

The logo for Exponent, featuring the word "Exponent" in a white serif font with a trademark symbol. The letter 'x' is stylized with a cursive-like flourish. The logo is set against a dark teal background that has a vertical strip of white circles on the left side and a faint, large-scale pattern of overlapping shapes at the bottom.

Final Report

Phase III Investigation: *In Situ* Reduction of Lead Bioaccessibility in Soils, Trail, British Columbia

Prepared for

Trail Lead Program
Trail, British Columbia



Draft Final Report

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Acronyms and Abbreviations

| | |
|------|--|
| HDPE | high-density polyethylene |
| HFO | hydrous ferric oxide |
| ICP | inductively coupled plasma |
| MDNR | Missouri Department of Natural Resources |
| NIST | National Institute of Standards and Technology |
| PBET | physiologically based extraction procedure |
| RBA | relative bioavailability adjustment |
| SPLP | synthetic precipitation leaching procedure |
| SRMs | standard reference materials |
| TCP | tribasic calcium phosphate |
| XRF | x-ray fluorescence |

Introduction

This study was the third phase of an investigation to evaluate the feasibility of using chemical amendments to reduce the bioaccessibility of metals—particularly lead—in soils affected by aerial fallout from smelter stack emissions in Trail, British Columbia, Canada. The Phase III study involved laboratory testing to compare the optimal amendment identified during earlier phases of the investigation—hydrous ferric oxide and tribasic calcium phosphate (HFO/TCP)—to other amendments that had been presented in recent literature and were not included in the previous investigation phases. Results of the Phase III investigation demonstrated that several amendments were more effective at reducing the soil lead bioaccessibility than the HFO/TCP amendment, and served as the basis for selecting three amendments for inclusion in the field testing at the site initiated in July 1998. These amendments include two commercially available amendments—Solucorp's MBS Amendment and RMT's EnviroBlend Formula A—and a non-proprietary mixture of iron filings and tribasic calcium phosphate (TCP).

During the Phase III investigation, Exponent also characterized the lead bioaccessibility of five soil samples collected from Joplin, Missouri, in an effort to validate the use of the physiologically based extraction procedure (PBET) on chemically amended soils. In addition, the bioaccessibility of lead, arsenic, and cadmium in of four soil samples from the Trail area was determined in support of the risk assessment effort at the site.

Methods

The following summarizes the general procedures used during the Phase III study:

1. A composite soil sample, the "Phase III Soil," was prepared from Trail soil samples archived at the Exponent laboratory in Boulder, Colorado.
2. Splits of the Phase III Soil were chemically amended according to the matrix presented in Table 1.
3. The amended sample splits and a split of "baseline" Phase III Soil (soil that was not amended) were artificially weathered using the procedure described below (note: the sample splits receiving the Solucorp MBS and the EnviroBlend Formula A and Formula B amendments were not weathered).
4. The bioaccessibility and leachability of lead, arsenic, and cadmium in the amended and baseline soil splits were evaluated using the PBET and synthetic precipitation leaching procedure (SPLP), respectively.

Phase III Test Methods

The Phase III Soil composite was prepared by combining 3.8 kg of archived soil from the Phase II investigation (Exponent 1999) with 1.3 kg of previously archived soil samples collected from the East Trail, West Trail, and Tadanac neighborhoods. All of the soils used to make the Phase III Soil had been sieved previously to <2 mm. The composite was homogenized in an end-over-end tumbler for 1 hour, and then split into three 1-kg subsamples and one 2-kg subsample. Two of the 1-kg subsamples were shipped to Solucorp and RMT, where 100-g sample splits of the subsamples were treated with the MBS and EnviroBlend Formula A and Formula B amendments, respectively. These sample splits were not subjected to the weathering procedure discussed below, but rather to a proprietary amendment process by Solucorp and RMT, respectively. The remaining 1-kg subsample was shipped to a third company that ultimately did not participate in the investigation. Thirteen 100-g splits were collected from the 2-kg subsample, 12 of which were treated with chemical amendments. The thirteenth split was used to characterize the baseline (pre-amendment) conditions of the soil. All thirteen splits were subjected to the weathering procedure.

The weathering procedure was performed by placing each 100-g sample split into a high-density polyethylene (HDPE) container, along with the appropriate mass of chemical amendment (Table 1) and 175 mL of deionized water. Each soil-water slurry was agitated for a one-week period. The slurries were then placed in an oven and dried at

50°C. Each of the dried, weathered-soil samples, and the soil samples amended with the MBS, EnviroBlend Formula A, and EnviroBlend Formula B, were split into two subsamples. One subsample was sieved through a 2-mm sieve and the other through a 250- μm sieve. The <250- μm soil fraction—the fraction most likely to adhere to a child's hand—was subjected to the PBET to determine the bioaccessibility of lead, arsenic, and cadmium in the soils. Metals leachability was evaluated on the <2-mm soil fraction using the SPLP to determine whether the amendments increased or decreased the potential for lead, arsenic, and cadmium to be dissolved from the soil by infiltrating rainwater. Detailed descriptions of the PBET and SPLP followed during this study are included as Attachments 1 and 2, respectively. Two splits were also collected from the original Phase III Soil, split into two subsamples, and sieved into <250- μm and <2-mm soil fractions for PBET and SPLP testing. These samples were used to provide further data on the baseline (pre-amendment) conditions of the soil.

Additional Testing

Additional laboratory tests were conducted during the Phase III investigation to validate the estimates of lead bioaccessibility by the PBET on chemically amended soils. Five soil samples—three amended and two corresponding baseline (non-amended) samples—from a smelter site in Joplin, Missouri were included in the Phase III testing. The PBET-determined lead bioaccessibility of the five Joplin soil samples was compared to the results of lead bioavailability studies in swine on these soils, to evaluate chemical amendment effectiveness. The swine study did not evaluate arsenic and cadmium bioavailability.

The lead, arsenic, and cadmium bioaccessibility of four additional Trail (baseline) soil composites was also determined during the Phase III investigation in support of the risk assessment efforts at the site. A detailed description of the test results is presented in *Draft Technical Memorandum No. 2.1* (Exponent 1998a).

Analytical Procedures

The total lead, arsenic, and cadmium concentrations in the <2-mm and the <250- μm fractions of the amended and baseline soil samples were determined by microwave/acid digestion (USEPA Method 3051) and inductively coupled plasma (ICP) analysis (USEPA Methods 6010 and 7060) at ACZ Laboratories in Steamboat Springs Colorado. Confirmatory lead analysis of the <250- μm sample was performed at the University of Colorado using x-ray fluorescence (XRF) spectroscopy. The lead, arsenic, and cadmium concentrations in the PBET and SPLP test extracts were measured at the University of Colorado using inductively coupled plasma analysis.

Results

Metals Concentrations in Amendment Study Soils

The total lead, arsenic, and cadmium concentrations, as determined by ICP analysis, of the study soils are summarized in Table 2. The lead concentrations of the <250- μm fraction of the two non-weathered and one weathered baseline soil samples were 1,700, 1,550, and 1,200 mg/kg, respectively. Similar lead concentrations were observed in the <2-mm fractions of the baseline samples (1,660, 1,910, and 1,120 mg/kg, respectively). Arsenic and cadmium concentrations in the <250- μm fraction of the three baseline samples ranged from 43 to 68 mg/kg and 20.9 to 27.8 mg/kg, respectively, and from 40 to 64 mg/kg and 18.1 to 31.4 mg/kg, respectively, in the <2-mm fraction. Note that all three metals follow a consistent decreasing trend in the three baseline soil samples (Table 2). ICP-measured lead concentrations in the <250- μm and the <2-mm fractions of the amended soil samples ranged between 973 and 1,760 mg/kg and 811 and 1,680 mg/kg, respectively. Arsenic and cadmium concentrations in these samples ranged from 36 to 64 mg/kg and 14.8 to 30.0 mg/kg, respectively, in the <250- μm fraction, and from 33 to 64 mg/kg and 15.8 and 35.2 mg/kg, respectively, in the <2-mm fraction.

Considerable variability was observed in soil-lead concentrations among samples. The relative standard deviation (the ratio of the standard deviation to the mean) of the lead, arsenic, and cadmium concentrations in the <250- μm fraction of the three baseline samples was 17, 22, and 14 percent, respectively, and similar variability in metals concentrations was observed in the <2-mm fraction. Accounting for dilution due to amendment addition, the soil-lead concentrations across the entire set of soil samples ranged from 1,040 to 1,909 mg/kg in the <250- μm fraction, and from 912 to 1,910 mg/kg in the <2-mm fraction. Furthermore, measured metals concentrations do not appear to be correlated between the two size fractions of each sample (Figures 1 and 2). Although some differences are expected between the two fractions of the same sample, due to nugget effects and sample heterogeneity, it is anticipated that metals concentrations between the two fractions would be well correlated if the analytical measurements are accurate.

The variability in the measured metals concentrations observed across the soil-sample splits was likely caused by a combination of incomplete homogenization of the original Phase III Soil prior to collecting the individual sample splits and analytical error during the ICP determination of soil metals concentrations. A review of the quality control data from ACZ revealed that two of the five laboratory matrix spikes had unsatisfactory recoveries (one above and one below the $\pm 20\%$ range considered acceptable by the laboratory), which were attributed to matrix interferences. These results suggest that analytical error may have been a factor in the observed variability in soil metals content. To investigate the potential analytical error, and to confirm the validity of the ICP

analyses, additional splits of the <250- μm fraction of the amended and baseline samples were analyzed by XRF. In general, the XRF analysis produced higher concentrations than those determined by ICP (Table 2). This result was anticipated due to the inefficiency inherent to the solids digestion step of the ICP analysis. The XRF measurements show a slight correlation to the ICP measurements ($r^2=0.47$, $n=14$; Figure 3), suggesting that the variability in the ICP results is due in part to sample heterogeneity. Had the variability been solely due to random analytical error in the ICP and XRF analyses, there would have been little to no correlation between the two data sets.

As discussed in the methods section, the composite sample consisted of several discrete soil samples that were combined and rotated end-over-end for one hour to homogenize. It is possible that this homogenization process was insufficient to thoroughly mix the soils. The results from Exponent's quality control samples (see the Quality Control section below) provide evidence that the observed variability in metals concentrations was at least in part the result of heterogeneity within the original Phase III Soil. Metals concentrations were similar in all of the replicate samples, and Exponent's quality control sample spikes and standard reference materials (SRMs) produced acceptable results. The reproducibility of replicate samples indicates that the metals were distributed evenly within the individual soil samples following the amendment procedure—suggesting that additional mixing occurred within each individual sample during the weathering (where applicable) and sieving procedures.

The samples that were subjected to the weathering procedure generally had lower metals concentrations than those not subjected to the procedure. It is unlikely that the weathering procedure would have significantly affected the total metals content of the samples. It is possible, though unlikely, that the apparent lower metals concentrations in the weathered samples were the result of adsorption of fine-grained soil particles—those that are most likely to be metals-rich—to the weathering apparatus prior to the ICP analysis. Due to the fact that the sample splits used for the weathering procedure were collected from the Phase III Soil at a different time than those not subjected to the procedure, it is also possible that they were drawn from a less metals-rich portion of the composite. Both of these phenomena would contribute to the observed variability in soil metals content across the samples.

Metals Bioaccessibility

Metals bioaccessibility is calculated from the PBET results using the following equation:

$$\text{Bioaccessibility (\%)} = \frac{\text{total mass of metal in the PBET extract}}{\text{total mass of metal in the extracted soil sample}} = \frac{C_e \times V_e}{C_s \times M_s} \times 100$$

where:

C_e = Metal concentration in the PBET extract (mg/L)

V_e = PBET extract Liquid Volume (L)

C_s = Metal concentration of the soil sample added to the PBET apparatus (mg/kg)

M_s = Mass of soil used in the PBET (kg).

Table 3 presents the lead, arsenic, and cadmium concentrations in the PBET extracts after 30- and 60-minute extraction periods. The maximum concentration observed was used in calculating the soil-metal bioaccessibility (Table 4).

Bioaccessibility is directly correlated to the inverse of the soil-metal concentration, so calculation of bioaccessibility in the samples (and interpretation of the results) is complicated by the uncertainty in the measured metals concentrations. A review of the data did not provide a conclusive indication of the cause of the observed variability among the soil samples. As a result, bioaccessibility was calculated using three different approaches:

- Based on the ICP-determined soil metals concentration: this approach assumes that the ICP measurements for each sample were accurate and that heterogeneity between sample splits was the primary cause of the observed variability in metals concentrations.
- Based on the average of the ICP-determined soil metals concentrations: this approach assumes that the soil metals were evenly distributed across the sample splits and that the average of the ICP measurements (accounting for dilution) is an accurate representation of the metals content in the soil samples. Therefore, the averages of the lead, arsenic, and cadmium ICP measurements among the samples were used to calculate the bioaccessibility of the metals in all of the samples.
- Based on the XRF-determined soil metals concentration: this approach assumes that the XRF measurements are an accurate representation of the actual soil-metals content of the samples.

In each of the calculations, the average of replicate soil-metal concentrations was used where applicable. All three approaches yielded similar conclusions in terms of comparing amendment performance (Table 4). In other words, the same amendments were shown to be the most effective no matter how the bioaccessibility was calculated.

In the following discussion, references to bioaccessibility (or percent reduction in bioaccessibility) are based on the soil-metals content measured by ICP in each individual sample. The values that follow in parentheses refer to the calculated bioaccessibility based on the average ICP-measured soil-metals concentration across the sample set. In

the case of lead bioaccessibility, a second value in the parentheses is the bioaccessibility calculated based on the XRF-determined soil-metals concentrations.

The respective average lead, arsenic, and cadmium bioaccessibilities of the two baseline (non-weathered) soil samples were 79 (93; 70)¹, 55 (68)² and 69 (77)³ percent, and were 76 (66; 76)¹, 49 (41)², and 73 (64)³ percent in the weathered baseline sample. There was a large difference in the bioaccessibility of the Phase III baseline soil compared to that measured in similar soil samples during Phase II testing. Phase II PBET testing of baseline soil samples collected from the Tadanac neighborhood of Trail resulted in lead, arsenic, and cadmium bioaccessibility values of 58, 91, and 46 percent, respectively (see Table 1 in PTI 1998; Exponent 1999). The apparent difference in the baseline bioaccessibility may be due to inherent differences between the Phase II and Phase III soil samples. It is more likely, though, that the difference is due to the lower extraction pH used in the Phase III PBET tests. The Phase II bioaccessibility tests were performed at pH 2.0, whereas the current standard procedure for bioaccessibility testing calls for an extraction pH of 1.5. This lower, more aggressive pH is believed to more accurately reflect the conditions in a child's digestive tract under fasted conditions.

In general, all of the amendments reduced the lead bioaccessibility in the soils relative to the baseline bioaccessibility (Table 4). Six amendments proved to be more effective than the optimal amendment identified during the Phase II study (5 wt % HFO and 0.5 wt percent TCP). The HFO/TCP amendment—which had resulted in a 65 percent reduction in lead bioaccessibility during the Phase II PBET testing (amendments 1–3 in the Phase II batch tests; Exponent 1999b)—resulted in only a 16 (35, 31)¹ percent reduction in lead bioaccessibility during the Phase III testing. The reduced performance of the HFO/TCP amendment between the Phase II and Phase III study may be due to 1) differences in the mineralogy of the Phase II and Phase III soils, 2) the lower extraction pH of the Phase III PBET, or 3) differences in the methods used to apply HFO during the two studies. Since a mineralogical characterization of the Phase III Soil has not been conducted, it is not possible to assess the effects of the mineral differences on the resulting changes in lead bioaccessibility. At the lower pH value of the Phase III PBET, more of the HFO (and associated adsorbed lead) would likely dissolve than would dissolve at the pH of the Phase II test. However, if this were the primary reason for the apparent difference between the Phase II and III study results, then the other iron-bearing amendments evaluated during the Phase III tests—Iron Rich and the iron filings—likely would not have performed well during the tests [the Iron Rich and iron filings amendments lowered

¹ Values calculated based on the average of the ICP-determined soil-lead concentration in all of the samples (accounting for amendment dilution) and the XRF-determined soil-lead concentrations in the individual soil samples, respectively.

² Value calculated based on the average of the ICP-determined soil-arsenic concentration in all of the samples (accounting for amendment dilution).

³ Value calculated based on the average of the ICP-determined soil-cadmium concentration in all of the samples (accounting for amendment dilution).

lead bioaccessibility by 30 (39; 29)¹ and 31 (50; 62)¹ percent, respectively]. The HFO preparation methods used during the two investigations were identical, with one exception; the HFO used during the Phase II test was aged only one day prior to its application to soils, whereas the HFO was aged 4 days prior to use during the Phase III tests. As HFO ages, the number of adsorption sites available to attenuate lead is diminished. Consequently, once the HFO is prepared in the field, there would only be a short period during which it could be applied effectively. Given this undesirable attribute, and the superior performance of other amendments, the HFO/TCP amendment was not recommended for the field trials.

The three amendments that were most effective at lowering lead bioaccessibility were RMT's EnviroBlend Formula A (added at 21 wt %), Solucorp's MBS (added at 4.6 wt %), and the non-proprietary mixture of 5 wt % iron as iron filings and 0.5 wt % phosphorus as TCP (total application rate of 8 wt %). These amendments lowered the lead bioaccessibility of the soils by 84 (85; 87)¹, 69 (61; 75)¹, and 31 (50; 62)¹ percent, respectively (Table 4). However, two of these amendments, EnviroBlend Formula A and Solucorp's MBS, increased the arsenic bioaccessibility by 43 (24)² and 76 (109)³ percent, respectively. The iron filings amendment lowered the arsenic bioaccessibility by 7 (31)² percent. EnviroBlend Formula A, Solucorp MBS, and iron filings lowered the cadmium bioaccessibility by 13 (15)³, 34 (25)³, and 35 (36)³ percent, respectively.

Metals Leachability of Amendment Study Soils

The results of the SPLP testing of the baseline and amended soils are presented in Table 5 as the mass of metal (mg) released to solution per mass of soil (kg) added to the SPLP apparatus. The two non-weathered baseline samples released 2.68 and <2 mg/kg lead, and the weathered baseline sample released 2.26 mg/kg lead. The amount of leachable lead increased slightly, to 3.80 and 4.72 mg/kg, respectively, when TCP and zeolite were used as the soil amendments, but was little changed in the soil samples amended with steel shot (2.60 mg/kg). All of the other amendments lowered the lead leachability to below 2 mg/kg, including the three most effective amendments with respect to lead bioaccessibility (EnviroBlend Formula A, Solucorp's MBS, and iron filings).

Appreciable amounts of cadmium were not released from any of the soil samples tested (Table 5). The amount of arsenic released from soil during the SPLP tests increased from <3 mg/kg in the three baseline soil samples, to 3.78 and 21.2 mg/kg in the samples amended with zeolite and iron filings, respectively. The arsenic released from the soil amended with iron filings was approximately a third of the total arsenic available in the soil (total arsenic concentration in the <2-mm soil fraction of the sample was 62 mg/kg; Table 2). This arsenic release likely occurred because the SPLP test is performed in a closed container, thus depriving the SPLP solution of atmospheric oxygen. In theory, the iron filings soil amendment (which consists predominantly of zero-valent iron) would slowly produce HFO due to oxidation by atmospheric oxygen present in the soil column. This HFO, in turn would, sequester soil arsenic, which is strongly adsorbed by HFO. However, in the closed SPLP system, the available oxygen would be consumed rapidly

and the production of HFO would be terminated. Furthermore, zero-valent iron is a strong reductant and would have likely created a reduced environment in the bottle. Under these reduced conditions, the HFO present in the system (both naturally occurring and that formed due to the initial oxidation of the iron fillings) would be reduced to soluble ferrous iron. The net result is that any arsenic that was adsorbed to the HFO would be released to the SPLP solution. Because oxygen would likely be abundant in the shallow site soils, such reducing conditions are unlikely to occur under field conditions, and the results of the SPLP tests may not be representative of the behavior of the iron filings amendment during a full-scale application.

Bioaccessibility Testing of Joplin Study Soils

Recent research conducted at the Missouri Department of Natural Resources (MDNR) comparing PBET results for lead to the relative bioavailability adjustment (RBA) for lead obtained using the U.S. EPA Region VIII swine model, suggests that the PBET accurately predicted the RBA of natural soils, but under-predicted the RBA of soils that had been chemically amended to reduce lead bioavailability. Because the PBET is the tool that has been used to assess the effectiveness of chemical amendments at reducing lead bioaccessibility in Trail soils, confirmation of MDNR's results would affect the way that the PBET results for amended soils should be interpreted. To address this, five soil samples (two baseline and three amended) derived from previous bench-scale and field-scale studies in Joplin, Missouri were evaluated using the PBET. The RBA for lead of each of these soils has been determined previously using the swine model.

Interpretation of the PBET results is confounded somewhat by the variability in the measured lead concentration in the field-trial soil samples (Table 6). Two soil substrates were included in these tests: bench-trial soil and field-trial soil. The measured lead concentration in the baseline field-trial soil was 4,820 mg/kg, but it was 2,890 mg/kg in the split of this sample that had been amended with 0.5 wt % phosphorus as phosphoric acid. These data suggest either that the lead in this sample is distributed unevenly within the sample, or that the laboratory analyses were in error. The quality control sample results were acceptable (discussed below), indicating that analytical error may not account for the variability observed in lead concentrations. However, data are insufficient for conclusive determination of the source of the observed variability. The metals concentrations in the four samples of the Joplin bench-trial soil were all very similar, suggesting that the results for these soils are more certain.

To facilitate the interpretation of the bioaccessibility data in light of the uncertainty in soil-lead concentrations, the bioaccessibility values for the Joplin soils were calculated using both the measured lead concentration in each sample and the average lead concentration in each of the two soil sample substrates (i.e., bench-trial soil and field-trial soil; Table 6). The PBET results for the bench-trial and field-trial soils amended with 1 wt % phosphoric acid (added as phosphoric acid) indicate a reduction in lead bioaccessibility of 26–38 percent or 29–45 percent, calculated using the measured and average soil-lead concentrations, respectively. These results agree well with the animal-model study, which suggested that the same amendment reduced the lead RBA of the soil by 31–36 percent (Table 7). Similarly, the calculated lead bioaccessibility reduction of 38 percent based on the average soil-lead concentration in the field-trial soil sample amended with 0.5 wt % phosphorus amendment (as phosphoric acid) compares reasonably-well with the animal study which indicated a reduction of 24 percent. However, the measured soil-lead concentration of this sample appears anomalously low relative to the other measurements. When this concentration is used to calculate the lead bioaccessibility, it indicates that the 0.5 wt % phosphorus amendment slightly increased

the lead bioaccessibility—despite the fact that there was less lead in the PBET extract of the sample relative to that of the non-amended field trial soil sample. The predicted increase in bioaccessibility is not consistent with the rest of the data, suggesting that the measured lead concentration of the sample is unrepresentative of the lead concentration in the field-trial soil.

There are three broad sources of uncertainty in the above evaluation of the Joplin data: 1) uncertainty in the estimate of RBA from the animal model, 2) variability in lead concentration in the soil substrate, and 3) analytical noise associated with the PBET procedure. Insufficient data are currently available to fully assess the contribution of the first two sources of uncertainty. Repeated PBET analysis of National Institute of Standards and Technology (NIST) SRM 2710 indicates that the lead bioaccessibility of this sample has historically ranged between 50 and 70 percent, with an average value of 63 percent (standard deviation of 7.9 percent, n=6). These data suggest that uncertainty associated with the PBET results is relatively small. Therefore, within the uncertainty associated with the actual lead concentration in the Joplin soil substrates, and the uncertainty associated with the RBA estimates, it appears that the PBET procedure provides a reasonably accurate estimate of *in vivo* values for the reduction in lead bioavailability achievable using *in situ* amendments.

Quality Control

Soil Quality Control Samples

The quality control samples submitted by Exponent to ACZ for soil-metals analysis by ICP included four sets of replicate soil samples and two sets of triplicate soil samples, two Joplin soil replicate samples, and the NIST SRM 2710, which was submitted for two analyses (Tables 8 and 9). All of these samples were submitted as blind samples. All replicate and triplicate results were within control (i.e., had a relative standard deviation or relative percent difference of less than 30 percent—the acceptable level of variability for metals analysis of solids). The NIST SRM 2710 was submitted as received from NIST. The measured concentrations of lead and arsenic in SRM 2710 were within 3 percent of the certified values (Table 9). The measured cadmium concentrations in SRM 2710 (23.5 and 23.0 mg/kg) were slightly higher than the certified concentration (20.0 mg/kg), but within 18 percent of the certified value. These results indicate that the soil concentration data were within control and of acceptable quality.

PBET Extract Quality Control

The quality control samples submitted during the analysis of the PBET extracts included four replicate samples, one triplicate sample, and a matrix spike (Table 10 and 11). In addition, the PBET was performed on the NIST SRM 2710 (Table 12). The relative standard deviation of the maximum lead, arsenic, and cadmium concentrations extracted during the triplicate analysis of the HFO-amended soil was 18, 29, and 11 percent, respectively. With the exception of the arsenic extracted from Joplin bench-trial soil amended with 1 percent phosphorus, which had a relative percent difference between replicates of 46 percent, the relative percent differences between replicates were below 23 percent. While no formal guidelines have been established for acceptable levels of variability in PBET results, it is our opinion that these data indicate that an acceptable level of variability was achieved during these tests.

To determine whether lead, arsenic, or cadmium was adsorbing to the PBET apparatus, the PBET procedure was performed using extraction solution that had been spiked with these elements at the concentrations shown in Table 11 (i.e., in the absence of soil in the apparatus). Spike recoveries ranged between 87 and 101 percent, indicating that no significant adsorption of these elements was occurring.

Prior to the current investigation, NIST SRM 2710 had been included in five PBET studies (Table 12), resulting in five previous measurements lead and arsenic bioaccessibility and three previous measurements of cadmium bioaccessibility. Comparisons among the values obtained during each study are based on the maximum

lead, arsenic, and cadmium concentrations observed during individual tests, because these are the values used to calculate the bioaccessibility. The average maximum lead, arsenic, and cadmium concentrations observed during previous studies were 31.4, 2.44, and 0.205 mg/L, respectively. The corresponding maximum values obtained during the Phase III investigation were 35.3, 2.99, and 0.166 mg/L, or within 12, 23, and 19 percent of the previous average maximum values, respectively.

SPLP Extract Quality Control Samples

The quality control samples included in the SPLP study included triplicate analysis of one sample (the HFO-amended sample) and replicate analysis of two samples (the non-weathered baseline Phase III Soil and the Portland cement-amended soil; Table 5). In addition, the SPLP quality control samples included an apparatus blank, an aqueous and a soil standard, and a matrix spike (Table 13). Neither arsenic nor cadmium was detected in the SPLP replicate and triplicate extracts, and lead was detected at only 0.143 mg/L (2.68 mg/kg) in one of the two baseline soil samples (Table 5). Given the low frequency of detection in the replicate samples, it is not possible to assess the precision of the SPLP test results.

A sample of the SPLP solution was submitted to the analytical laboratory, where it was analyzed twice as a blank sample (Table 13). Cadmium was detected during one of these two analyses at 0.24 mg/L. Based on the soil-to-water ratio used during the SPLP procedure (0.2 kg:0.4 L), this would correspond to a cadmium concentration in soil (had it been present during this test) of 0.48 mg/kg. Given this result, the one SPLP analysis where cadmium was detected—0.3 mg/L in the baseline Phase III soil—has been qualified with a “J” qualifier (Table 5).

The analytical laboratory used the blank SPLP solution to prepare a laboratory spike sample containing 5 mg/L each of lead, arsenic, and cadmium. Results from these samples ranged between 5.08 and 5.21 mg/L (Table 13), or within 104 percent of the known concentrations. These data indicate that the ICP analyses of the SPLP extracts were accurate.

To determine whether lead, arsenic, or cadmium was adsorbing to the SPLP apparatus, the SPLP procedure was performed using extraction solution that had been spiked with these elements at 5, 0.2, and 0.2 mg/L, respectively. Unlike the PBET spike results described earlier, the SPLP spike recovery efficiency presented in Table 13 was based on the calculated spike concentration, rather than the measured metal concentration. Lead and cadmium recoveries were acceptable (105 and 112 percent, respectively), but the arsenic spike recovery (174 percent) was unacceptably high. However, without data describing the true original arsenic concentration in the extract, it is not possible to distinguish between analytical error, SPLP procedural error, and error occurring during preparation of the spike.

SPLP extracts of the NIST SRM 2710 sample contained no detectable lead, arsenic, or cadmium. Because this is the first study in which Exponent has included this sample in the SPLP test matrix, there are no previously collected data with which to compare these results.

Conclusions

The results of the Phase III investigation demonstrated that several chemical amendments were more effective at reducing the soil-lead bioaccessibility than the optimal amendment from the Phase II investigation (HFO/TCP). Three of these amendments—RMT's EnviroBlend Formula A, Solucorp's MBS, and iron filings with phosphate (as TCP)—were selected for inclusion in the field pilot-scale testing that was initiated in July of 1998. Considerable uncertainty was observed in the measured metals concentrations of the individual laboratory samples—somewhat obfuscating the Phase III results. However, comparison of amendment efficiency based on both the soil-metals measurements in each individual sample and based on the average soil metals concentrations across the entire sample set, yielded the same results (i.e., in each case, RMT's EnviroBlend Formula A, Solucorp's MBS, and iron filings with phosphate were the top performers).

Sample heterogeneity is thought to have been a primary cause of the observed variability in the measured metals concentrations across the samples. As a result, considerable care was taken to minimize sample heterogeneity during the subsequent, ongoing field investigation (a detailed description of the field-trial procedures is provided in the Work Plan for Field Testing of In Situ Soil Amendments; Exponent 1998b). Phase III SPLP testing indicated that the iron filings may increase the arsenic leachability of the soil. However, this result is thought to be an artifact of the anoxic conditions created during the test. As a result, the SPLP procedure has been modified for the Field Tests such that it will be conducted under oxygenated conditions, to confirm that arsenic is unlikely to leach from soils amended with iron filings under field conditions. Both the EnviroBlend Formula A and the MBS amendments were found to increase arsenic bioaccessibility. As a result, RMT (the provider of the EnviroBlend Formula A amendment) and Solucorp (the provider of the MBS amendment) conducted additional tests to refine their amendment's effectiveness with respect to arsenic bioaccessibility. In addition, RMT refined their amendment to achieve a lower, more feasible total application rate (the Phase III formulation was added at 21 wt %).

Although the results of the Phase III validation testing indicate that the PBET does not underpredict soil-lead bioaccessibility, the validation effort was not conclusive because of the uncertainty associated with the soil-lead measurements. When calculated based on the average lead concentration across the sample set, reductions in bioaccessibility resulting from the amendments agreed well with those estimated based on the animal test results.

References

Exponent 1999. Phase II Investigation Report: *In situ* reduction of lead bioaccessibility in soils, Trail, British Columbia. Prepared for Trail Lead Program. Trail, British Columbia.

Exponent 1998a. Draft Technical Memorandum 2.1. Prepared for Trail Lead Program. Trail, British Columbia.

Exponent 1998b. Work plan for field testing of *in situ* soil amendment, Trail, British Columbia. Prepared for Trail Lead Program, Trail, BC. Exponent, Boulder, CO.

Appendix A

Figures

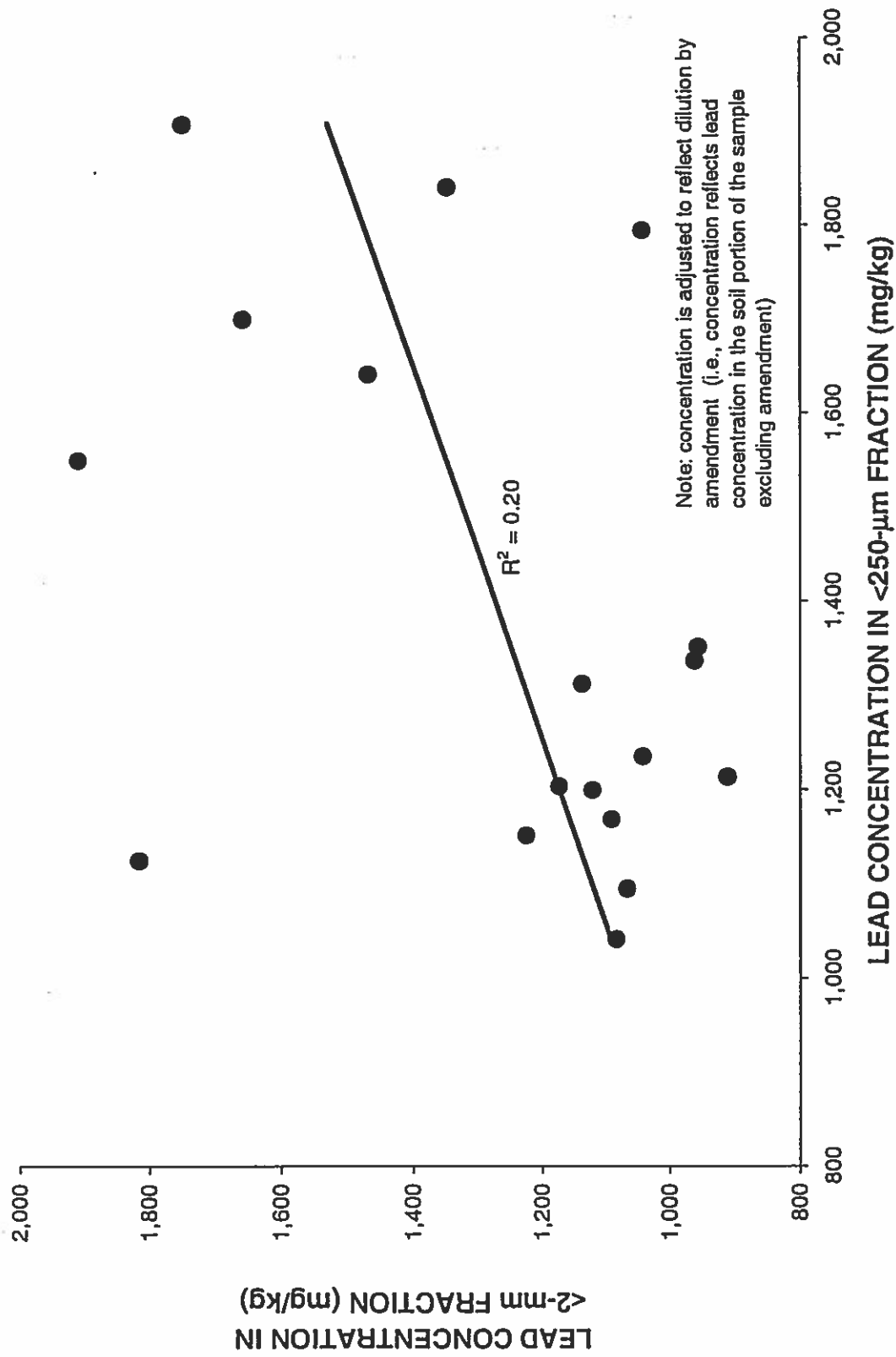


Figure 1. Comparison between lead concentrations measured in the <250- μ m and <2-mm soil fractions using ICP analysis.

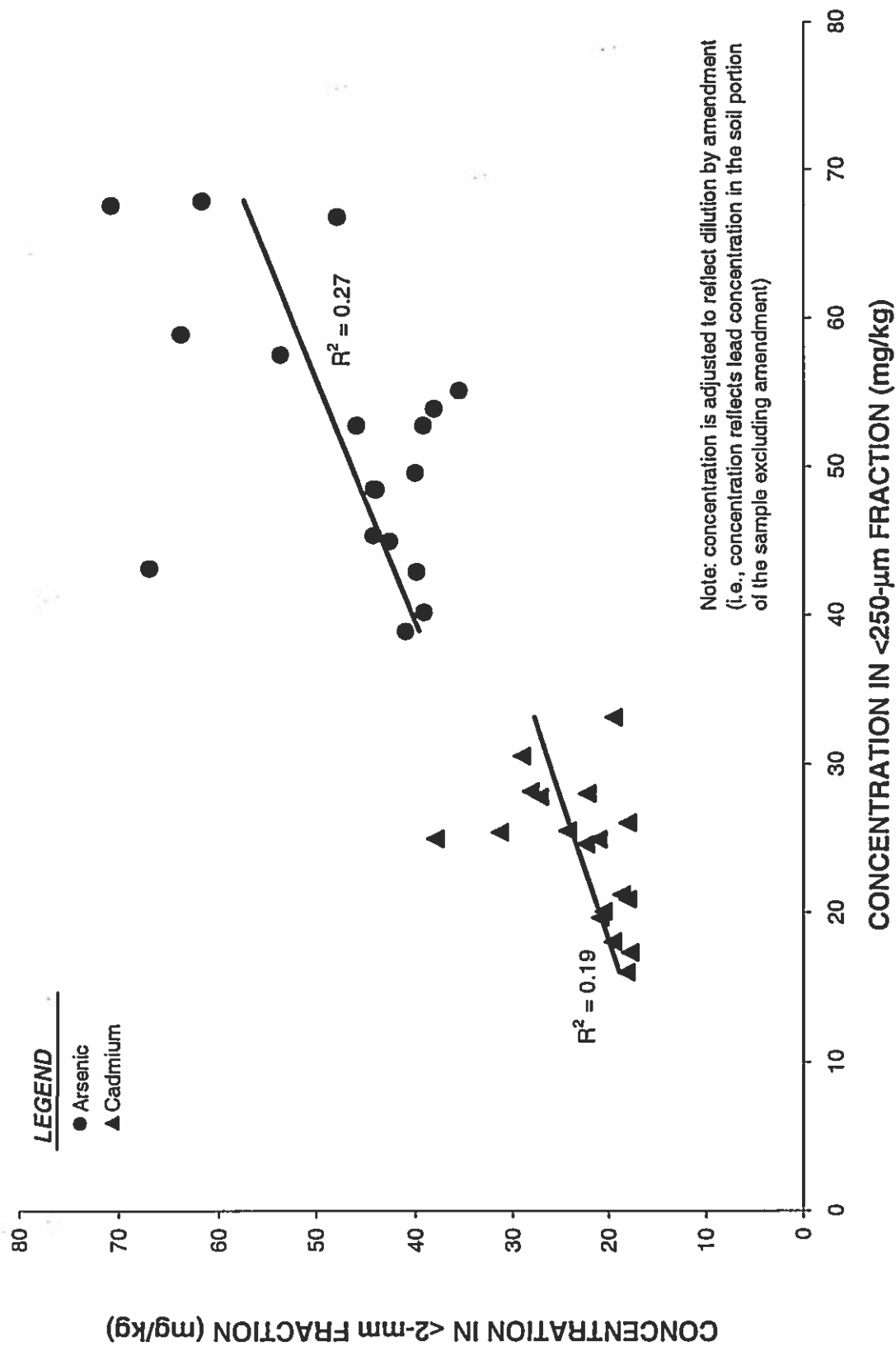


Figure 2. Comparison between arsenic and cadmium concentrations measured in the <250-μm fraction soils using XRF and ICP analysis.

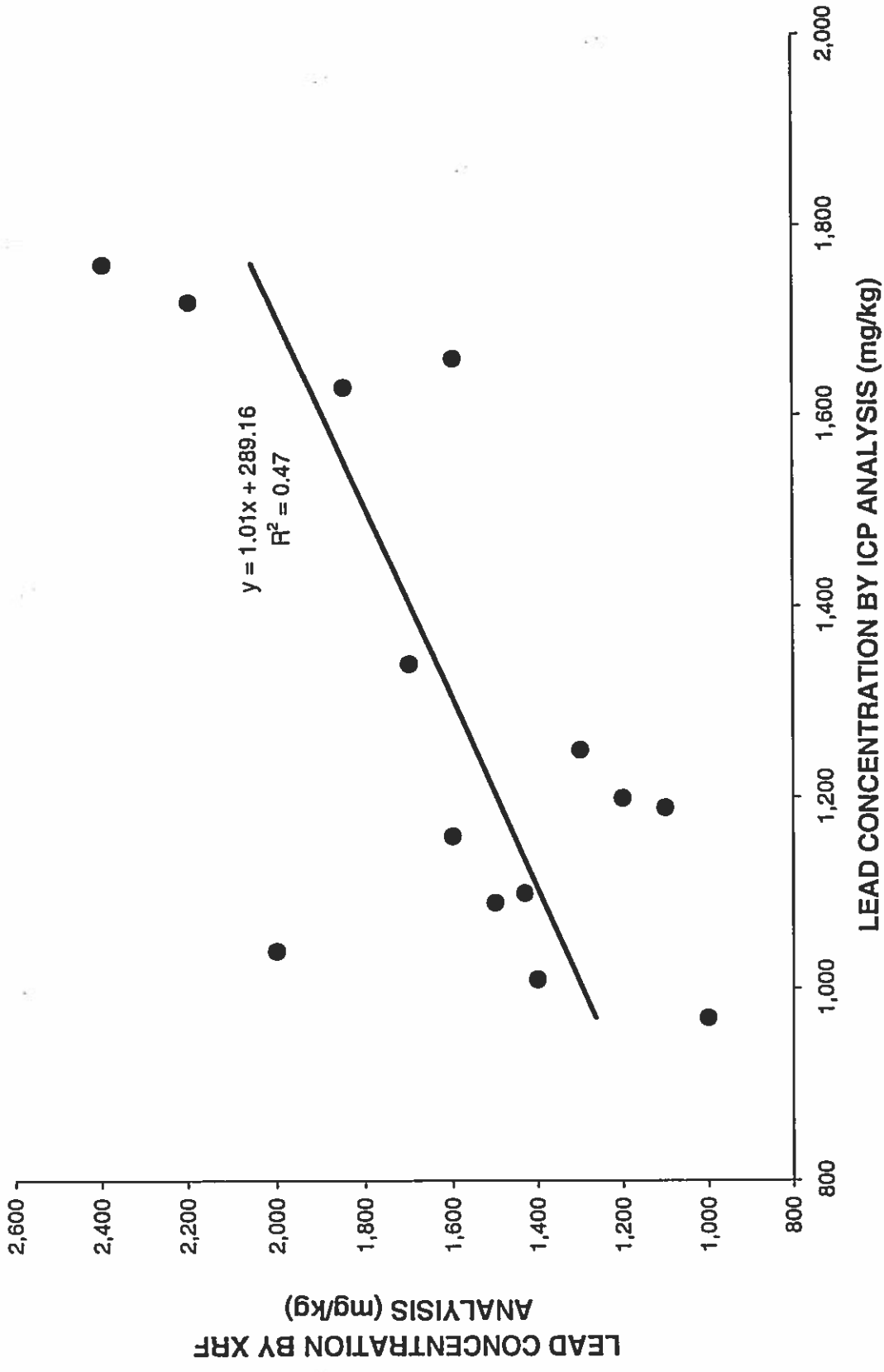


Figure 3. Comparison between lead concentrations measured in the <250- μ m fraction soils using XRF and ICP analysis.

Appendix B

Tables

Table 1. Amendment formulation and test matrix

| | Amendment Application Rate | Included in Weathering Procedure? | Mass of Amendment or Soil Added to Each Test (g unless otherwise noted) | | | | | | | | | | Included in SPLP Testing? |
|-------------------------------------|---------------------------------|-----------------------------------|---|-------------------|----------------------|--------------|-----------|------------------|-----------------|------------|---------|-----|---------------------------|
| | | | Soil | Activated Alumina | Hydrous ferric oxide | Iron filings | Iron rich | Phosphate as TCP | Portland cement | Steel shot | Zeolite | TCP | |
| Amendment Test Samples | | | | | | | | | | | | | |
| Baseline Soils | | | | | | | | | | | | | |
| Phase III Soil | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Phase III Soil (replicate) | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Weathered Phase III Soil | -- | Yes | 100 | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Amended Soils | | | | | | | | | | | | | |
| Activated alumina | 5% (as Al) ^a | Yes | 100 | 9.4 | -- | -- | -- | -- | -- | -- | 2.7 | 0.1 | Yes |
| EnviroBlend Formula A | 22.5% (total wt %) ^b | No | 100 | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| EnviroBlend Formula B | 11% (total wt %) ^b | No | 100 | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Hydrous ferric oxide | 5% (as Fe) ^a | Yes | 100 | -- | 15.1 | -- | -- | -- | -- | -- | 2.7 | 0.1 | Yes |
| Hydrous ferric oxide (replicate) | 5% (as Fe) ^a | Yes | 100 | -- | 15.1 | -- | -- | -- | -- | -- | 2.7 | 0.1 | Yes |
| Hydrous ferric oxide (triplicate) | 5% (as Fe) ^a | Yes | 100 | -- | 15.1 | -- | -- | -- | -- | -- | 2.7 | 0.1 | Yes |
| Iron filings | 5% (as Fe) ^a | Yes | 100 | -- | -- | 5 | -- | -- | -- | -- | 2.7 | 0.1 | Yes |
| Iron rich | 5% (as Fe) ^a | Yes | 100 | -- | -- | -- | 5 | -- | -- | -- | 2.7 | 0.1 | Yes |
| Manganese oxide | 5% (as Mn) ^{a,c} | Yes | 100 | -- | -- | -- | -- | 7.2 | -- | -- | 2.7 | 0.1 | Yes |
| Phosphate as TCP | 0.5% (as P) | Yes | 100 | -- | -- | -- | -- | -- | -- | -- | 2.7 | 0.1 | Yes |
| Portland cement | 3% (total wt %) ^a | Yes | 100 | -- | -- | -- | -- | -- | 2.5 | -- | 2.7 | 0.1 | Yes |
| Solucorp MBS | 4.6% (total wt %) ^b | No | 100 | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Steel shot | 5% (as Fe) ^a | Yes | 100 | -- | -- | -- | -- | -- | -- | 5 | -- | 2.7 | 0.1 |
| Steel shot (replicate) | 5% (as Fe) ^a | Yes | 100 | -- | -- | -- | -- | -- | -- | 5 | -- | 2.7 | 0.1 |
| Zeolite | 2.5% (total wt %) ^a | Yes | 100 | -- | -- | -- | -- | -- | -- | -- | 2.5 | 2.7 | 0.1 |
| Quality Control Samples | | | | | | | | | | | | | |
| NIST 2710 Montana soil | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Spiked stomach solution | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | Yes |
| Risk Assessment Soils | | | | | | | | | | | | | |
| Tadnac Soil | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| East Trail | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| West Trail | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Property boundary soil | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Joplin Soils | | | | | | | | | | | | | |
| Unamended Soils | | | | | | | | | | | | | |
| Joplin bench-trial soil | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Joplin field-trial soil | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Joplin field-trial soil (replicate) | -- | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Amended Soils | | | | | | | | | | | | | |
| Joplin bench-trial soil | 1% (as P) ^d | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Joplin field-trial soil | 1% (as P) ^d | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Joplin field-trial soil (replicate) | 1% (as P) ^d | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |
| Joplin field-trial soil | 0.5% (as P) ^d | No | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | No |

^a In addition to the primary amendment, phosphate will be added to the soil using 0.5 wt% P as TCP
^b The amendment rates were provided by the independent contractor.
^c Manganese was added as Mn₂O₃.
^d An 85-percent stock solution of H₃PO₄ will be used to prepare the phosphoric acid-bearing amendments.

Table 2. Total lead, arsenic, and cadmium concentration in the Phase III *in situ* soil amendment investigation soils (All units mg/kg)

| | Replicate Number | <250 mm fraction | | | | <2 mm fraction | | |
|---|------------------|------------------|-------|---------|---------|----------------|---------|---------|
| | | Lead | | Arsenic | Cadmium | Lead | Arsenic | Cadmium |
| | | ICP | XRF | | | | | |
| Amendment Test Samples | | | | | | | | |
| Baseline Soil | | | | | | | | |
| Non-Weathered Phase III Soil | 1 | 1,700 | 1,850 | 68 | 27.8 | 1,660 | 62 | 27.2 |
| | 2 | 1,550 | | 59 | 25.4 | 1,910 | 64 | 31.4 |
| Weathered Phase III Soil | -- | 1,200 | 1,200 | 43 | 20.9 | 1,120 | 40 | 18.1 |
| Amended Soil | | | | | | | | |
| Activated alumina | -- | 973 | 1,000 | 40 | 15.4 | 947 | 38 | 15.8 |
| EnviroBlend Formula A | -- | 1,340 | 1,700 | 47 | 23.0 | 1,200 | 44 | 23.0 |
| EnviroBlend Formula B | -- | 1,720 | 2,200 | 61 | 27.5 | 1,580 | 64 | 26.2 |
| Hydrous ferric oxide | 1 | 1,080 | 1,430 | 48 | 22.2 | 811 | 34 | 18.8 |
| | 2 | 1,040 | | 47 | 22.7 | 970 | 41 | 21.6 |
| | 3 | 1,190 | | 47 | 21.9 | 856 | 35 | 19.9 |
| Iron filings | -- | 1,040 | 2,000 | 40 | 23.1 | 1,680 | 62 | 35.2 |
| Iron rich | -- | 1,250 | 1,300 | 51 | 24.1 | 885 | 33 | 16.7 |
| Manganese oxide | -- | 1,190 | 1,100 | 44 | 30.0 | 1,030 | 40 | 17.7 |
| Phosphate as TCP | -- | 1,010 | 1,400 | 39 | 17.5 | 1,050 | 38 | 19.0 |
| Portland cement | 1 | 1,170 | 1,600 | 47 | 20.1 | 986 | 38 | 17.7 |
| | 2 | 1,140 | | 46 | 18.6 | 1,110 | 42 | 19.8 |
| Solucorp MBS | -- | 1,760 | 2,400 | 64 | 26.8 | 1,290 | 46 | 21.3 |
| Steel shot | -- | 1,660 | 1,600 | 36 | 14.8 | 965 | 38 | 16.8 |
| Zeolite | -- | 1,090 | 1,500 | 43 | 19.0 | 1,160 | 42 | 19.5 |
| Quality Control Sample | | | | | | | | |
| NIST 2710 Montana soil | 1 | 4,970 | 590 | 23 | -- | -- | -- | -- |
| | 2 | 4,940 | 610 | 23.5 | -- | -- | -- | -- |
| Joplin Soils | | | | | | | | |
| Baseline Soil | | | | | | | | |
| Bench-trial soil | 1 | 3,990 | -- | 10.9 | 28.7 | -- | -- | -- |
| | 2 | 4,080 | -- | 10.7 | 28.0 | -- | -- | -- |
| Field-trial soil | -- | 4,820 | -- | 12.0 | 29.0 | -- | -- | -- |
| Amended Soil | | | | | | | | |
| Bench-trial soil + 1% P as H ₃ PO ₄ | 1 | 3,780 | -- | 9.0 | 26.5 | -- | -- | -- |
| | 2 | 3,940 | -- | 8.9 | 25.6 | -- | -- | -- |
| Field-trial soil + 0.5% P as H ₃ PO ₄ | -- | 2,890 | -- | 8.0 | 18.4 | -- | -- | -- |
| Field-trial soil + 1% P as H ₃ PO ₄ | -- | 4,280 | -- | 7.0 | 24.8 | -- | -- | -- |
| Risk Assessment Soils | | | | | | | | |
| East Trail | -- | 4,940 | -- | 200 | 81.7 | -- | -- | -- |
| Property boundary soil | -- | 12,600 | -- | 460 | 82.7 | -- | -- | -- |
| Tadinac Soil | -- | 4,250 | -- | 149 | 62.7 | -- | -- | -- |
| West Trail | -- | 3,850 | -- | 119 | 66.7 | -- | -- | -- |

Table 3. Lead, arsenic, and cadmium concentrations in the PBET extracts

| | Replicate Number | Extraction Time (min) | pH | Pb (mg/L) | As (mg/L) | Cd (mg/L) |
|-------------------------------|------------------|-----------------------|------|-----------|-----------|-----------|
| Amendment Test Samples | | | | | | |
| Baseline Soil | | | | | | |
| Phase III Soil | 1 | 30 | 1.47 | 12.9 | 0.31 | 0.19 |
| | 1 | 60 | 1.47 | 12.8 | 0.39 | 0.19 |
| Phase III Soil | 2 | 30 | 1.53 | 13.0 | 0.29 | 0.17 |
| | 2 | 60 | 1.53 | 13.1 | 0.31 | 0.18 |
| Weathered Phase III Soil | -- | 30 | 1.58 | 8.5 | 0.20 | 0.15 |
| | -- | 60 | 1.56 | 9.2 | 0.21 | 0.15 |
| Amended Soil | | | | | | |
| Activated alumina | -- | 30 | 1.66 | 6.8 | 0.22 | 0.10 |
| | -- | 60 | 1.55 | 7.2 | 0.29 | 0.10 |
| EnviroBlend, Formula A | -- | 30 | 1.64 | 1.3 | 0.33 | 0.15 |
| | -- | 60 | 1.63 | 1.7 | 0.37 | 0.15 |
| EnviroBlend, Formula B | -- | 30 | 1.48 | 11.4 | 0.30 | 0.16 |
| | -- | 60 | 1.45 | 12.3 | 0.44 | 0.18 |
| Hydrous ferric oxide | 1 | 30 | 1.59 | 6.2 | 0.15 | 0.15 |
| | 1 | 60 | 1.62 | 8.2 | 0.20 | 0.16 |
| Hydrous ferric oxide | 2 | 30 | 1.59 | 7.4 | 0.14 | 0.13 |
| | 2 | 60 | 1.54 | 7.9 | 0.19 | 0.14 |
| Hydrous ferric oxide | 3 | 30 | 1.61 | 5.7 | 0.10 | 0.11 |
| | 3 | 60 | 1.61 | 5.8 | 0.11 | 0.12 |
| Iron filings | -- | 30 | 1.41 | 5.1 | 0.17 | 0.09 |
| | -- | 60 | 1.37 | 5.6 | 0.19 | 0.11 |
| Iron rich | -- | 30 | 1.61 | 6.4 | 0.15 | 0.11 |
| | -- | 60 | 1.63 | 6.8 | 0.25 | 0.12 |
| Manganese oxide | -- | 30 | 1.51 | 7.3 | 0.19 | 0.10 |
| | -- | 60 | 1.53 | 7.8 | 0.31 | 0.12 |
| Phosphate as TCP | -- | 30 | 1.52 | 5.8 | 0.16 | 0.10 |
| | -- | 60 | 1.51 | 6.1 | 0.30 | 0.10 |
| Portland cement | 1 | 30 | 1.32 | 9.8 | 0.39 | 0.13 |
| | 1 | 60 | 1.31 | 9.9 | 0.61 | 0.14 |
| Portland cement | 2 | 30 | 1.55 | 10.5 | 0.42 | 0.14 |
| | 2 | 60 | 1.55 | 11.2 | 0.52 | 0.16 |
| Solucorp MBS | -- | 30 | 1.63 | 4.3 | 0.57 | 0.10 |
| | -- | 60 | 1.59 | 4.5 | 0.61 | 0.13 |
| Steel shot | -- | 30 | 1.57 | 9.0 | 0.22 | 0.09 |
| | -- | 60 | 1.52 | 11.1 | 0.28 | 0.10 |
| Zeolite | -- | 30 | 1.64 | 8.1 | 0.31 | 0.12 |
| | -- | 60 | 1.64 | 7.6 | 0.38 | 0.12 |

Table 3. (cont.)

| Study/Sample Type/Sample Name | Replicate Number | Extraction Time (min) | pH | Pb (mg/L) | As (mg/L) | Cd (mg/L) |
|---|------------------|-----------------------|------|-----------|-----------|-----------|
| Risk Assessment Soils | | | | | | |
| East Trail | -- | 30 | 1.56 | 31.3 | 0.86 | 0.52 |
| | -- | 60 | 1.56 | 31.7 | 1.11 | 0.55 |
| Property boundary soil | -- | 30 | 1.58 | 71.4 | 1.92 | 0.31 |
| | -- | 60 | 1.58 | 74.3 | 2.35 | 0.34 |
| Tadinac Soil | -- | 30 | 1.61 | 26.2 | 0.54 | 0.34 |
| | -- | 60 | 1.61 | 27.4 | 0.66 | 0.36 |
| West Trail | -- | 30 | 1.65 | 21.5 | 5.38 | 0.37 |
| | -- | 60 | 1.58 | 21.8 | 0.59 | 0.37 |
| Quality Control Samples | | | | | | |
| NIST 2710 Montana soil | -- | 30 | 1.60 | 35.3 | 2.71 | 0.16 |
| | -- | 60 | 1.59 | 33.9 | 2.99 | 0.17 |
| Spiked stomach solution | -- | 30 | 1.53 | 5.2 | 0.53 | 0.48 |
| | -- | 60 | 1.49 | 4.7 | 0.56 | 0.46 |
| | -- ^a | -- | -- | 5.4 | 0.55 | 0.52 |
| Joplin Soils | | | | | | |
| Bench-trial soil | 1 | 30 | 1.62 | 21.9 | 0.07 | 0.14 |
| | 1 | 60 | 1.60 | 22.1 | 0.15 | 0.14 |
| Bench-trial soil | 2 | 30 | 1.59 | 22.9 | 0.11 | 0.15 |
| | 2 | 60 | 1.57 | 23.5 | 0.15 | 0.15 |
| Bench-trial soil + 1% P as H ₃ PO ₄ | 1 | 30 | 1.65 | 14.8 | 0.11 | 0.11 |
| | 1 | 60 | 1.64 | 16.0 | 0.12 | 0.12 |
| Bench-trial soil + 1% P as H ₃ PO ₄ | 2 | 30 | 1.63 | 15.7 | 0.17 | 0.13 |
| | 2 | 60 | 1.62 | 16.3 | 0.19 | 0.13 |
| Field-trial soil | -- | 30 | 1.60 | 23.6 | 0.07 | 0.14 |
| | -- | 60 | 1.58 | 26.6 | 0.03 | 0.15 |
| Field-trial soil + 0.5% P as H ₃ PO ₄ | -- | 30 | 1.52 | 15.7 | 0.14 | 0.13 |
| | -- | 60 | 1.47 | 16.4 | 0.18 | 0.13 |
| Field-trial soil + 1% P as H ₃ PO ₄ | -- | 30 | 1.62 | 14.1 | 0.19 | 0.15 |
| | -- | 60 | 1.60 | 14.6 | 0.18 | 0.15 |

^a This split of the spiked solution was not subjected to the in vitro procedure, and is the standard against which the other spike samples should be compared.

Table 4. PBET results for baseline and amended Phase III soils

| | Total Mass of Metal In Soil Added to the PBET (mg) ^c | | | | | | | | | | | | Measured Concentration (mg/L) in PBET Extracts (mg/L) | | | | | | Total Mass of Metal PBET Extract (mg) ^f | | | | | |
|--------------------------|---|-------|------|-------------------------|------|------|------|-------|-------|-------------------|-------------------|-------------------|---|-------|-------|----|----|----|--|----|----|----|--|--|
| | Measured Concentration In Soil (mg/kg) | | | PBET Soil Mass (g, dry) | | | Pb | | | As | | | Cd | | | Pb | | | As | | | Cd | | |
| | ICP | XRF | | ICP | XRF | | ICP | XRF | | ICP | XRF | | Pb | As | Cd | Pb | As | Cd | Pb | As | Cd | | | |
| Baseline Soil | | | | | | | | | | | | | | | | | | | | | | | | |
| Phase III Soil | 1,630 ^a | 1,850 | 63.5 | 26.6 | 1.50 | 2.45 | 2.78 | 0.095 | 0.040 | 13.0 ^b | 0.35 ^b | 0.18 ^b | 1.94 | 0.052 | 0.028 | | | | | | | | | |
| Weathered Phase III Soil | 1,200 | 1,200 | 43.0 | 20.9 | 1.51 | 1.81 | 1.81 | 0.065 | 0.031 | 9.2 | 0.21 | 0.15 | 1.38 | 0.032 | 0.023 | | | | | | | | | |
| Amended Soil | | | | | | | | | | | | | | | | | | | | | | | | |
| Activated alumina | 973 | 1,000 | 40.0 | 15.4 | 1.51 | 1.47 | 1.51 | 0.060 | 0.023 | 7.2 | 0.29 | 0.10 | 1.08 | 0.044 | 0.015 | | | | | | | | | |
| EnviroBlend, Formula A | 1,340 | 1,700 | 47.0 | 23.0 | 1.58 | 2.11 | 2.68 | 0.074 | 0.036 | 1.7 | 0.37 | 0.15 | 0.26 | 0.055 | 0.023 | | | | | | | | | |
| EnviroBlend, Formula B | 1,720 | 2,200 | 61.0 | 27.5 | 1.51 | 2.60 | 3.33 | 0.092 | 0.042 | 12.3 | 0.44 | 0.18 | 1.84 | 0.065 | 0.027 | | | | | | | | | |
| Hydrous ferric oxide | 1,103 ^b | 1,430 | 47.3 | 22.3 | 1.51 | 1.67 | 2.17 | 0.072 | 0.034 | 7.3 ^d | 0.17 ^d | 0.14 ^d | 1.10 | 0.025 | 0.021 | | | | | | | | | |
| Iron filings | 1,040 | 2,000 | 40.0 | 23.1 | 1.50 | 1.56 | 3.01 | 0.060 | 0.035 | 5.6 | 0.19 | 0.11 | 0.84 | 0.029 | 0.016 | | | | | | | | | |
| Iron rich | 1,250 | 1,300 | 51.0 | 24.1 | 1.51 | 1.89 | 1.96 | 0.077 | 0.036 | 6.8 | 0.25 | 0.12 | 1.02 | 0.037 | 0.018 | | | | | | | | | |
| Manganese oxide | 1,190 | 1,100 | 44.0 | 30.0 | 1.50 | 1.79 | 1.65 | 0.066 | 0.045 | 7.8 | 0.31 | 0.12 | 1.17 | 0.046 | 0.018 | | | | | | | | | |
| Phosphate as TCP | 1,010 | 1,400 | 39.0 | 17.5 | 1.50 | 1.52 | 2.10 | 0.059 | 0.026 | 6.1 | 0.30 | 0.10 | 0.92 | 0.045 | 0.015 | | | | | | | | | |
| Portland cement | 1,155 ^a | 1,600 | 46.5 | 19.4 | 1.51 | 1.74 | 2.41 | 0.070 | 0.029 | 10.6 ^e | 0.56 ^e | 0.15 ^e | 1.58 | 0.084 | 0.022 | | | | | | | | | |
| Solucorp MBS | 1,760 | 2,400 | 64.0 | 26.8 | 1.57 | 2.76 | 3.76 | 0.100 | 0.042 | 4.5 | 0.61 | 0.13 | 0.68 | 0.092 | 0.020 | | | | | | | | | |
| Steel shot | 1,660 | 1,600 | 36.0 | 14.8 | 1.50 | 2.50 | 2.41 | 0.054 | 0.022 | 11.1 | 0.28 | 0.10 | 1.67 | 0.041 | 0.015 | | | | | | | | | |
| Zeolite | 1,090 | 1,500 | 43.0 | 19.0 | 1.50 | 1.64 | 2.26 | 0.065 | 0.029 | 8.1 | 0.38 | 0.12 | 1.22 | 0.056 | 0.018 | | | | | | | | | |

Table 4. (cont.)

| | Bioaccessibility ^a | | | | | | Percent Reduction in Bioaccessibility ^d | | | | | | | | | | | | |
|--------------------------|-------------------------------|----------------------|------------------|------------------|----------------------|------------------|--|----------------------|------------------|------------------|----------------------|------------------|------------------|----------------------|------------------|------------------|----------------------|------------------|--|
| | Pb | | | As | | | Cd | | | Pb | | | As | | | Cd | | | |
| | ICP ^b | Average ^c | XRF ^e | ICP ^b | Average ^c | XRF ^e | ICP ^b | Average ^c | XRF ^e | ICP ^b | Average ^c | XRF ^e | ICP ^b | Average ^c | XRF ^e | ICP ^b | Average ^c | XRF ^e | |
| Unamended Soil | | | | | | | | | | | | | | | | | | | |
| Phase III Soil | 79% | 93% | 70% | 55% | 68% | 69% | 69% | 77% | | | | | | | | | | | |
| Weathered Phase III Soil | 76% | 66% | 76% | 49% | 41% | 73% | 73% | 64% | | | | | | | | | | | |
| Amended Soil | | | | | | | | | | | | | | | | | | | |
| Activated alumina | 73% | 51% | 71% | 73% | 57% | 66% | 66% | 42% | 6% | 35% | 2% | -41% | -5% | 8% | 40% | | | | |
| EnviroBlend, Formula A | 12% | 12% | 10% | 74% | 68% | 62% | 62% | 60% | 84% | 85% | 87% | -43% | -24% | 13% | 15% | | | | |
| EnviroBlend, Formula B | 71% | 87% | 55% | 71% | 84% | 65% | 65% | 74% | 9% | -10% | 24% | -37% | -55% | 9% | -5% | | | | |
| Hydrous ferric oxide | 66% | 52% | 51% | 35% | 33% | 63% | 63% | 58% | 16% | 35% | 31% | 32% | 40% | 12% | 17% | | | | |
| Iron filings | 54% | 40% | 28% | 48% | 38% | 47% | 47% | 45% | 31% | 50% | 62% | 7% | 31% | 35% | 36% | | | | |
| Iron rich | 54% | 49% | 52% | 49% | 48% | 51% | 51% | 51% | 30% | 39% | 29% | 6% | 11% | 29% | 28% | | | | |
| Manganese oxide | 65% | 56% | 71% | 69% | 60% | 40% | 40% | 50% | 16% | 30% | 4% | -34% | -9% | 45% | 30% | | | | |
| Phosphate as TCP | 61% | 44% | 44% | 77% | 58% | 59% | 59% | 43% | 22% | 45% | 40% | -48% | -7% | 18% | 39% | | | | |
| Portland cement | 91% | 75% | 66% | 121% | 109% | 76% | 76% | 61% | -17% | 5% | 10% | -133% | -101% | -6% | 13% | | | | |
| Solucorp MBS | 25% | 31% | 18% | 91% | 114% | 47% | 47% | 53% | 69% | 61% | 75% | -76% | -109% | 34% | 25% | | | | |
| Steel shot | 67% | 79% | 69% | 76% | 54% | 69% | 69% | 43% | 14% | 0% | 5% | -48% | 1% | 3% | 39% | | | | |
| Zeolite | 74% | 58% | 54% | 87% | 73% | 63% | 63% | 50% | 5% | 27% | 26% | -66% | -34% | 11% | 28% | | | | |

^a This sample was evaluated in duplicate. The average result is reported here. See Table 8 for duplicate results.

^b This sample was evaluated in triplicate. The average result is reported here. See Table 8 for triplicate results.

^c PBET soil mass x soil metal concentration.

^d The PBET for this samples was performed in triplicate. The average result is reported here. See Table 10 for triplicate results.

^e The PBET for this samples was performed in duplicate. The average result is reported. See Table 10 for duplicate results.

^f Volume of PBET extract (150 mL) x metal concentration in PBET extract

^g Total mass of metal in PBET extract / total mass of metal in soil.

^h Bioaccessibility based on ICP-determined metal concentration in each soil subsample.

ⁱ Bioaccessibility based on the average of the ICP-determined metal concentration across the soil sample set.

^j Bioaccessibility based on XRF-determined metal concentration in each soil subsample.

^k Calculated as (bioaccessibility of baseline soil - bioaccessibility of amended soil) / (bioaccessibility of baseline soil)

Table 5. SPLP-extractable lead, arsenic, and cadmium concentration in soils

| | Replicate Number | SPLP-Extractable Concentration (mg/kg) | | |
|--------------------------|------------------|--|---------|---------|
| | | Lead | Arsenic | Cadmium |
| Baseline Soil | | | | |
| Phase III Soil | 1 | 2.68 | 3 u | 0.3 j |
| | 2 | 2 u | 3 u | 0.3 u |
| Weathered Phase III Soil | -- | 2.26 | 3 u | 0.3 u |
| Amended Soil | | | | |
| Activated alumina | -- | 2 u | 3 u | 0.3 u |
| EnviroBlend, Formula A | -- | 2 u | 3 u | 0.3 u |
| EnviroBlend, Formula B | -- | 2 u | 3 u | 0.3 u |
| Hydrous ferric oxide | 1 | 2 u | 3 u | 0.3 u |
| | 2 | 2 u | 3 u | 0.3 u |
| | 3 | 2 u | 3 u | 0.3 u |
| Iron filings | -- | 2 u | 21.2 | 0.3 u |
| Iron rich | -- | 2 u | 3 u | 0.3 u |
| Manganese oxide | -- | 2 u | 3 u | 0.3 u |
| Phosphate as TCP | -- | 3.80 | 3 u | 0.3 u |
| Portland cement | 1 | 2 u | 3 u | 0.3 u |
| | 2 | 2 u | 3 u | 0.3 u |
| Solucorp MBS | -- | 2 u | 3 u | 0.3 u |
| Steel shot | -- | 2.60 | 3 u | 0.3 u |
| Zeolite | -- | 4.72 | 3.78 | 0.3 u |

Note: u - not detected; value represents detection limit

j - cadmium was detected in the blank sample at 0.24 mg/L which is equivalent to a SPLP-extractable concentration of 0.48 mg-Cd/kg-soil. Therefore, this result was qualified.

Table 6. Lead, arsenic, and cadmium bioaccessibility values for Joplin soil samples

| | Measured Concentration in PBET | | | | | | | | |
|---|--------------------------------|-------------------|-------------------|------------------------------|-------------------|-------------------|-------------------------------|------|-----|
| | Metal Concentration (mg/kg) | | | Extracts (mg/L) ^b | | | Bioaccessibility ^d | | |
| | Pb | As | Cd | Pb | As | Cd | Pb | As | Cd |
| Bench-Trial Soil | 4,035 ^a | 10.8 ^a | 28.4 ^a | 22.8 ^c | 0.15 ^c | 0.15 ^c | 57% | 140% | 53% |
| Bench-Trial Soil + 1% P as H ₃ PO ₄ | 3,860 ^a | 9.0 ^a | 26.1 ^a | 16.2 ^c | 0.16 ^c | 0.13 ^c | 42% | 176% | 49% |
| Average Lead Concentration: | 3,950 | | | | | | | | |
| Field-Trial Soil | 4,820 | 12.0 | 29.0 | 26.6 | 0.07 | 0.15 | 55% | 54% | 52% |
| Field-Trial Soil + 0.5% P as H ₃ PO ₄ | 2,890 | 8.0 | 18.4 | 16.4 | 0.18 | 0.13 | 57% | 223% | 70% |
| Field-Trial Soil + 1% P as H ₃ PO ₄ | 4,280 | 7.0 | 24.8 | 14.6 | 0.19 | 0.15 | 34% | 266% | 61% |
| Average Lead Concentration: | 4,000 | | | | | | | | |

^a The soil metal concentration of this sample was determined in duplicate. The average result is reported.

^b Maximum lead concentration in the PBET extract following 30 and 60 minutes of reaction.

^c The PBET for this samples was performed in duplicate. The average result is reported.

^d Total mass of metal in PBET extract divided by the total mass of metal in soil added to PBET.

Table 7. Relative lead bioavailability and lead bioaccessibility results for amended and unamended Joplin soils

| | Animal Model Results | | PBET Results | | | |
|-------------------------------------|-----------------------|--------------------------|---|------------------------------------|---|------------------------------------|
| | Lead RBA ^b | Percent Reduction In RBA | Based on Measured Lead Concentration ^d | | Based on Measured Lead Concentration ^e | |
| | | | Lead Bioaccessibility | Reduction in Lead Bioaccessibility | Lead Bioaccessibility | Reduction in Lead Bioaccessibility |
| Bench-Trial Soil | | | | | | |
| Baseline soil | 0.59 | -- | 57% | -- | 58% | -- |
| Soil amended with 1% P ^a | 0.38 | 36% | 42% | 26% | 41% | 29% |
| Field-Trial Soil | | | | | | |
| Baseline soil | 0.59 ^c | -- | 55% | -- | 67% | -- |
| Soil amended w/ 0.5% P ^a | 0.45 ^c | 24% | 57% | -3% | 41% | 38% |
| Soil amended w/ 1% P ^a | 0.41 ^c | 31% | 34% | 38% | 36% | 45% |

^a Amended with H₃PO₄

^b Relative bioavailability adjustment values reported by Stan Casteel.

^c Draft results. Final results pending.

^d The individual sample lead concentration presented in Table 6 were used to calculate the bioaccessibility values.

^e An average lead concentration for each of the two substrates was calculated from the individual measurements presented in Table 2.

Table 8. Results of replicate analysis of soil samples
 (All units mg/kg unless otherwise noted)

| Sample ID | Replicate Number | <2-mm Fraction | | | <250-µm Fraction | | |
|-------------------------|------------------|----------------|---------|---------|------------------|---------|---------|
| | | Lead | Arsenic | Cadmium | Lead | Arsenic | Cadmium |
| Hydrous ferric oxide | 1 | 811 | 34.0 | 18.8 | 1080 | 48.0 | 22.2 |
| | 2 | 970 | 41.0 | 21.6 | 1040 | 47.0 | 22.7 |
| | 3 | 856 | 35 | 19.9 | 1190 | 47 | 21.9 |
| | Average: | 879 | 37 | 20 | 1103 | 47 | 22 |
| | RSD: | 9% | 10% | 7% | 7% | 1% | 2% |
| Phase III soil | 1 | 1660 | 62.0 | 27.2 | 1700 | 68.0 | 27.8 |
| | 2 | 1910 | 64.0 | 31.4 | 1550 | 59.0 | 25.4 |
| | Average: | 1785 | 63 | 29.3 | 1625 | 63.5 | 26.6 |
| | RPD: | 14% | 3% | 14% | 9% | 14% | 9% |
| | Portland cement | 1 | 986 | 38.0 | 17.7 | 1170 | 47.0 |
| | 2 | 1110 | 42.0 | 19.8 | 1140 | 46.0 | 18.6 |
| | Average: | 1048 | 40 | 18.75 | 1155 | 46.5 | 19.35 |
| | RPD: | 12% | 10% | 11% | 3% | 2% | 8% |
| Joplin Bench-Trial Soil | 1 | 3,780 | 9.0 | 26.5 | -- | -- | -- |
| | 2 | 3,940 | 8.9 | 25.6 | -- | -- | -- |
| | Average: | 3,860 | 8.95 | 26.05 | -- | -- | -- |
| | RPD: | 4% | 1% | 3% | -- | -- | -- |
| Joplin Field-Trial Soil | 1 | 3,990 | 10.9 | 28.7 | -- | -- | -- |
| | 2 | 4,080 | 10.7 | 28.0 | -- | -- | -- |
| | Average: | 4,035 | 10.8 | 28.35 | -- | -- | -- |
| | RPD: | 2% | 2% | 2% | -- | -- | -- |

Note: RSD - relative standard deviation

Table 9. Analytical results for NIST SRM 2710
(All units mg/kg)

| Analyte | Certified Concentration ^a | Measured Concentration ^a | |
|---------|--------------------------------------|-------------------------------------|-------------|
| | | Replicate 1 | Replicate 2 |
| Lead | 5100 | 4970 | 4940 |
| Arsenic | 590 | 590 | 610 |
| Cadmium | 20 | 23.0 | 23.5 |

^a Both the certified results as reported by NIST and the total concentration reported in this study are for samples digested using EPA method 3051 and ICP analysis of the extracts.

Table 10. Lead, arsenic, and cadmium concentrations in the replicate and triplicate PBET extracts
(Units mg/L unless otherwise noted)

| | Replicate Number | Lead | | Arsenic | | Cadmium | |
|-------------------------|------------------|---------|-----------|---------|---------|---------|---------|
| | | 30 Min. | 60 Min. | 30 Min. | 60 Min. | 30 Min. | 60 Min. |
| Hydrous ferric oxide | 1 | 6.20 | 8.17 | 0.153 | 0.200 | 0.150 | 0.156 |
| | 2 | 7.37 | 7.94 | 0.136 | 0.194 | 0.132 | 0.142 |
| | 3 | 5.69 | 5.79 | 0.096 | 0.113 | 0.108 | 0.124 |
| | | | RSD = 18% | | 29% | | 11% |
| Phase III Soil | 1 | 12.9 | 12.8 | 0.313 | 0.389 | 0.192 | 0.187 |
| | 2 | 13.0 | 13.1 | 0.291 | 0.308 | 0.165 | 0.176 |
| | | | RSD = 2% | | 23% | | 9% |
| Portland cement | 1 | 9.79 | 9.91 | 0.387 | 0.608 | 0.132 | 0.139 |
| | 2 | 10.5 | 11.2 | 0.424 | 0.518 | 0.144 | 0.156 |
| | | | RSD = 12% | | 16% | | 12% |
| Bench-trial soil | 1 | 21.9 | 22.1 | 0.069 | 0.154 | 0.144 | 0.142 |
| | 2 | 22.9 | 23.5 | 0.109 | 0.149 | 0.145 | 0.154 |
| | | | RSD = 6% | | 3% | | 7% |
| Bench-trial soil + 1% P | 1 | 14.8 | 16 | 0.111 | 0.121 | 0.108 | 0.122 |
| | 2 | 15.7 | 16.3 | 0.173 | 0.194 | 0.127 | 0.132 |
| | | | RSD = 2% | | 46% | | 8% |

Note: RSD - relative standard deviation

Table 11. Lead, arsenic, and cadmium concentrations in the spiked PBET extract samples

| Analyte | Initial Spike Concentration | Spike Concentration ^a | | Spike Recovery (percent) | |
|---------|-----------------------------|----------------------------------|-----------|--------------------------|-----------|
| | | 30 Minute | 60 Minute | 30 Minute | 60 Minute |
| Lead | 5.37 | 5.15 | 4.67 | 96% | 87% |
| Arsenic | 0.550 | 0.525 | 0.557 | 95% | 101% |
| Cadmium | 0.518 | 0.482 | 0.456 | 93% | 88% |

^a The PBET stomach solution was spiked with lead, arsenic, and cadmium at the concentration indicated in the initial spike concentration column. The spiked solution was then subjected to the PBET in the absence of soil.

Table 12. Lead, arsenic, and cadmium concentrations in PBET extracts of the NIST SRM 2710
(All units mg/L)

| | Lead | | Arsenic | | Cadmium | |
|--|---------|---------|---------|---------|---------|---------|
| | 30 Min. | 60 Min. | 30 Min. | 60 Min. | 30 Min. | 60 Min. |
| | | Maximum | | Maximum | | Maximum |
| Previous Studies | | | | | | |
| Michigan Site | 33 | 34.6 | -- | -- | -- | -- |
| KPC replicate 1 | 22.8 | 26 | 1.5 | 1.84 | 0.259 | 0.28 |
| KPC replicate 2 | 30.4 | 31.9 | 2.62 | 2.84 | 0.165 | 0.166 |
| KPC replicate 3 | 33 | 36 | 2.8 | 3.2 | 0.16 | 0.169 |
| Pleasant Hill | -- | -- | 2.04 | 2.28 | -- | -- |
| Rosiclare | 27.1 | 28.5 | 1.75 | 2.03 | -- | -- |
| Universal Minerals | -- | -- | | | | |
| Average of existing data | | 31.4 | | 2.44 | | 0.205 |
| Phase III Study | 35.3 | 33.9 | 2.71 | 2.99 | 0.162 | 0.166 |
| Relative percent difference ^a | | 12% | | 23% | | 19% |

^aRelative percent difference between the average maximum concentration of previous analysis of this sample, and the Phase III Study results.

Table 13. Quality control samples analyzed during the SPLP tests
(All units mg/L)

| | Replicate Number | Lead | Arsenic | Cadmium |
|--|------------------|--------|---------|---------|
| SPLP Solution Blank ^a | 1 | 0.1 u | 0.15 u | 0.024 |
| | 2 | 0.1 u | 0.15 u | 0.015 u |
| 5 ppm laboratory standard ^b | 1 | 5.11 | 5.13 | 5.08 |
| | 2 | 5.15 | 5.21 | 5.20 |
| | 3 | 5.20 | 5.16 | 5.15 |
| NIST 2710 Montana soild | -- | 0.10 u | 0.15 u | 0.015 u |
| Spiked SPLP solution ^c | -- | 5.23 | 0.35 | 0.22 |
| Spike recovery | | 105% | 174% | 112% |

Note: u - not detected; value represents detection limit

^a Sample of the SPLP solution that was not subjected to the SPLP procedure.

^b Laboratory standard containing 5 mg/L each of lead, arsenic and cadmium.

^c SPLP solution spiked with 5, 0.2, and 0.2 mg/L of lead, arsenic and cadmium, respectively. The spiked solutions were subjected to the SPLP tests in the absence of soil.

Attachment 1

**Procedures For *In Vitro*
Metals Bioassessibility**



Procedures For *In Vitro* Metals Bioassessibility Testing

The *in vitro* bioassessibility tests will be conducted according to the method outlined below. All chemicals cited are from the Sigma Chemical Company, unless otherwise noted.

- Prepare the stomach solution by adding the following compounds to 1 L of deionized water (stirred continually on a stir plate)
 - 1.25 g pepsin (50 mg, activity of 800–2,500 units/mg)
 - 0.50 g citrate (Fisher Chemical Co.)
 - 0.50 g malate (Aldrich Chemical Co.)
 - 420 μ L lactic acid (synthetic syrup 85 percent w/w)
 - 500 μ L acetic acid (97 percent w/w; Fisher Chemical Co.).
- Adjust the pH of the stomach solution to 1.5 by adding a measured volume of concentrated HCl.
- Add 150 mL of stomach solution to the 200-mL acrylic reaction vessel.
- Sparge the stomach solution with argon for 10 minutes to remove oxygen.
- Add 1.5 g of soil and seal the reaction vessel.
- Submerge the reaction vessel approximately half-way into a temperature-controlled water bath heated to maintain a constant 37 °C in the reaction vessel.
- Allow the soil/stomach solution to stand (no agitation) for 10 minutes.
- Stir the mixture with a plastic propeller stir rod mounted in a rheostat-controlled motor (Arrow Engineering Model 1750 motor on a rheostat setting of 2, resulting in approximately 150 rpm for the stir rod).
- Check the pH at 5-minute intervals, and readjust to pH 1.5 with HCl if necessary.
- Collect 5-mL and 50-mL samples at 30 and 60 minutes respectively, using a stainless-steel hypodermic syringe to pierce the sampling septum. Filter the samples through a 0.45- μ m acetate syringe filter.

- After the final sample is collected, measure and record the pH and final volume of the flask contents.
- Preserve the 5-mL sample with 50 μ L concentrated nitric acid and the 50-mL sample with 500 μ L concentrated nitric acid.
- Refrigerate the samples, and ship on ice to the University of Colorado laboratory.
- Analyze each of the two stomach-phase samples for lead, arsenic, and cadmium concentration, by inductively coupled plasma (ICP) spectroscopy.

Attachment 2

**Synthetic Precipitation
Leachate Procedure
(Modified)**



Synthetic Precipitation Leachate Procedure (MODIFIED)

The Synthetic Precipitation Leachate Procedure (SPLP) is conducted as follows:

1. Add 60 mL of concentrated H_2SO_4 and 40 mL of concentrated HNO_3 to 1 L of deionized water. This solution will be used to prepare the stock SPLP solution.
2. Prepare approximately 10 L of the SPLP extraction solution by adding dropwise the stock acid solution to 10 L of deionized water until the pH of the deionized water is 5.00 ± 0.05 s.u. Record the pH of the SPLP extraction solution.
3. Using 1 L of the SPLP extraction solution, prepare an SPLP spike solution in a labeled 1-L HDPE bottle. Amend the solution such that it contains 5 mg/L each of arsenic, lead, and cadmium.
4. Label and tare (to within 0.01 g) a 500-mL HDPE bottle and cover.
5. Add approximately 20 g of soil to the bottle and measure the mass of the bottle+soil to within 0.01 g.
6. Record the pH and specific conductivity of the SPLP extraction solution to ensure that the pH is 5.00 ± 0.05 s.u.. Add 400 mL of the SPLP extraction solution to the bottle, record the time and mass of the bottle+soil+extraction solution.
7. Repeat steps 4 through 6 for all of the soil samples.
8. Label and tare a 500-mL HDPE bottle and cover.
9. Record the pH and specific conductivity of the SPLP spike solution to ensure that the pH is 5.00 ± 0.05 s.u. Add 400 mL of the extraction solution to the bottle, and record the time and mass of the bottle+extraction solution.
10. Preserve the remaining SPLP spike solution with approximately 1 mL of concentrated HNO_3 , and submit it to CU for ICP analysis of arsenic, cadmium, and lead.
11. Place all of the bottles in an end-over-end rotating mixer. Rotate at 30 rpm for 18 ± 2 hours.
12. After at least 16 hours, remove the bottles from the end-over-end rotator and allow the solids to settle for 1–2 hours.

13. Filter all of the samples (including the sample containing the SPLP spike solution) through a 0.6- to 0.8- μm glass-fiber filter (e.g., Whatman GFF 0.7 μm filter).
14. Decant approximately 5–10 mL of the filtrate into a small beaker, and use this to measure the pH and specific conductivity of the filtrate.
15. Preserve the remaining filtrate with approximately 1 mL of concentrated HNO_3 , and submit it to CU for ICP analysis of arsenic, cadmium, and lead.
16. Transfer 400 mL of the SPLP extraction solution to a labeled 500-mL HDPE bottle, preserve it with approximately 1 mL of concentrated HNO_3 , and submit it to CU for ICP analysis of arsenic, cadmium, and lead.