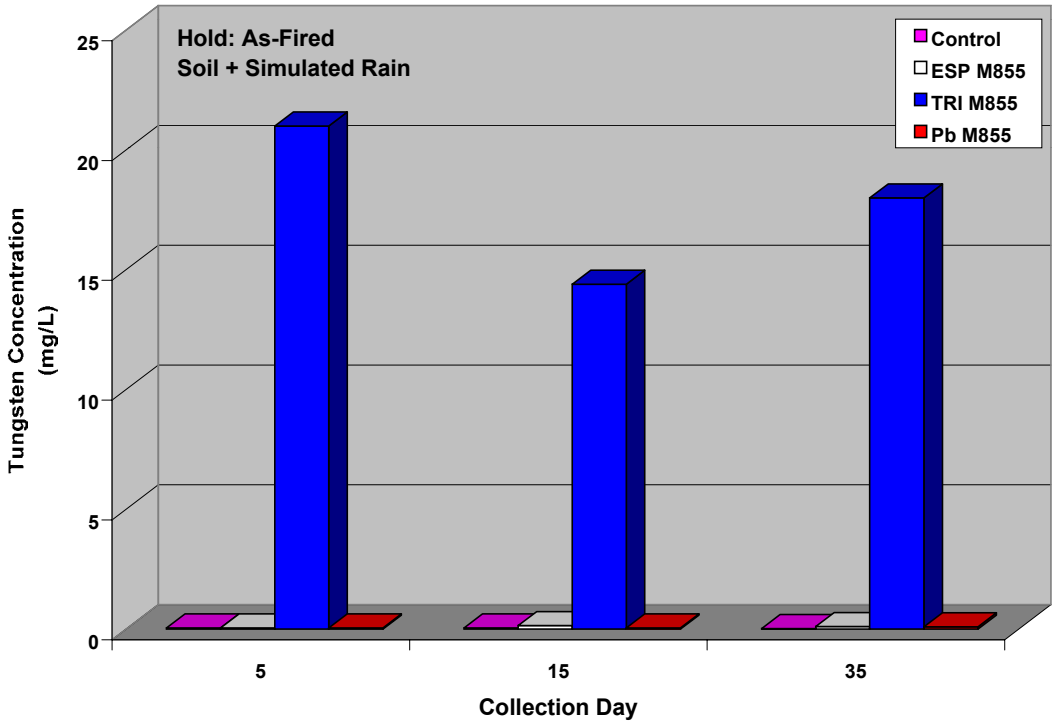

Figure K-2. Tungsten concentration in leachate for hold test using soil and rain water

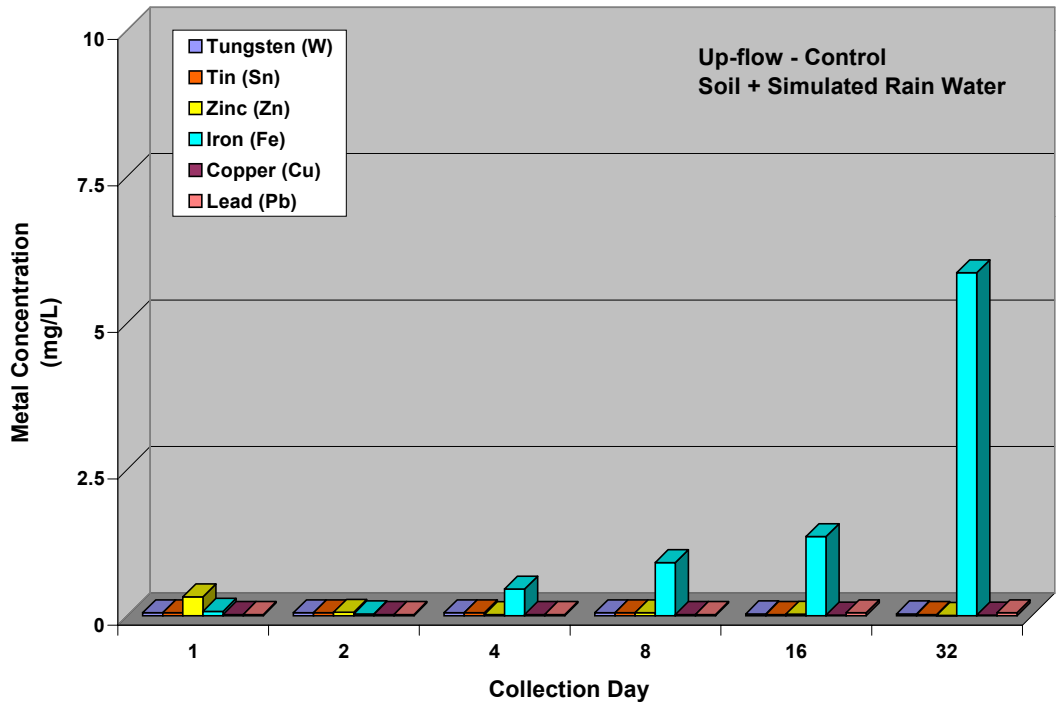

Figure K-3. Metal concentrations in leachate for control sample from the up-flow test using soil and rain water.

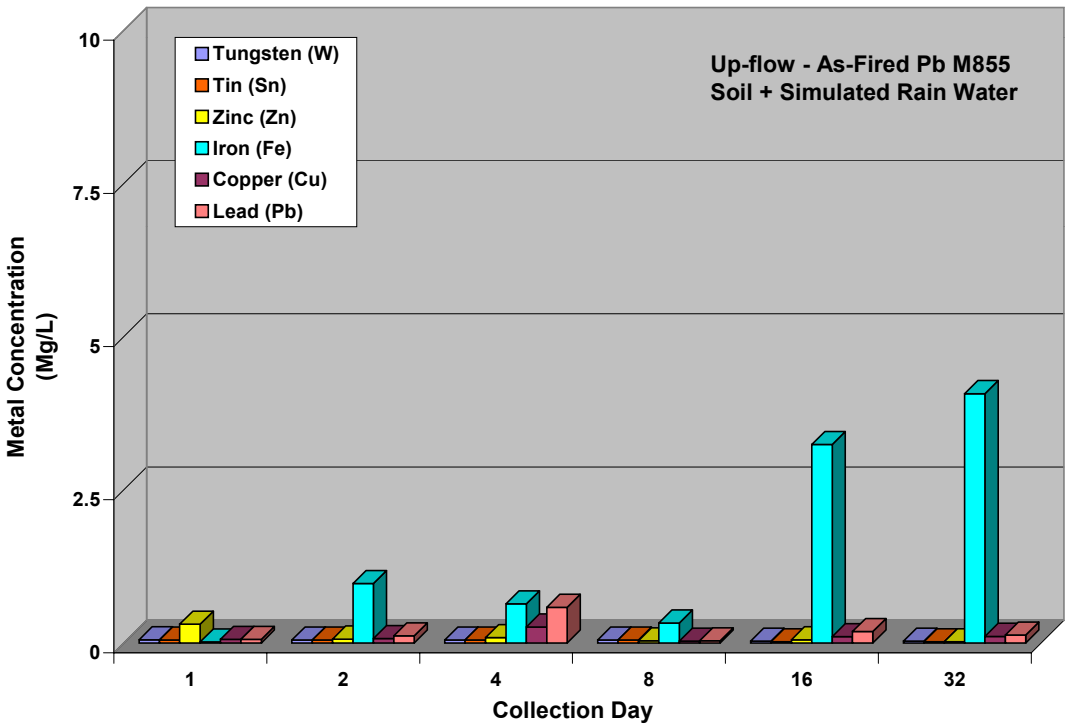

Figure K-4. Metal concentrations in leachate for lead M855 sample from the up-flow test using soil and rain water.

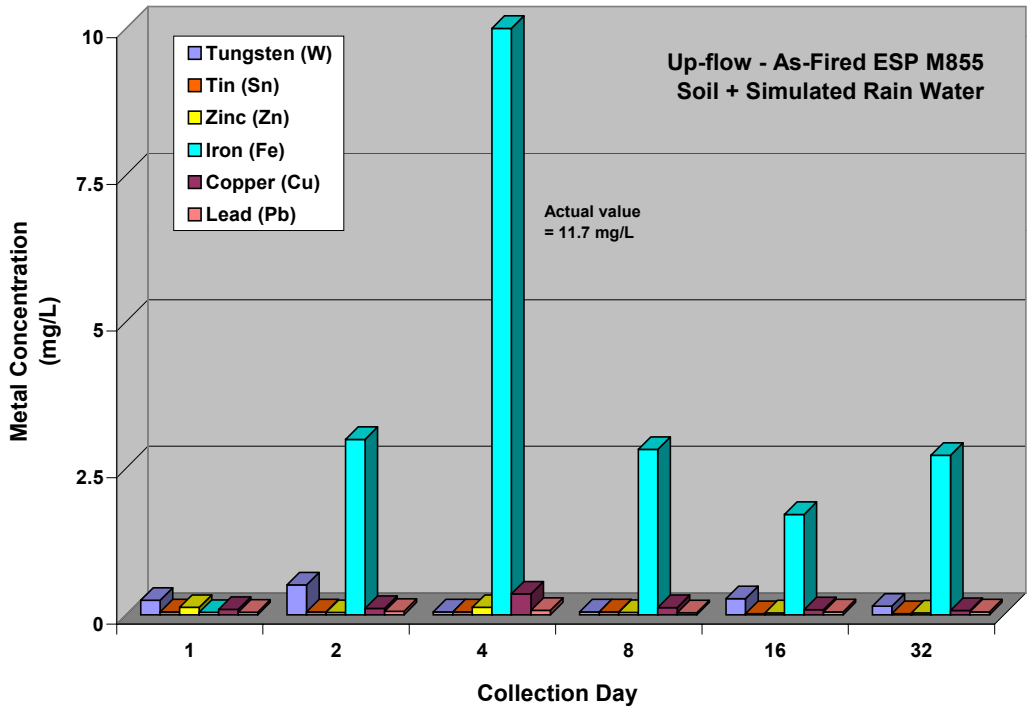

Figure K-5. Metal concentrations in leachate for ESP M855 sample from the up-flow test using soil and rain water.

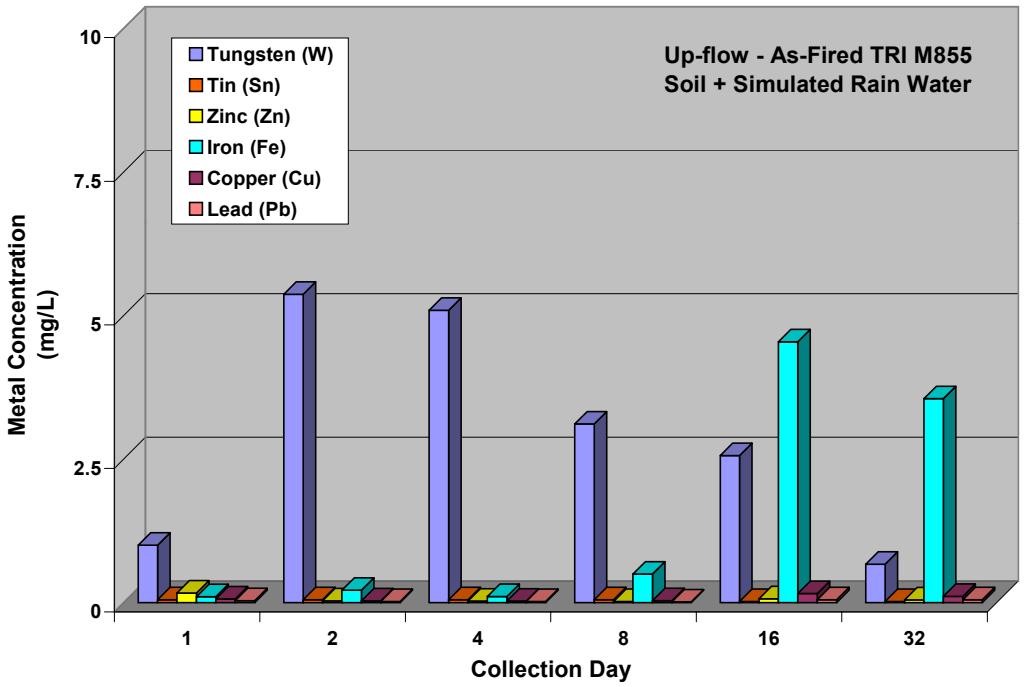

Figure K-6. Metal concentrations in leachate for TRI M855 sample from the up-flow test using soil and rain water

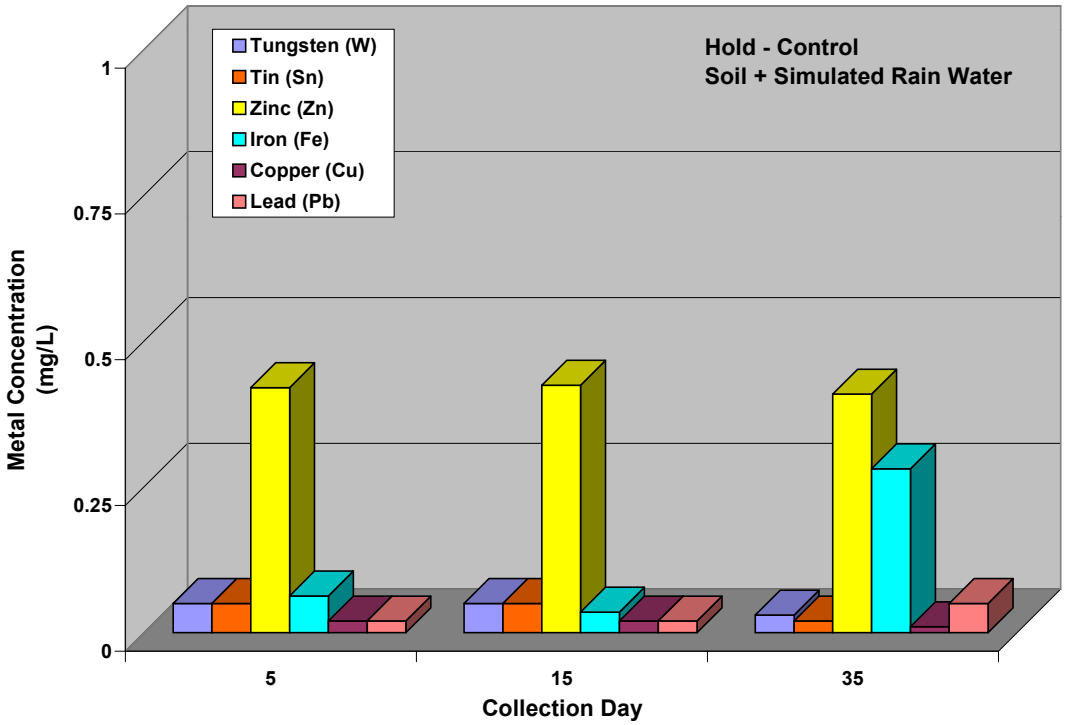

Figure K-7. Metal concentrations in leachate for the control sample from the hold test using soil and rain water.

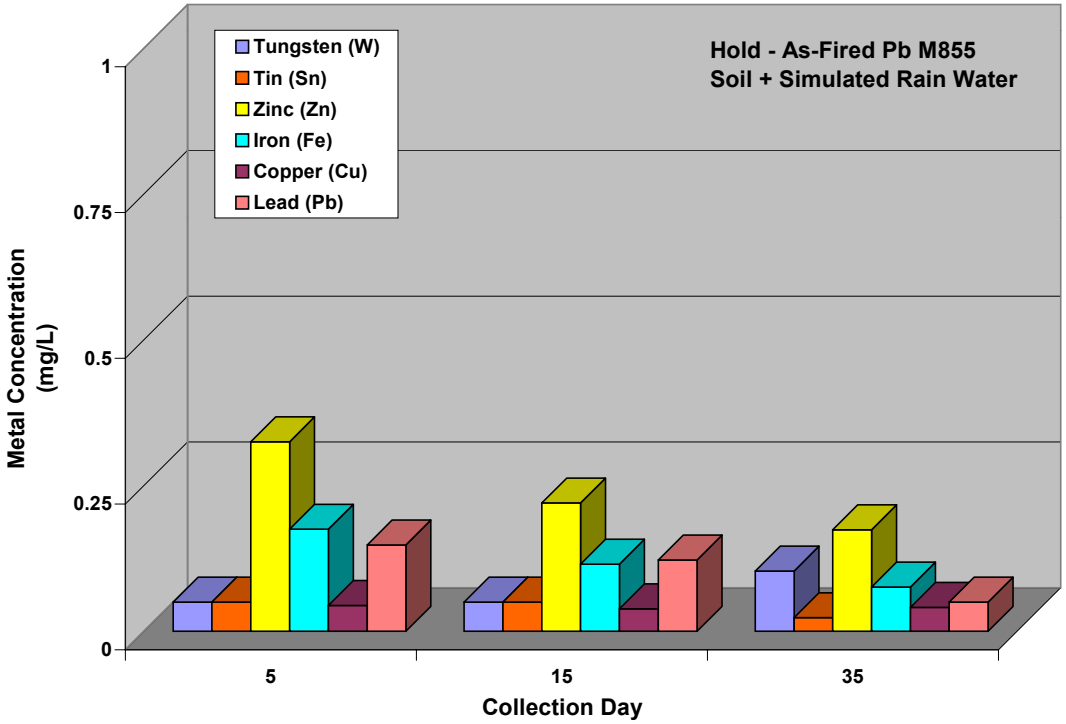

Figure K-8. Metal concentrations in leachate for lead M855 mm sample from the hold test using soil and rain water.

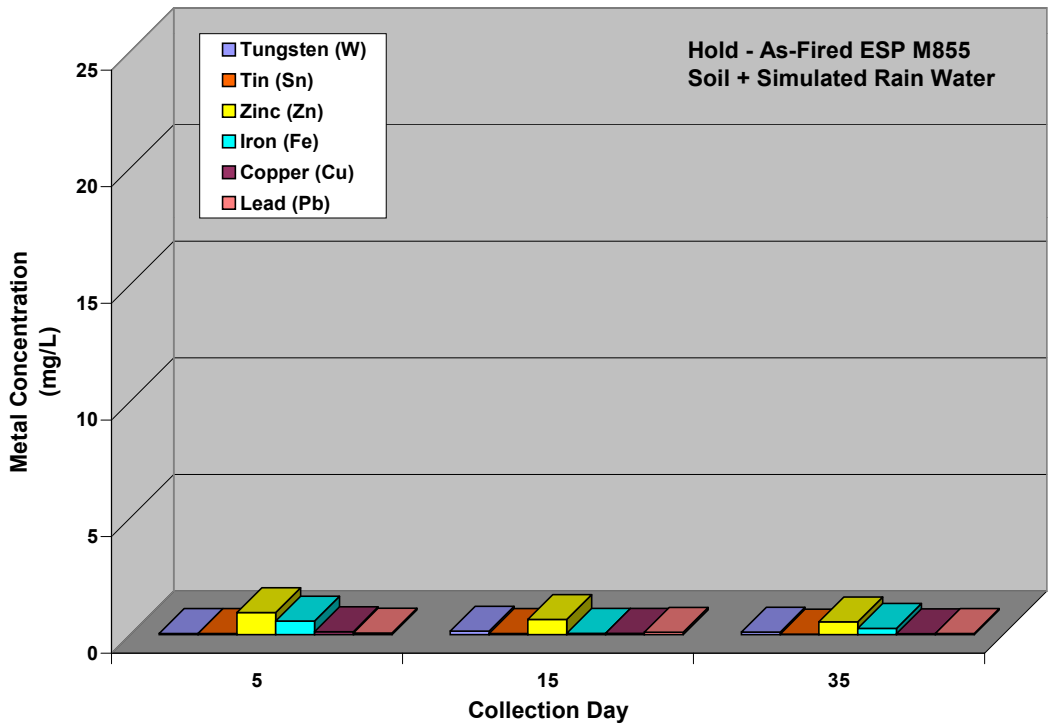

Figure K-9. Metal concentrations in leachate for the ESP M855 sample from the hold test using soil and rain water.

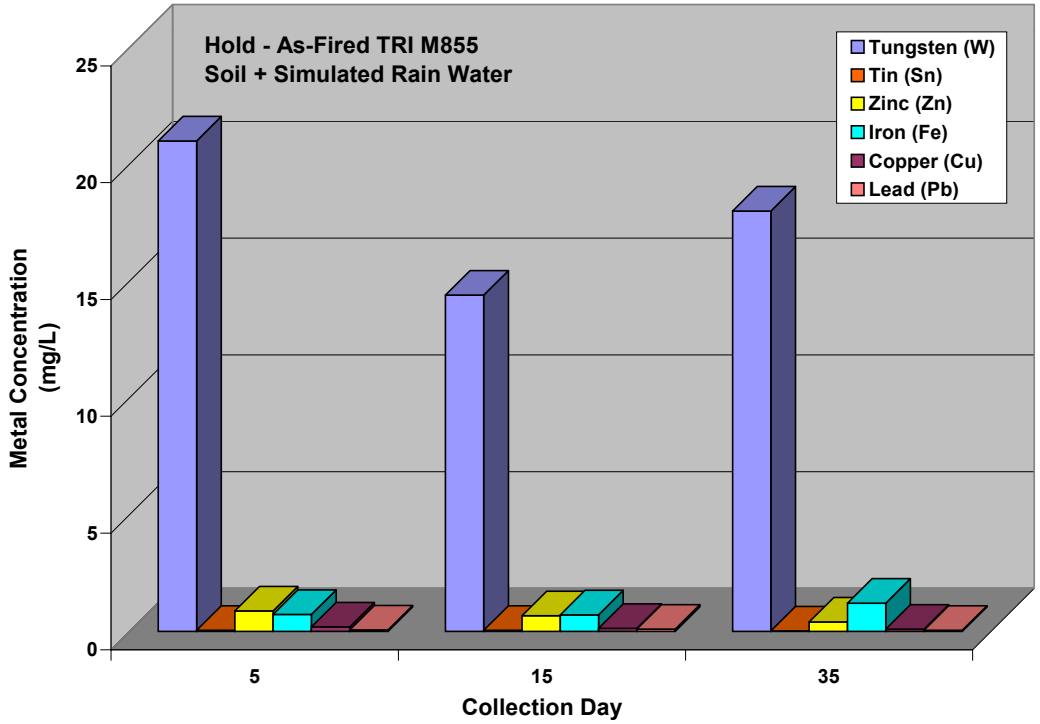

Figure K-10. Metal concentrations in leachate for the TRI M855 sample from the hold test using soil and rain water.

AGING STUDIES L. Simulated Environment

Media: *Sand*
Solvent: *Rain water*
Technique(s): Up-Flow
Bullet types: ESP-1 M855
TRI-1 M855

Summary: See Sections A and M. The high concentrations of tungsten in the leachant were likely due to small particles washing out of the sand bed and through the filter paper.

Table L-1. Up-Flow By Column: Sand + Simulated Rain Water
TRI and ESP M855 accelerated aging study
(Concentrations in mg/L)

Description: Bullet materials added to sand and aged in a moist, heated environment. Deionized water added to mixture to keep "wet" and held at 130°C for 48 days. Mixture then added to center section of column and leached.

Material	Mass
Sand	500 g
Jacket	80 g
Steel Penetrator	40 g
Core	130 g
No. equivalent	62 bullets

Detection limits

W	Sn	Zn	Fe	Cu	Pb
0.05	0.05	0.02	0.03	0.02	0.05

ESP-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	7950	368	30.2	201	49.2	2.6
2	4560	200	16.6	110	26.8	1.45
4	1950	88	0.739	45.7	10.1	0.622
8	105	27.6	2.17	12.6	2.37	0.151
16	33.1	6.3	0.541	2.47	0.471	0.0961
32	35.9	2.12	0.18	0.808	0.142	< 0.05

TRI-1 M855

Day	W	Sn	Zn	Fe	Cu	Pb
1	9510	2.95	35.5	175	29.7	3.35
2	6150	1.75	20.6	104	14.5	1.99
4	1260	0.362	3.75	22.8	1.89	0.365
8	96.9	0.0644	0.476	3.04	0.259	0.0478
16	26.7	< 0.05	0.17	1.05	0.0747	< 0.05
32	19.1	< 0.05	0.055	0.39	0.0321	< 0.05

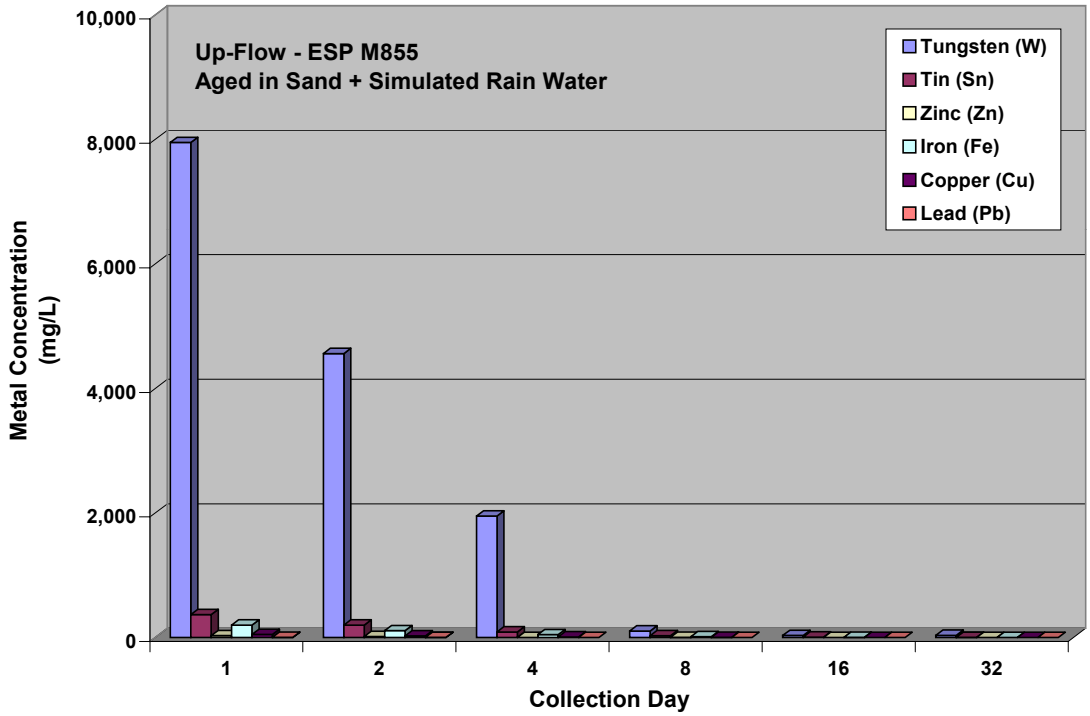

Figure L-1. Metal concentrations in leachate for ESP M855 sample from the up-flow test using sand and rain water.

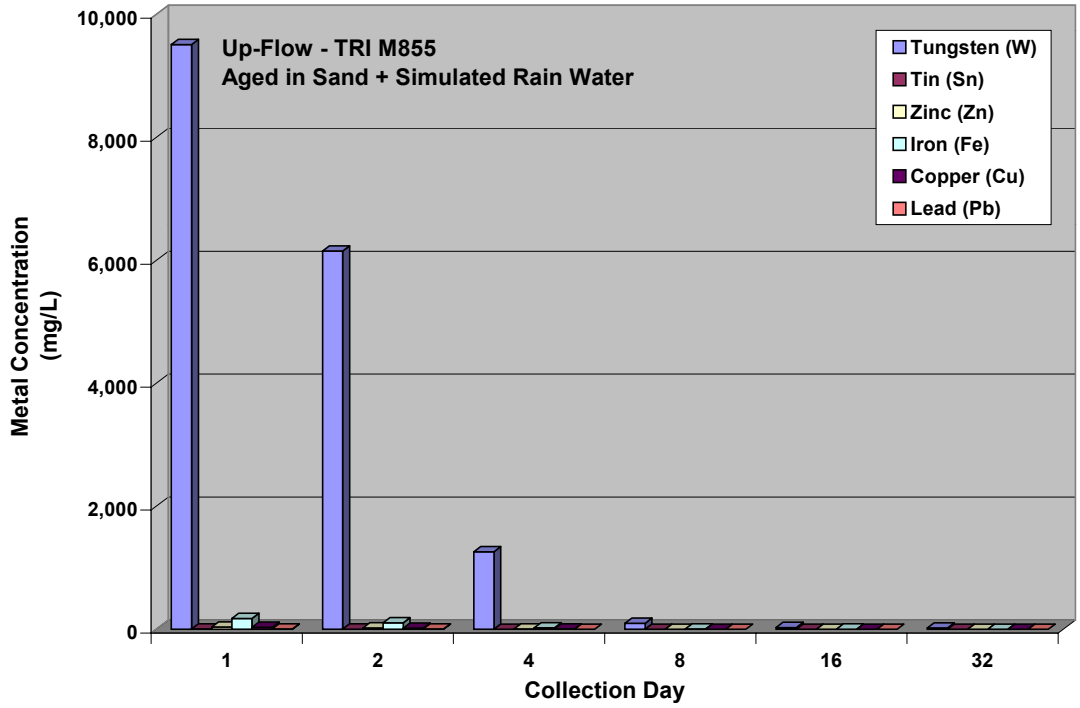

Figure L-2. Metal concentrations in leachate for TRI M855 sample from the up-flow test using sand and rain water

AGING STUDIES M. Simulated Aged Materials

Media: *Sand*
Solvent: *Rain water*
Technique(s): Up-Flow
Bullet types: not applicable
Materials: Metal powders

Summary: In this study, tungsten oxide and tungstic acid in different ratios were substituted for the tungsten powder in the tungsten-tin bullet material to simulate the corrosion of tungsten in a moist environment. High concentrations of tungsten were found in the leachate from columns with the mixtures containing tungstic acid. This was not expected for both WO_3 and H_2WO_4 are insoluble in water or acids.

It was hypothesized that the particle size of the tungstic acid might allow material to be washed though the columns and filter media. Particle size analysis found that 10% of the tungstic acid particles were between 0.1 and 0.5 microns in diameter. Sand is a very porous media and the filter paper used in the leaching columns has a pore size of 0.7 microns. This combination would allow the small tungstic acid particles to pass into the leachate.

Table M-1. Up-Flow By Column: Sand + De-ionized Water
Simulated aging using mixtures of metal and oxide powders
(Concentrations in mg/L)

All weights in grains

Mixture	W	Sn	Oxide	Acid	Total
Pure Metal	306.8	213.2	0.0	0.0	520.0
50/50 Oxide	153.4	213.2	193.4	0.0	560.0
All Oxide	0.0	213.2	386.8	0.0	600.0
50/50 Acid	153.4	213.2	0.0	208.4	575.0
All Acid	0.0	213.2	0.0	416.8	630.0
50/25/25 Met/Ox/Ac	153.4	213.2	96.7	104.2	567.5

Detection limits

W	Sn	Zn	Fe	Cu	Pb
0.1	0.01	0.005	0.01	0.003	0.01

De-ionized water

W	Sn	Zn	Fe	Cu	Pb
< 0.1	< 0.01	< 0.005	0.0105	0.004	0.0123

Tungstic Acid - Tin

Collection Day	W	Sn	Zn	Fe	Cu	Pb
1	2660	4.35	0.743	4.38	0.0313	0.275
2	1090	3.12	0.505	2.83	0.0213	0.177
4	223	1.21	0.129	0.821	0.0047	0.0838
8	47.5	0.357	0.0344	0.211	< 0.003	0.0622
16	12	0.082	0.0153	0.051	0.0039	0.0454
32	6.19	0.02	0.0071	0.09	< 0.003	0.011

Tungsten Oxide - Tin

Collection Day	W	Sn	Zn	Fe	Cu	Pb
1	22.7	0.015	0.0162	0.0134	0.0073	0.0389
2	18.3	0.0151	0.0185	0.033	0.0034	0.0416
4	11.6	0.012	0.0112	0.0139	0.0032	0.0347
8	6.79	0.011	0.0107	0.0264	< 0.003	0.0267
16	5	< 0.01	0.0065	0.0179	< 0.003	0.0266
32	9.14	< 0.01	0.0091	0.0126	< 0.003	0.0159

Tungsten - Tin

Collection Day	W	Sn	Zn	Fe	Cu	Pb
1	15.8	0.0107	0.0279	0.0564	0.007	0.0224
2	11	< 0.01	0.0188	0.0127	0.0044	0.0236
4	11	< 0.01	0.0107	0.0552	< 0.003	0.0191
8	10.8	< 0.01	0.006	0.0362	< 0.003	0.0189
16	5.88	0.025	0.0073	0.0355	< 0.003	0.0258
32	9.03	< 0.01	0.0226	0.0243	< 0.003	0.0111

Table M-1. Up-Flow By Column: Sand + De-ionized Water (continued)

Simulated aging using mixtures of metal and oxide powders

(Concentrations in mg/L)

50 W + 50 WO3 - Tin

Collection Day	W	Sn	Zn	Fe	Cu	Pb
1	10.3	0.0124	0.131	0.257	0.0154	0.038
2	27.6	0.0103	0.0506	0.2	0.0056	0.0325
4	18.1	0.0104	0.0257	0.0161	0.0046	0.0314
8	7.48	0.0106	0.0126	0.0203	0.0038	0.0301
16	7.44	0.0163	0.0061	0.0179	< 0.003	0.0272
32	8.59	< 0.01	0.0075	0.0111	< 0.003	0.0156

50 W + 50 H2WO4 - Tin

Collection Day	W	Sn	Zn	Fe	Cu	Pb
1	462	0.741	0.14	0.253	0.0127	0.0765
2	403	1.99	0.104	0.713	0.01	0.0691
4	137	1.37	0.0422	0.695	0.0053	0.0478
8	44	0.485	0.0186	0.476	0.0038	0.0328
16	16.3	0.183	0.0196	0.186	0.0498	0.0186
32	8.97	0.0462	0.0076	0.13	< 0.003	0.014

50 W + 25 WO3 + H2WO4 - Tin

Collection Day	W	Sn	Zn	Fe	Cu	Pb
1	198	0.858	0.096	0.16	0.0075	0.0368
2	192	1.56	0.0724	0.467	0.0057	0.0308
4	62.4	0.9	0.0275	0.609	0.003	0.0208
8	11.5	0.165	0.0626	0.206	0.0823	0.014
16	4.44	0.0144	0.0128	0.0666	0.003	0.01
32	6.64	0.0141	0.0093	0.0253	0.003	0.0167

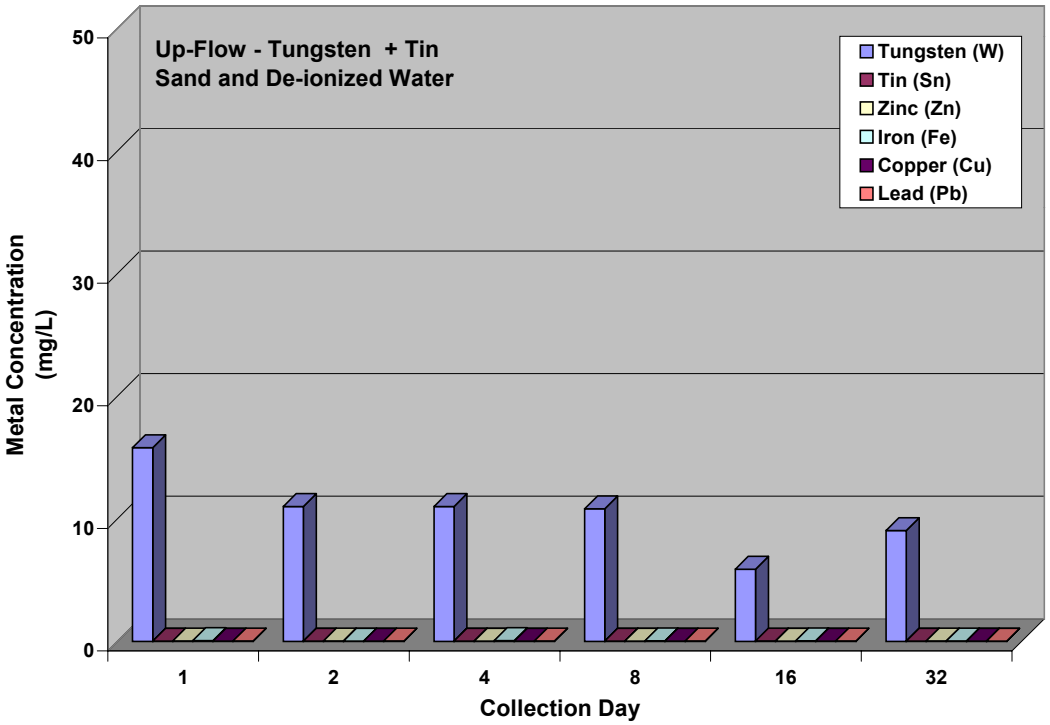

Figure M-1. Metal concentrations in leachate for tungsten + tin sample from the up-flow test using sand and rain water.

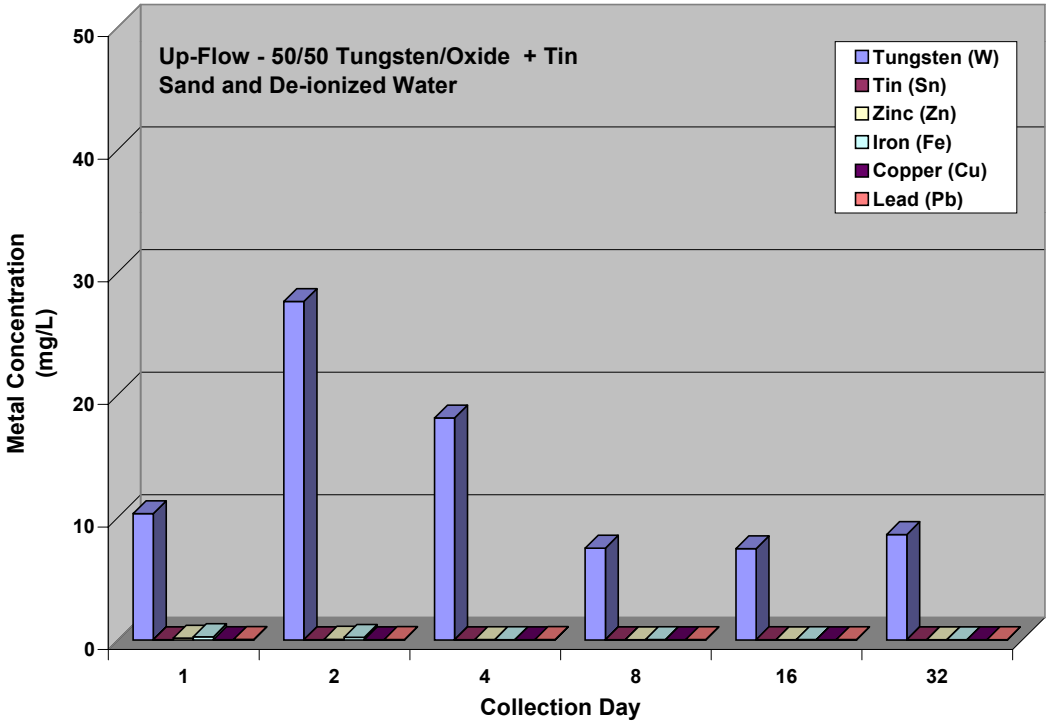

Figure M-2. Metal concentrations in leachate for 50/50 tungsten/tungsten oxide + tin sample from the up-flow test using sand and rain water.

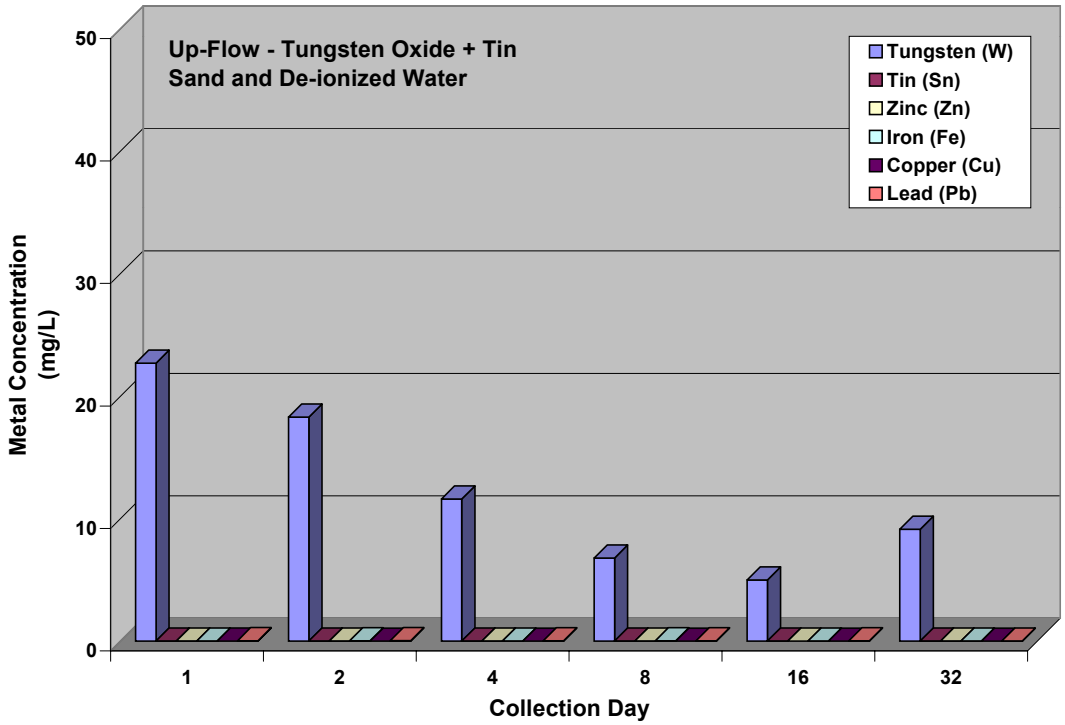

Figure M-3. Metal concentrations in leachate for tungsten oxide + tin sample from the up-flow test using sand and rain water.

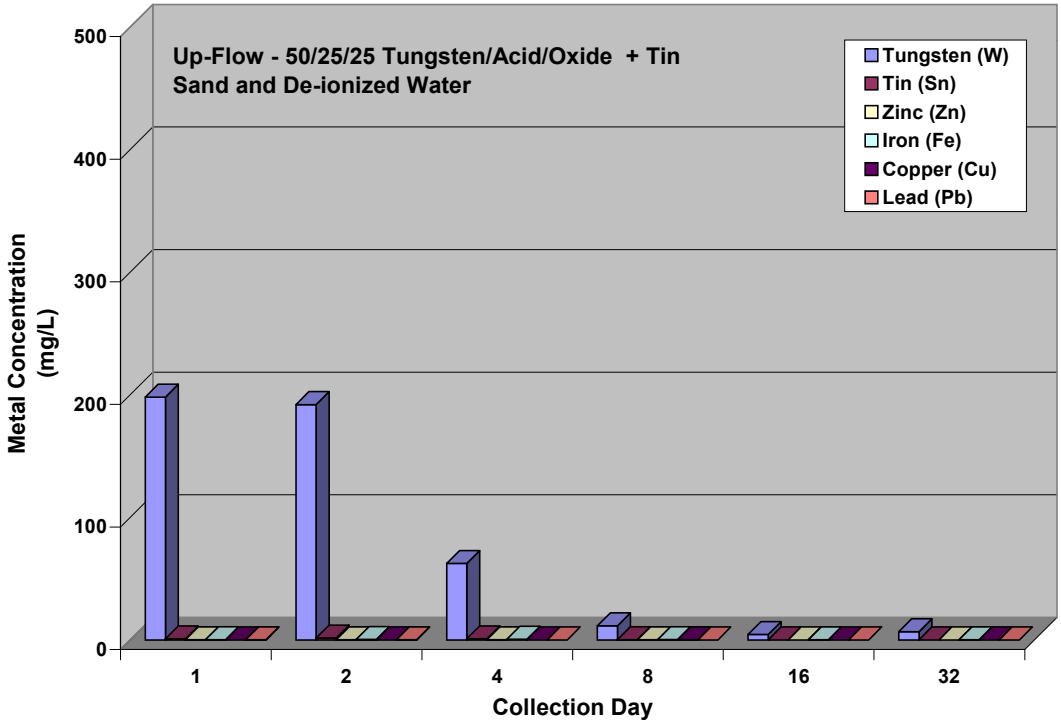

Figure M-4. Metal concentrations in leachate for 50/25/25 tungsten/tungstic acid/tungsten oxide + tin sample from the up-flow test using sand and rain water.

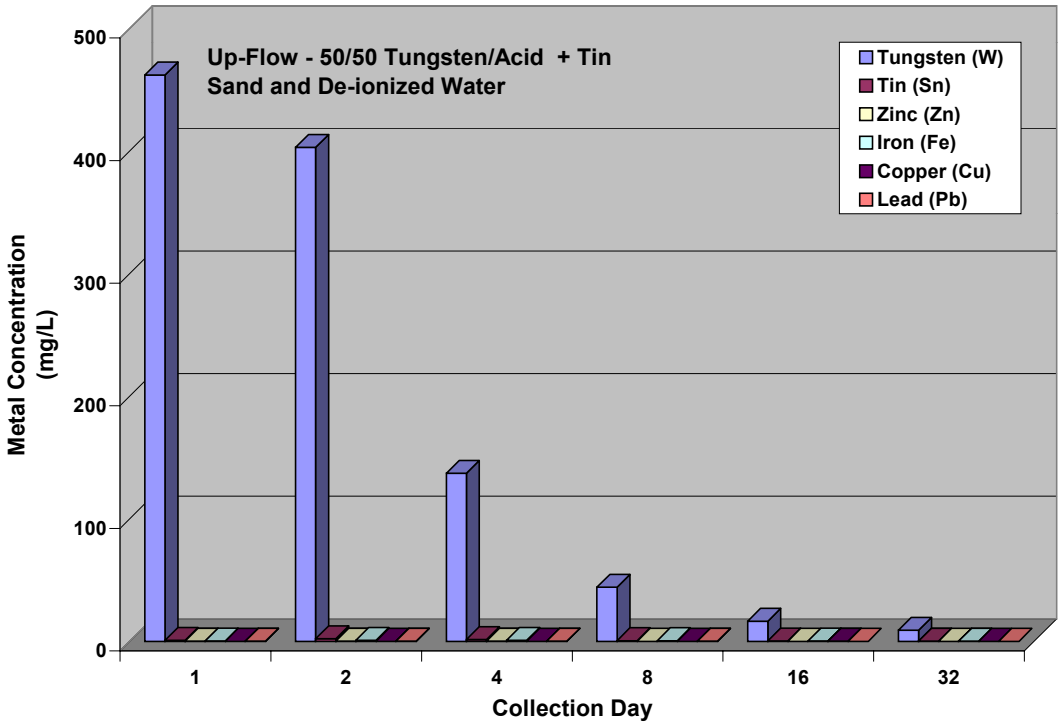

Figure M-5. Metal concentrations in leachate for 50/50 tungsten/tungstic acid + tin sample from the up-flow test using sand and rain water.

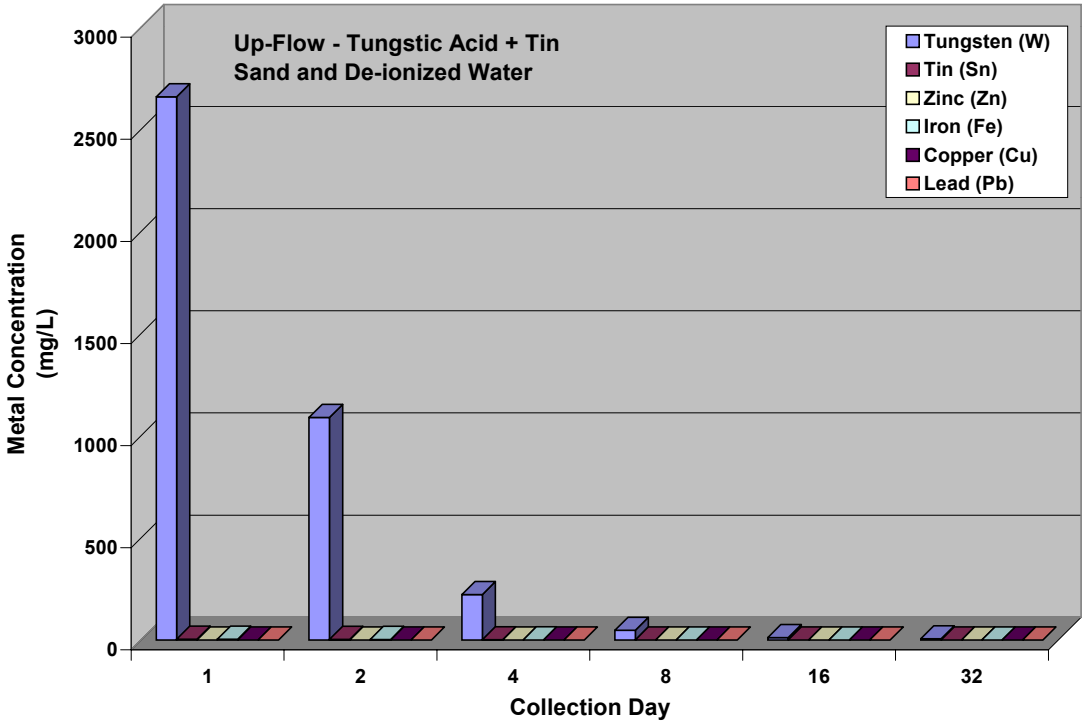

Figure M-6. Metal concentrations in leachate for tungstic acid + tin sample from the up-flow test using sand and rain water.

AGING STUDIES N. Outdoor Extended Exposures

Media: *Sand*
Solvent: *Rain water*
Technique(s): Hold
Bullet types: Lead M855
ESP-1 M855
TRI-1 M855

Summary:

Table N-1. Outdoor exposure: Sand + Simulated Rain

Metals Analysis: Hold - 5 day leaching
(Concentrations in mg/L)

Detection limits

Month	W	Sn	Zn	Fe	Cu	Pb
1	0.05	0.05	0.01	0.03	0.02	0.02
Remainder	0.03	0.02	0.01	0.05	0.01	0.02

Control (Soil Only)

Month	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	0.107	0.793	90.8	0.0693	0.355
3	< 0.03	0.0205	0.12	19.8	0.0348	0.0507
6	< 0.03	0.0289	0.17	48.6	0.0396	0.114
9	< 0.03	< 0.02	0.0203	2.9	0.0141	0.0571
12	< 0.03	0.142	0.0472	6.02	0.0178	0.0684

ESP-1 M855

Month	W	Sn	Zn	Fe	Cu	Pb
1	0.968	0.0892	0.339	34.4	0.0919	0.148
3	1.08	0.0615	0.203	31.8	0.0773	0.0624
6	11.3	0.667	1.09	96.8	0.591	0.215
9	209	3.99	8.86	1210	8.32	1.79
12	50.2	1.83	3.9	677	3.35	0.923

TRI-1 M855

Month	W	Sn	Zn	Fe	Cu	Pb
1	39.2	0.617	4.2	78.8	2.21	1.81
3	< 0.03	0.104	0.82	138	0.56	0.371
6	15.5	0.124	2.02	350	2.35	0.623
9	206	1.12	9.66	2250	10.9	2.87
12	67.6	0.509	8.09	1880	7.55	2.33

Lead M855

Month	W	Sn	Zn	Fe	Cu	Pb
1	< 0.05	< 0.05	0.226	147	0.0192	1.87
3	< 0.03	< 0.02	0.0464	94.7	0.0173	1.55
6	< 0.03	< 0.02	0.0452	95.1	0.0208	1.65
9	< 0.03	0.0253	0.0573	110	0.0314	1.71
12	< 0.03	0.0227	0.0334	47.6	0.0239	1.19

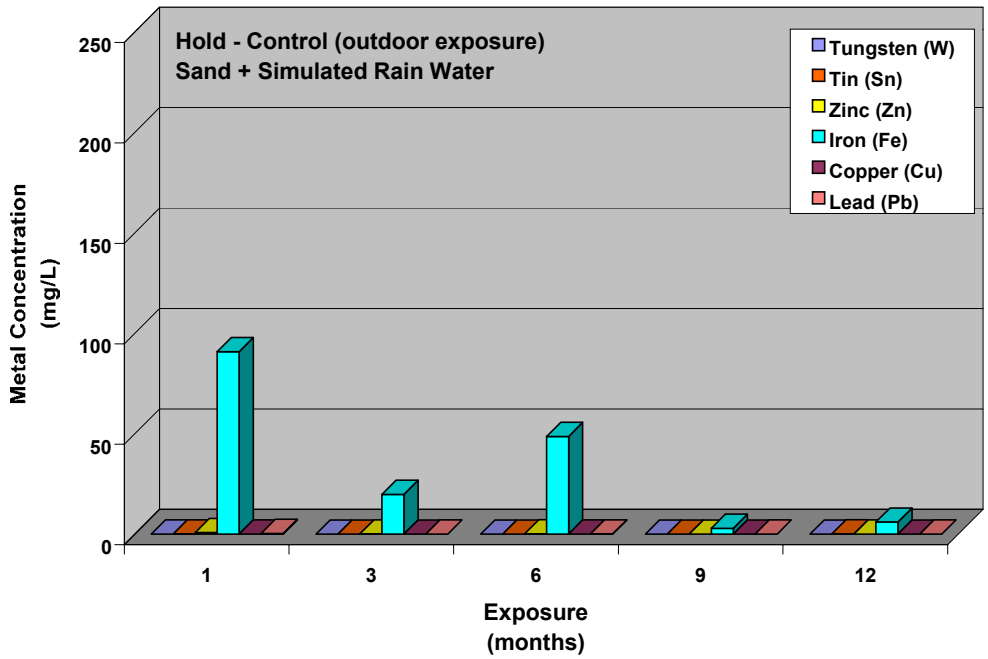

Figure N-1. Metal concentrations in leachate for control sample from the hold test using sand and rain water.

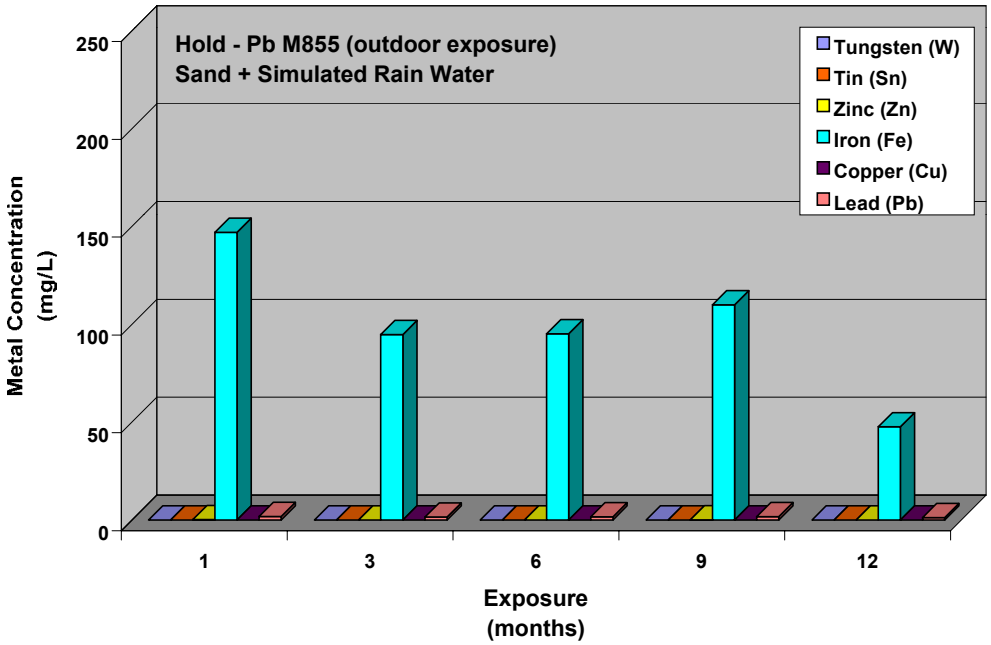

Figure N-2. Metal concentrations in leachate for lead M855 sample from the hold test using sand and rain water.

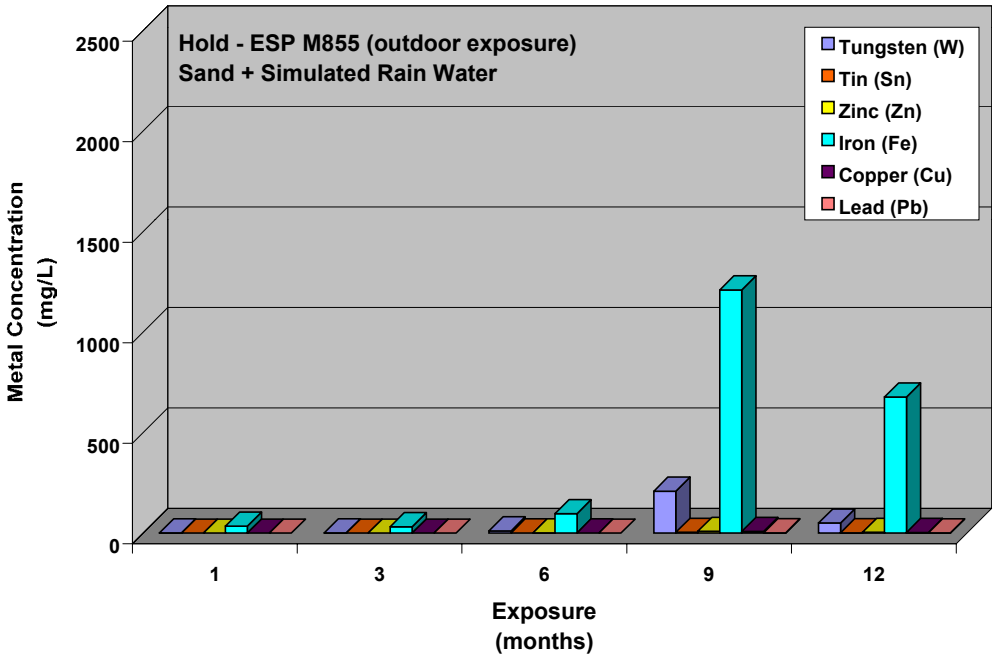

Figure N-3. Metal concentrations in leachate for ESP M855 sample from the hold test using sand and rain water.

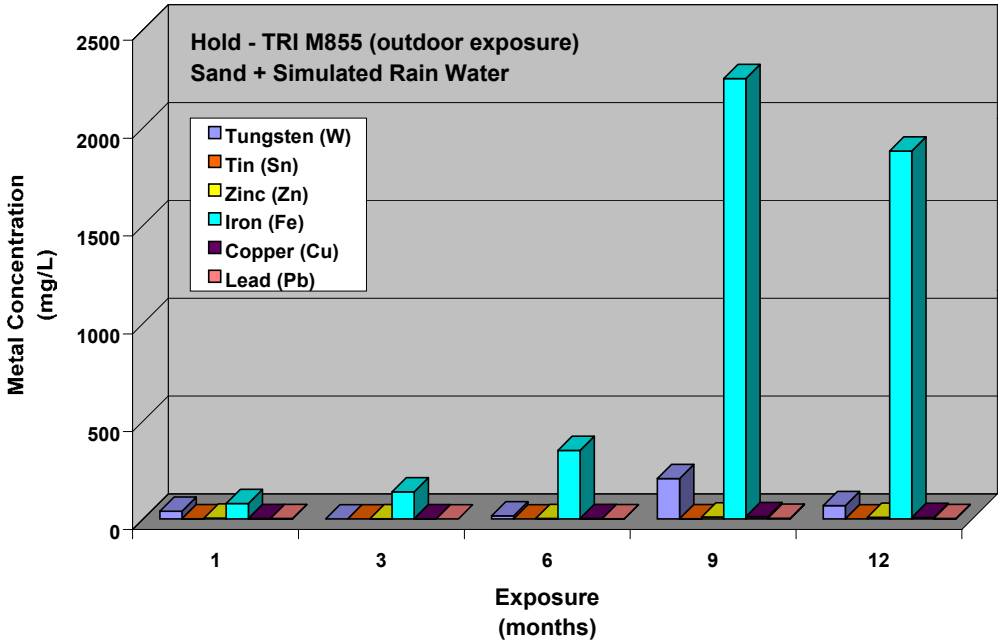

Figure N-4. Metal concentrations in leachate for TRI M855 sample from the hold test using sand and rain water.

AGING STUDIES O. Outdoor Extended Exposures

Media: *Soil*
Solvent: *Rain water*
Technique(s): Up-Flow and Hold
Bullet types: Lead M855
ESP-1 M855
TRI-1 M855

Summary:

Table O-1. Outdoor exposure: Soil + Simulated Rain
Metals Analysis: Hold - 5 day leaching
Concentrations in mg/L

Detection limits

Month	W	Sn	Zn	Fe	Cu	Pb
1	0.05	0.05	0.01	0.03	0.02	0.02
Remainder	0.03	0.02	0.01	0.05	0.01	0.02

Control (Sand Only)

Month	W	Sn	Zn	Fe	Cu	Pb
1	0.57	< 0.05	0.0148	0.033	< 0.02	< 0.02
3	0.03	< 0.02	1.21	24.5	0.0399	0.106
6	1.17	0.0209	1.36	23.5	0.042	0.105
9	< 0.03	0.0361	1.66	42	0.0608	0.202
12	< 0.03	0.0303	0.932	29.8	0.0427	0.155

ESP-1 M855

Month	W	Sn	Zn	Fe	Cu	Pb
1	137	< 0.05	0.0401	< 0.05	< 0.02	0.067
3	4.25	0.299	0.858	17.2	0.0722	0.0573
6	5.35	0.29	0.422	5.33	0.0456	0.0299
9	5.49	0.357	0.479	5.8	0.0444	0.0425
12	5.93	0.69	0.917	24.1	0.0952	0.133

TRI-1 M855

Month	W	Sn	Zn	Fe	Cu	Pb
1	126	< 0.05	0.0469	0.104	< 0.02	0.0652
3	< 0.03	0.0668	0.763	18.7	0.529	0.216
6	12.8	0.0473	1.04	34.3	0.802	0.183
9	85.4	0.0448	0.527	21.4	0.405	0.145
12	5.99	0.0276	0.313	15.3	0.109	0.0953

Lead M855

Month	W	Sn	Zn	Fe	Cu	Pb
1	0.38	< 0.05	0.0144	0.091	< 0.02	0.0368
3	< 0.03	< 0.02	0.6	25.7	0.0586	5.42
6	< 0.03	0.0388	0.843	31.2	0.0968	7.66
9	< 0.03	0.0285	0.738	23.2	0.0929	6.38
12	< 0.03	0.0241	0.294	12.6	0.0489	2.35

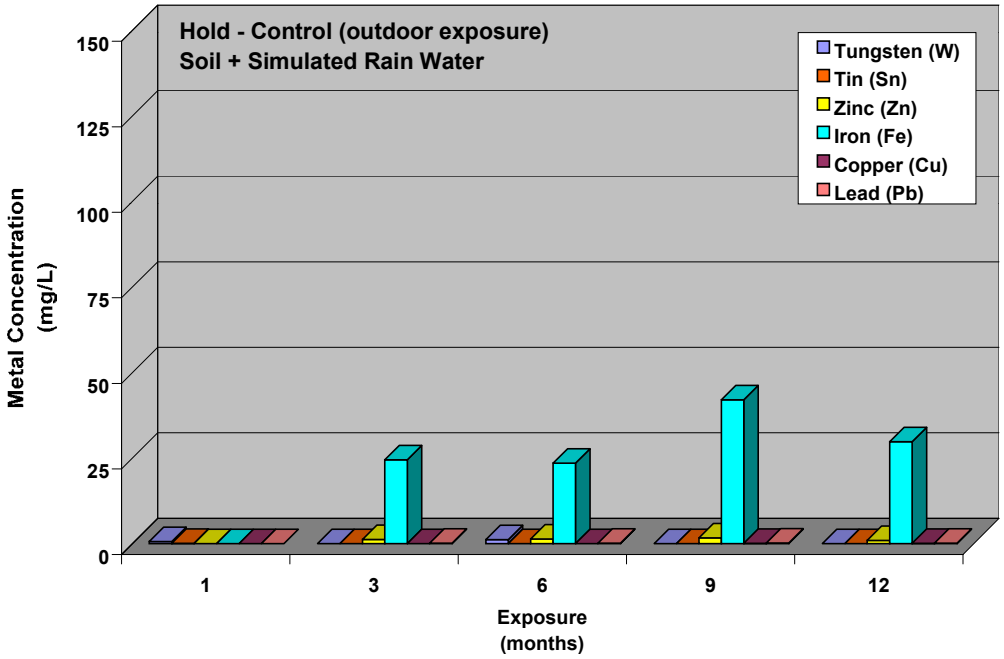

Figure O-1. Metal concentrations in leachate for control sample from the hold test using soil and rain water.

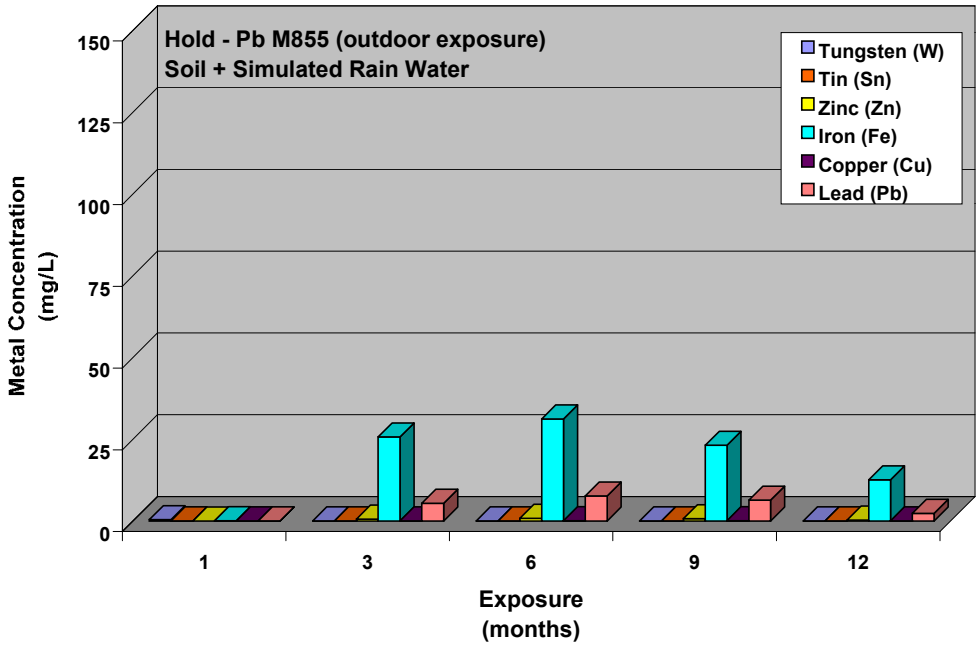

Figure O-2. Metal concentrations in leachate for lead M855 sample from the hold test using soil and rain water.

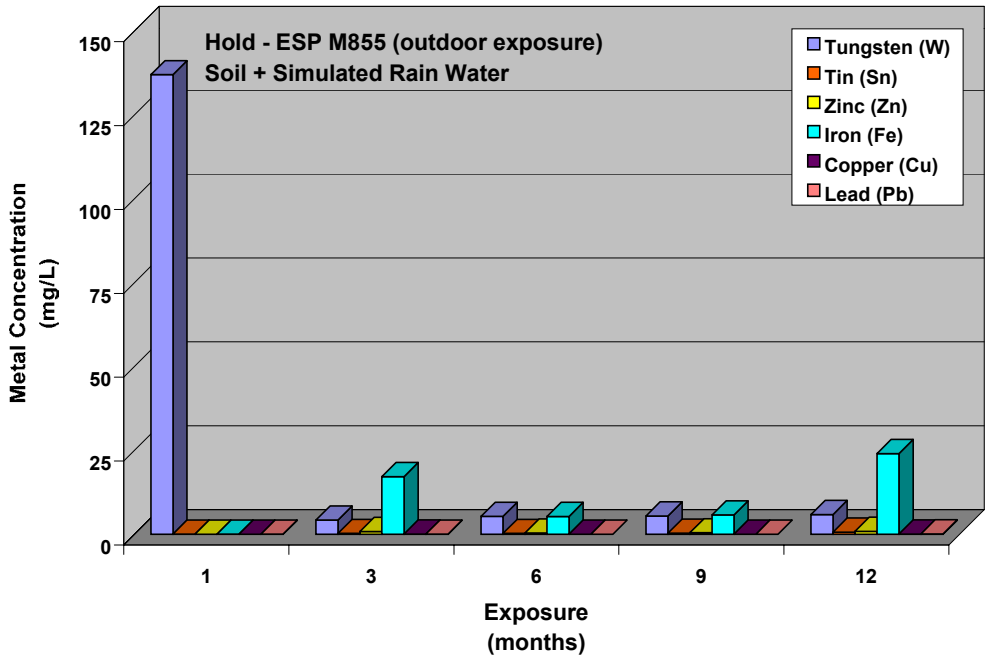

Figure O-3. Metal concentrations in leachate for ESP M855 sample from the hold test using soil and rain water.

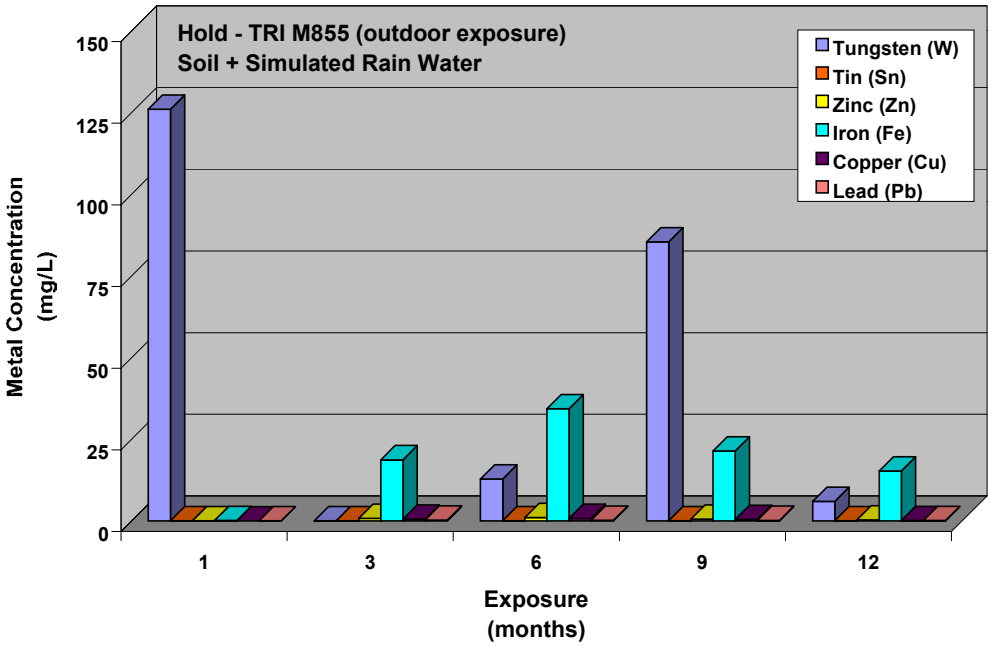

Figure O-4. Metal concentrations in leachate for TRI M855 sample from the hold test using soil and rain water