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Bioaccessibility of lead from lead-contaminated soil upon phosphate amendment using a physiologically-based extraction test

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BIOACCESSIBILITY OF LEAD FROM LEAD-CONTAMINATED SOIL UPON PHOSPHATE AMENDMENT USING A PHYSIOLOGICALLY-BASED EXTRACTION TEST

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ABSTRACT

Lead is known to cause health problems in humans, especially children, and an effective in-situ remediation option has been sought for years. Adding phosphoric acid (PA) to contaminated soil causes a reaction that binds the lead to phosphate to produce pyromorphite ($Pb_5(PO_4)_3Cl$), a form of lead believed to be non-bioavailable; however, field trials have given varied results (Bosso et al 2008; Munksgaard and Lottermoser 2011; Tang et al. 2009). One explanation for these results might be the impact of the agent used to raise pH after phosphoric acid addition. In order to examine this explanation soil was collected from the Bonne Terre area in Missouri, which is known to have a high lead content due to past smelting activities. The soil was mixed with PA before calcium hydroxide and sodium hydroxide were added to the soil to neutralize the pH changes caused by the PA addition, and to determine whether the pH amendment impacted the rate of pyromorphite formation. The soil was then run through a physiologically-based extraction test (PBET) that simulates a child's stomach process to evaluate the success of the remediation attempt. The soil was monitored for a month after amendment addition, with all soil samples run through the PBET and a flame atomic absorption spectrometer to analyze the samples. Upon discovering that the change in concentration of extractable lead in soil was not statistically significant, an *in-vitro* test was conducted to discover what was occurring in the soil. Titration experiments were conducted based on the idea that pyromorphite was forming in the soil, but the low stomach pH was causing it to re-dissolve. The titration experiments showed that below pH 3, pyromorphite dissolves, a hitherto overlooked phenomenon.

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LIST OF ABBREVIATIONS AND ALTERNATE TERMS

i. PBET Physiologically based extraction test

ii. FAA Flame Atomic Absorption Spectrometer

iii. GFAA Graphite Furnace Atomic Absorption Spectroscopy

iv. Pb lead

v. Lime Calcium hydroxide ($Ca(OH)_2$)

vi. Lye Sodium hydroxide (NaOH)

vii. Pyromorphite Chloropyromorphite ($Pb_5(PO_4)_3Cl$)

viii. PA phosphoric acid (H_3PO_4)

ix. DI deionized (usually referring to deionized water)

x. Soln solution

xi. Conc concentration

xii. Std. Dev standard deviation

xiii. MDL method detection limit

1. INTRODUCTION

Why is lead in soil a concern? High lead levels in soil can cause health problems for humans, as well as for the flora and fauna who come into contact with the soil (Pourrut et al. 2011). Lead poisoning is a serious disease, especially for children, which can cause anemia and adverse effects on cognitive development (Ryan et al. 2004; Chaney, Sterrett, and Mielke 1984). Lead may be introduced into the body through oral ingestion, especially with young children who tend to put their toys and hands in their mouths often, and can also be introduced through the food chain due to plant uptake, leaching to surface and groundwater, or animal grazing (Tang et al. 2009). While lead is a naturally occurring metal that can be found in most soils, some areas have elevated lead levels due to human influence, such as old paint chips, leaded gasoline, mining and smelting operations, waste incineration, and even pesticide application. Lead tends to remain near the surface of the soil, which only makes it easier for children to come into contact with the lead (Ryan et al. 2004). While remediation of lead contaminated soils is primarily focused on areas near mining and smelting facilities, elevated lead levels can be found just about anywhere, including urban soils and gardens. Although the amount of lead exposure from inadvertent soil ingestion is greater than that from consuming products of urban gardens, lead contamination in gardens should not be overlooked as plants can uptake lead from the soil, as well as accumulate a surface coating of lead contaminated soil or dust (Chaney, Sterrett, and Mielke 1984). There are a number of factors that influence human lead absorption from soil and dust, primarily: nutritional factors, amount of soil and dust ingested, and concentration of lead in the soil and dust ingested (Chaney, Sterrett, and Mielke 1984). One of the best ways to reduce lead exposure in areas of lead contamination is to perform routine household cleaning and ensure children washed their hands frequently, especially after playing outside. Other ways to limit lead intake from contaminated soil, dust, and produce is to provide children with a balanced diet with adequate iron and calcium, keep gardens a safe distance from roadways and buildings with lead-based paint, wash all fruits and vegetables before consumption, replace or cover contaminated soil, and plant grass on bare yard areas (Chaney, Sterrett, and Mielke 1984). Nonetheless, recent blood lead level (BLL) testing data indicates 712 children under six years of age were identified with elevated BLLs in Missouri alone, primarily due to old houses still containing lead-based paint and other old fixtures that may leach lead (McManus et al. 2015).

1.1. METHODS OF DEALING WITH LEAD IN SOIL

Lead and other heavy metals can be difficult to deal with, as many common remediation techniques will not work (such as soil vapor extraction, bioremediation, vacuum extraction, phytoremediation, and natural attenuation). The most effective lead remediation method is currently soil removal, which can cost between $80 and $170 per cubic yard of soil excavated in Missouri, and is dependent on what area of the state the excavation is taking place ("Cost to Remove Dirt" 2015). Other options include covering (using sod, mulch, or clean soil) or dilution (by mixing with uncontaminated soil) which can also be costly, depending on the extent of the soil contamination (Ryan et al. 2004; Scheckel et al. 2013). Many researchers have attempted to use phytoremediation, but most have found that while lab experiments suggest plants could work, the field experiments produce results suggesting plants could take over 100 years to bring soil lead levels within an acceptable range (Brunetti et al. 2011; Van der Ent et al. 2012; Cheng et al. 2015). Phytoremediation also tends to also raise the question of what happens to the lead-containing plants once they have accumulated as much lead as they can. The lead cannot be broken down into harmless forms or volatilize away, so the plants must be collected and disposed of in some way that does not re-contaminate an area with the lead in the plants. Immobilization is another option to deal with lead contaminated soils, and the theory is that changing the form of lead to something that is stable and not bioaccessible, such as adding phosphate to lead contaminated soil to form pyromorphite, would solve the problem without going through the costly soil removal process. The two main types of lead contaminated sites are shooting range soils and mining/smelting impacted soils. Lead immobilization for shooting range soils has been extensively studied, and almost all researchers seem to agree that phosphate amendment is not a viable option for shooting range soils (Butkus and Johnson 2011; Dermatas et al. 2008; Chrysochoou 2007). This could be due to the relatively large particle size of the lead present in the soil, the potential presence and interference of other compounds from the spent bullets that could undergo leaching during the amendment process, or other factors.

1.2. PYROMORPHITE AS A MEANS OF REMEDIATION

Quite a few studies and models have been published related to the theory of lead immobilization in soil. These studies all seem to agree that the most viable option for lead immobilization is to add a phosphate source to the soil in order to transform the lead species present in the soil into pyromorphite, which is a form of lead that is relatively stable at a wide range of temperature, pH, and soil conditions. According to Porter et al. (Porter et al. 2004), the three best candidates for lead immobilization are galena, chloropyromorphite, and wulfenite. While galena, PbS, is relatively insoluble, it is subject to oxidation when exposed to air and will form anglesite, $PbSO_4$, which is much more soluble. Forming wulfenite, $PbMoO_4$, requires adding molybdates to the soil, which would only cause other problems (considering the harmful impacts that large quantities of molybdenum has on humans). Chloropyromorphite, $Pb_5(PO_4)_3Cl$, only requires the addition of phosphate (which is a common fertilizer and relatively harmless to living organisms), and is the most stable lead mineral found under normal environmental conditions, making chloropyromorphite the most practical form of lead for lead immobilization (Porter et al. 2004; Scheckel and Ryan 2002). Hydroxy- and fluoropyromorphite also may form and have similarly low solubility; collectively the term pyromorphite is used for all three forms. When calculating how much phosphate to add to the soil in order to achieve lead immobilization, other phosphate receptors must be considered. Aluminum, iron, calcium, magnesium, and manganese are all elements commonly found in soil that could impact pyromorphite formation; however, aluminum, iron, and magnesium will not control the phosphate as long as calcium is present in its usual abundance, and manganese is typically present in concentrations lower than typical phosphate concentrations, so calcium and manganese are the greatest phosphate sinks in soil (Porter et al. 2004). Calcium is typically present in large enough concentration that this could impact the effectiveness of the soil amendment if not enough phosphate is added to react with both the calcium and the lead present in the soil. In addition to transforming the lead to pyromorphite, phosphate addition leads to precipitation of the calcium in the soil to apatite, which could impact the friability of the soil (Porter et al. 2004; Miretzky and Fernandez-Cirelly 2008).

Pyromorphite ($Pb_5(PO_4)_3X$) is a general term that is used for three compounds, determined by the ion represented by X in the pyromorphite chemical formula. The most common pyromorphite varieties are chloropyromorphite (X=Cl), hydroxypyromorphite (X=OH), and fluoropyromorphite (X=F). The three most common forms of apatite ($Ca_5(PO_4)_3X$) are fluorapatite ($Ca_5(PO_4)_3F_2$) chlorapatite ($Ca_5(PO_4)_3Cl_2$) and hydroxyapatite ($Ca_5(PO_4)_3OH_2$). Apatite can substitute the Ca^{2+} ion for a Pb^{2+} ion to transform from a common apatite to a form of pyromorphite. Although a wide range of other substitutions is possible (and briefly discussed previously in this section), the interaction of Pb^{+2}, Ca^{2+}, and PO_4^{3-} is the primary concern when transforming apatite into pyromorphite. This transformation results in a Pb^{+2} ion becoming part of a relatively insoluble pyromorphite, and is thus no longer bioavailable, which is a highly attractive form of lead remediation in soil. According to the conference paper by Chairat C. et al (2004), there is a poor understanding of the thermodynamic and kinetic properties of apatite in near surface processes. They state that apatite dissolution rates have been measured at pHs from 2 to 11.8, and dissolution rates of apatite decrease monotonically with increasing pH. The problem with binding lead to pyromorphite is that the ion substitution could result in transformation of pyromorphite into apatite, as the Porter et al and Miretzky and Fernandez-Cirelly papers warn, if enough calcium is present in the soil to replace the lead ions.

A relatively recent study of pyromorphite solubility conducted by Topolska et al (2016) determined the solubility of pyromorphite in dissolution experiments and found the solubility constant for pyromorphite to be $K_{sp,298}=10^{-79.6\pm0.15}$, the enthalpy of formation to be ΔH°_{f}=-4108.4±7.9 kJ·mol^{-1}, and the Gibbs free energy of formation to be ΔG°_{f}=-3764.3±3.5 kJ·mol^{-1}. These numbers were determined using synthetic pyromorphite, and the data showed the enthalpy of dissolution reaction decreased with the increase of temperature. According to Miretzky and Fernandez-Cirelli (2008) mobility of lead depends on many factors: Pb speciation and total Pb soil content, the type of soil, soil pH, moisture content of the soil, and water infiltration. Lead phosphates have low solubility, several orders of magnitude less soluble than the analogous carbonates and sulphates. A decrease in solubility is also a decrease in mobility, which reduces the risk of lead moving from soils into groundwater or surface water. When Pb and P interact they reduce Pb solubility and bioavailability by forming pyromorphite, which has extremely low solubility and is thus extremely attractive as a remediation method. Lowering the soil pH was found to significantly enhance dissolution of soil Pb and encourage pyromorphite formation, as pyromorphite formation is kinetically controlled by pH, solubility of the phosphate source, and solubility of the Pb species.

1.3. PHOSPHATE AMENDMENTS

There have been many studies on pyromorphite formation using phosphate sources, and many different phosphate sources have been evaluated. Some of the most common phosphate sources are rock phosphate ($Ca_3(PO_4)_2$), phosphoric acid (H_3PO_4), hydroxyapatite ($Ca_5(PO_4)_3(OH)$), calcium phosphate ($Ca_3(PO_4)_2$), and monocalcium phosphate ($Ca(H_2PO_4)_2$). Lead extractability into the soil solution and resulting pyromorphite formation tends to increase with increased P concentration and P solubility (Scheckel et al. 2005). For this reason, phosphoric acid tends to be the most effective in terms of lead immobilization, and the literature reports relative equilibrium was achieved over a period ranging from minutes to days, primarily dependent on the media (with liquid media attaining equilibrium within minutes, and soils taking days). Phosphoric acid is not the ideal solution for lead contaminated sites due to the fact that more acidic solutions are more effective at mobilizing lead (allowing it to more easily bind with the phosphate), and the reaction of the public when told their neighborhood soils will be treated with acid. The decrease in soil pH caused by phosphoric acid addition can also cause leaching of heavy metals, especially in low-buffering sandy soils (Melamed et al. 2003). Calcium phosphate and phosphate rock have very little impact on soil pH, making them more attractive choices for remediation efforts, even though they work more slowly than the phosphoric acid amendments.

Other phosphate amendment options, such as fish bones, cow bones, calcined oyster shells, DAP (Diammonia phosphate), agricultural limestone, potassium orthophosphate, and even biosolids compost have been studied and proved to be successful at transforming lead to less soluble (pyromorphite-like) forms (Giammar et al. 2008; Moon et al. 2013; Basta and McGowen 2004; Munksgaard and Lottermoser 2011; Brown et al. 2003). Many of these phosphate options can lead to the release and/or mobility of As, Cu, Mn, Sb, Zn, and other potential detrimental elements, or have other factors that make them less attractive when compared to the traditional phosphoric acid or phosphate rock options.

1.4. BIOAVAILABILITY ANALYSIS METHODS

Tests for lead in soil such as x-ray fluorescence do not indicate the potential for biological uptake of lead. Bioavailability can be tested *in-vivo* or *in-vitro*. While using human subjects for *in-vivo* tests would yield the most accurate results, few researchers have been able to use actual human test subjects due to ethical and monetary dilemmas. Graziano et al. (1996) are one of the few groups to perform lead bioavailability tests on human subjects. They examined the bioavailability of lead attributed to lead crystal decanters. Their test subjects were carefully chosen and monitored, and the test itself involved giving the subjects sherry containing a known lead concentration (the lead concentration was due to the sherry being stored in a lead crystal decanter for 3 years). They found that lead intake from wine can be significant, and can even exceed that from diet, water, air, and dust combined. The most common *in-vivo* tests depend on using pigs or mice because more tests can be performed on these subjects without causing as many ethical and monetary setbacks (Juhasz et al.

2014; Ryan et al. 2004; Scheckel et al. 2013). One potential problem with using pigs and mice as *in-vivo* models is that no single animal can mimic the GI tract of a human, and there is some debate as to which animal is most appropriate to research *in-vivo* lead bioavailability (Ryan et al. 2004). *In-vivo* studies can be extremely expensive, and include many complicating factors that make *in-vivo* studies unappealing to most researchers.

The two most common *in-vitro* methods of testing lead concentration in soil which might be bioavailable, dubbed bioaccessible lead, are sequential extraction and the physiologically-based extraction test (PBET) (Scheckel 2005; Wragg and Cave 2003). It is commonly believed that although sequential extraction has its uses, it tends to over-predict how much lead is in the form of pyromorphite (Scheckel et al. 2005; Scheckel et al. 2003; Ryan et al. 2001; Tai 2013). The sequential extraction process itself has great potential to create insoluble forms of lead (such as pyromorphite) during the process, which would account for the over-prediction of pyromorphite, causing sequential extraction to be an ineffective indicator of bioaccessible lead in the tested soil. For this reason, PBET tends to be considered a more reliable choice for determining lead bioaccessibility.

The PBET, although not a perfect replica of the human digestive system, can act as a less expensive (and less ethically controversial) means to determine lead bioavailability in soils. Ruby et al.'s article (1996) was one of the first to use a PBET to test lead bioavailability. They observed that lead bioavailability was primarily controlled by the stomach phase of the PBET, and that when the acidic stomach environment was neutralized the lead tended to precipitate or adsorb and was thus not available for absorption by a human GI tract. While the PBET was made based on data from human children, it is hard to test how well the model would correlate to *in-vivo* tests as intentionally causing lead poisoning in children is highly unethical. The research groups who tested how well PBET data fits *in-vivo* data use either mice or pigs, as these are believed to have similar digestive systems to human children. Juhasz et al. (2014) was one of the few research groups to use mice in their experiments. When the mice were fed lead contaminated soil after phosphate amendment application, the pyromorphite concentration in the mice waste was greater than that in the initial soil fed to the mice. Their research indicates that phosphate amendments result in a decrease in relative bioavailability of lead, and that gastrointestinal processes could lead to formation of insoluble lead forms even if the soil fed to the mice still contained soluble lead forms.

1.5. IMPACTS OF pH ON BIOAVAILABILITY

Ruby et al.'s article (1996) observed that lead bioavailability was primarily controlled by the stomach phase of the PBET, and that when the acidic stomach environment was neutralized the lead tended to precipitate or adsorb and was thus not available for absorption by a human GI tract. Based on their PBET data at stomach pH of 2.5 in a linear regression model they found the PBET accurately predicted relative lead bioavailability in rats. The Sprague-Dawley rat model can then be used to estimate absolute lead bioavailability in children. The PBET was created based on fasting conditions of a human child, which would produce the most soluble lead and thus would be the most conservative GI conditions. They found that fasting pH can range from 1 to 4, and had a mean fasting pH value of 1.7 to 1.8. Other researchers have tested the PBET at varying pHs in order to determine whether PBET pH effected lead extractability. Scheckel et al. (2005) performed a PBET at 3 different pHs (1.5, 2.0, and 2.5) and observed a decrease in extractability with an increase in pH, so more lead was bioavailable at lower pH. Tang et al. (2004) looked at both the gastric and intestinal phases, and observed that while there was a high amount of soluble lead present in the gastric phase (at pH 1.7), the amount of soluble lead dropped significantly in the intestinal phase (pH 7). Li et al. (2013) also observed that the bioaccessibility of soil lead was pH-dependent and that lead became less bioaccessible after the pH drop in the intestinal phase. Wragg and Cave (2003) also agree that the small intestinal phase of the PBET can be ignored for lead bioaccessibility studies, as lead is relatively insoluble at pH values greater than 5.5 and would therefore be excreted with other solid matter. Although the common theme in these papers is that lead bioaccessibility increases as pH decreases, it was widely believed that pyromorphite's stability over a wide range of pH's would allow pyromorphite to pass through the entire digestive process relatively unchanged.

1.6. DOES PHOSPHATE REMEDIATION WORK: A SHORT REVIEW

Studies considering the thermodynamics, solubility, and kinetics of lead immobilization all agree that, in theory at least, phosphate addition to form pyromorphite would be the best option (Porter et al. 2004; Scheckel and Ryan 2002). Many researchers have noticed that phosphate addition to soil causes a decrease in extractable lead (Ruby, Davis, and Nicholson 1994; Giammar, Xie, and Pasteris 2008; Melamed et al. 2003; Cao et al. 2003; Moon et al. 2013; Yang et al. 2001; Tang and Yang 2012; Tang et al. 2009; Miretzky and Fernandez-Cirelli 2008; Basta and McGowen 2004; Bosso, Enzweiler, and Angélica 2008; Tang et al. 2004; Laperche et al. 1997; Juhasz et al. 2014). Some researchers have even verified with X-ray diffraction (XRD) and scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM-EDX) that phosphate addition does indeed form pyromorphite in the soil (Ruby, Davis, and Nicholson 1994; Melamed et al. 2003; Cao et al. 2003; Moon et al. 2013; Yang et al. 2001; Tang et al. 2009; Bosso, Enzweiler, and Angélica 2008; Laperche et al. 1997; Juhasz et al. 2014). It has been reported in a few cases, though, that although field tests show a decrease in lead bioavailability, the corresponding *in-vivo* tests tend to show a greater amount of bioaccessible lead than the *in-vitro* tests led them to expect (X Tang et al. 2009). Other researchers tend to be hesitant to support phosphate amendments due to the potential for phosphate leachate and possible promotion of eutrophication (Munksgaard and Lottermoser 2011).

Mosby's thesis paper (2000) was part of a study that took place in multiple locations by a couple of research groups working together on the research but writing their own separate papers. These studies looked at lead contaminated soil in lab, field, pig, and plant studies. Mosby's focus was the lab and field soil studies, however his paper gives a summary of the results for the pig and plant studies performed in the other labs on the same soil by the other researchers. For the lab study he mixed phosphoric acid (PA) and potassium chloride (KCl) to the soil in an attempt to form chloropyromorphite. The field studies involved multiple plots with various ratios and types of amendments. Ten days after the field treatments, quicklime was added to the soil and fescue was planted. At the end of the lab and field studies he observed that the lead species present in the soil had transformed to chloropyromorphite, as desired. The fescue plots indicated that the lead tended to stay in and around the roots of the plants, and seemed to support the idea that phosphate amendment led to immobile forms of lead in soil. Meanwhile, the soil that was sent off for the pig studies did not produce the results they were expecting. The pigs were fed the soil and then data was collected from the pigs to obtain a relative bioavailability (RBA) of lead from the soil. The pig studies showed that the RBA of lead increased after the phosphate treatments for their positive control plot soils (the soil with only PA added), which was the opposite of what the researchers were expecting. After identifying chloropyromorphite formation in the soil, which is widely regarded as the least bioavailable form of lead, the researchers expected the pig studies to confirm the common theory that phosphate remediation would lead to a drastic decrease in RBA of lead from ingesting lead-contaminated soil.

1.7. RELATED RESEARCH IN THE FIELD

While most research focuses on whether phosphate addition will lead to a decrease in bioavailability, there have been a handful of related articles that deal with other aspects of phosphate amendment. Yang and Mosby (2006) evaluated rototilling, surface application, and pressure injection as potential treatment methods, and found that rototilling was the most effective in terms of phosphate homogeneity and the reduction of lead bioaccessibility in the treated soil zone. Another study looked into the effect of temperature on pyromorphite formation, and observed that higher temperatures favored transformation of soil lead to non-bioaccessible forms (Yang et al. 2001). Other soil conditions have also been studied, in an attempt to determine what soil conditions are the most favorable to promote pyromorphite formation. Debala et al. (2013) looked at the addition of organic acids to the soil, and determined that adding organic acids does not promote the formation of pyromorphite in phosphate amended soils containing lead. They theorized that organic acids are naturally present in rhizosphere soil, and can be considered a factor contributing to the poor efficiency of metal phosphate formation in phosphate amended soil. Topolska et al. (2013) found that phosphate-solubilizing bacteria in the soil have the potential to increase the solubility of pyromorphite, so knowledge of the soil ecosystem and conscious phosphate management could be crucial for long-term effectiveness of phosphate amendments. In addition to favorable soil conditions, many researchers have attempted to determine if plants could be used as a detection system to determine what phosphate source is the most effective at forming un-bioaccessible forms of lead. A study on plant lead uptake showed that shoot tissue lead contents decreased significantly after P amendment, however root lead increased after P addition (X. Cao et al. 2002). The researchers determined that a mix of phosphate rock and phosphoric acid would be the most effective amendment to immobilize lead in contaminated soils while minimizing pH impact and eutrophication potential. There have even been studies on how to prevent lead bioaccessibility after the soil has already been ingested. Scheckel and Ryan (2003) observed that the phosphoric acid derived from cola soft drinks causes an instantaneous formation of pyromorphite from bioavailable lead sources ($PbCl_2$ and Pb paint) under stomach conditions. Based on this research, they believe that drinking any beverage containing phosphoric acid could nearly eliminate the absorption of bioavailable lead via ingested lead sources.

2. METHODS

2.1. PURPOSE AND EXPERIMENT OVERVIEW

Lead contaminated soil was initially studied to determine whether lye amendment would act as a more efficient pH stabilizer than lime in soils that had been treated with phosphoric acid in order to form lead compounds that were not bioaccessible. Quicklime is a common amendment for soils with elevated pH; however, addition of calcium compounds to lead contaminated soils amended with phosphoric acid could cause dissolution of the otherwise stable lead compounds, back into bioaccessible forms of lead. The PBET analysis method was chosen in order to compare lead bioaccessibility in a fasting child's digestive system. Initial objectives included the following:

- Characterize soil
- Add amendments to soil and track changes in Pb concentration with PBET
- Analyze impact of lye vs. lime amendment on Pb concentration

After weeks of data showing no statistically significant change in concentration, a bench-scale study was performed to determine an explanation for the unexpected data. This led to a refocusing of the research objectives to include the following:

- Run titration tests to verify pyromorphite solubility
- Compare titration pH data to PBET pH to explain unexpected results

The bench-scale study indicated that although a precipitate formed when adding phosphate to lead nitrate (indicating the presence of a lead form such as pyromorphite which would not be bioaccessible), this precipitate dissolved once the pH was lowered below a pH of 2. A more in-depth titration study was then performed in order to test the hypothesis that lead becomes soluble at low pH, which would explain the unexpected PBET results, which were performed using a fasting child's stomach pH of approximately 1.8. A follow-up experiment was then run on the lead-contaminated soils at a pH of 1.8 and a pH of 3, in order to further test the theory that the pH of a fasting child's stomach would re-dissolve the lead compounds thought to be not bioaccessible (after phosphate amendment).

2.2. SOIL CHARACTERISTICS

Soil characteristics were determined in order to accurately determine how much of each amendment to add to each bucket of soil, as described in the following sections.

2.2.1. Collection. Soil was collected from the USEPA soil repository in Bonne Terre, Missouri off of Hedgeapple Lane and brought to the lab in plastic tubs covered in plastic tarps. Soil at the repository originates from yards which had more than 400 ppb lead based on survey with x-ray fluorescence spectrometers. Standard practice is to excavate the first one to two feet of lead-contaminated residential yards.

2.2.2. Homogenization and Characterization. Once in the lab, the soil was homogenized by shoveling all the soil into a pile in the middle of a tarp, then shoveling the soil from the pile outward, creating a large ring of soil around where the original soil mound used to stand. The ring of soil was then pushed back together to create a mound of soil in the center of the tarp. This process was repeated three to five times with a steel shovel to ensure the soil was well-mixed. Soil was then transferred to five-gallon plastic buckets by plastic hand trowel to a depth of approximately one foot (roughly three-quarters filled). Each bucket of soil was tested for calcium using a Perkin Elmer Flame Absorption Spectrometer (FAA), and pH using a pH probe. For the calcium test, 10 g soil samples were collected by hand and dissolved in 50 mL HCl overnight before the liquid portion was run through the FAA to obtain a calcium concentration.

To determine the pH of the soil, two tests were run and compared, a test with DI water and a test with $CaCl_2$. Using $CaCl_2$ is believed to give a more accurate pH value as it is said to be more resistant to seasonal changes in salts and other soil factors (USDA 2014). The first test involved mixing the soil in a 1:1 ratio with DI water to create a soil slurry from which the pH was determined. The second test was performed by dissolving soil in a 0.01 M $CaCl_2$ solution at a 1:2 ratio. These two tests were then compared and the average used as the pH for each bucket. No post-amendment pH's were collected due to unexpected results that led to refocusing the experiment more on the PBET process and less on the soil itself. Another researcher at Missouri S&T conducting similar research on Bonne Terre soils (and using the same pH testing method) provided a pH from his experiment that correlates with the post-amendment pH for the bucket of soil containing only phosphoric acid (PA). This pH, provided by Austin Doss of Missouri S&T, gives a post-PA amendment pH of approximately 6.02.

To obtain an estimate of the initial lead concentration in the soil, multiple soil samples were collected and averaged to determine the approximate overall lead concentration in the soil. For each sample bucket, a soil sample was taken from five locations around the surface of the soil and these samples were hand-mixed to obtain a representative composite sample for each bucket. After these representative samples were collected, they were all sieved through a 250 micron (#60) sieve before being placed in an oven set to 100°C to dry overnight. From these dried soil samples, a 0.5 g sample was taken to represent each bucket of soil. An additional sample was taken that was an equal mix of all eight soil buckets, as an overall cumulative soil sample. Each sample was dissolved in 5 mL of HCl to mobilize the lead, then diluted with DI water to a total volume of 50 mL. After being mixed overnight, the samples were vacuum filtered and the liquid extracted was analyzed using the FAA to determine the lead content for each bucket, and an overall lead content of the soil.

Soil moisture was periodically determined by collecting soil in a pre-weighed aluminum tare can, then the tare can of soil was weighed, dried overnight in an oven, and then weighed again to determine the water content in the soil based on the change in weight of the soil in the tare can. Each bucket of soil was weighed on a large scale and the water weight was then subtracted from the weight of the soil (based on a soil moisture test taken around the same time as when the soil buckets were weighed), and the dry soil weight was then used for the amendment addition calculations. To ensure the soil in the lab matched approximate field conditions, the soil was watered prior to amendment additions to raise the moisture content. This was accomplished by adding around 1 L of distilled water every couple days and measuring the water content until the water content reached between 25% and 30%. These numbers were used based on Cornell University's claim that the volumetric soil moisture content remaining at field capacity is about 15 to 25% for sandy soils, 35 to 45% for loam soils, and 45 to 55% for clay soils (Cornell University 2010), and the observation that the soil used was a mix of sandy and loam soil.

2.3. PHYSIOLOGICALLY BASED EXTRACTION TEST (PBET) EXPERIMENT

The physiologically based extraction test (PBET) was used to simulate a human child's stomach process to evaluate the change in concentration of bioavailable lead.

2.3.1. Soil Amendments and Sampling. Eight treatment variations were used at differing time intervals, as shown in Table 2.1. The amount of lab-grade phosphoric acid (H_3PO_4), lime ($Ca(OH)_2$), or lye (NaOH) to add were determined from measured soil properties. The volume of additions, and corresponding calculations, can be found in Appendix A.

Table 2.1: Soil Treatments – Variations of Lime and Lye

Bucket	Initial Amendment	Day 5 Amendment	Day 20 Amendment
1	None - control		
2	PA		
3	PA + lime		
4	PA	+ lime	
5	PA		+lime
6	PA + lye		
7	PA	+lye	
8	PA		+lye

The lead-to-phosphorous and calcium-to-phosphorous ratios used were 5:3 (determined through stoichiometry of chloropyromorphite, $Pb_5(PO_4)_3Cl$, and apatite, $Ca_5(PO_4)_3(OH, Cl, F)$). The molar amount of lead and calcium were used to determine the molar amount of phosphoric acid required to form pyromorphite, assuming phosphate would preferentially or competitively react with calcium to form apatite. Phosphoric acid would leave soil quite acidic, so the amount of required neutralization also was calculated. After determining the molar amount of phosphoric acid required to form pyromorphite and apatite, and taking into account that at a pH around 7 the phosphoric acid would be about half monobasic and half dibasic, the amount of lye (NaOH) required to counteract the H^+ ions released was determined stoichiometrically. The amount of PA (and corresponding lime) necessary to counteract the lime required was determined using the solver function in excel, as the lime contains calcium, so more phosphorous had to be added to balance out the calcium addition and resulting assumed apatite formation. Calculations can be found in Appendix A.

The initial amendment of phosphoric acid was added to all buckets, excluding the control, and the addition of either lime or lye was added to the buckets at different time intervals in order to determine if a time delay on pH neutralizer addition impacted the effectiveness of the phosphoric acid in forming pyromorphite (see Table 2.1). All amendments were mixed into the soil using a plastic hand trowel, ensuring the top 6 inches of soil was well mixed. Figure 2.1 shows the setup of the buckets, which were numbered 1 to 4 (bottom left to bottom right of the photo) and 5 to 8 (top right to top left of the photo). The white substance visible in the figure in buckets 5 and 8 are lime and lye, respectively, and they are about to be hand-mixed into the soil.

Figure 2.1: Bucket Setup – After Amendment Addition, Immediately Before Mixing

To simulate rainfall, 652 mL of distilled water was added to each bucket every three days, based on the average annual rainfall data for the Bonne Terre area. The Bonne Terre area experiences approximately 43 inches of rainfall annually, with an average temperature around 55°F ("Climate Missouri" 2014). Samples were collected from 2-4 inches below the surface of the soil from multiple locations around the bucket, then these samples were mixed together to form a representative sample of the bucket. The sample was then sieved using a 250 µm sieve. The <250 µm soil was used for the PBET analysis because it is the size range which can adhere to hands and thus be available for digestion (Ryan et al. 2004). After sieving, the soil was oven dried for a minimum of 12 hours at 110° C. Of this dried soil sample, 0.4 g was assayed by PBET, with the resulting lead content found by FAA. This process was repeated for a total of three times for each bucket of soil. Soil characterization data can be found in Appendix A.

The method detection limit (MDL) for the FAA was determined based on the samples of 0, 1, 2, 3, 4, and 5 mg/L from the initial lead sample testing. While the FAA started off reading the 0 mg/L sample as an adsorption of 0.000, by the time all the samples had been run the 0 mg/L sample was consistently giving an adsorption of 0.011, and thus everything below an adsorption of 0.011 was below detection for the FAA. That adsorption was greater than the 2 mg/L sample, and was close to the 3 mg/L sample. The MDL graph in Appendix A, Figure A 1.2 shows the range of values below detection for the FAA, along with error bars for the rest of the values evaluated. Before any soil was run through the PBET system, blanks containing no soil were run through the system to ensure none of the equipment would introduce lead into the experiment. Other quality control and quality assurance precautions were taken throughout the experiment, and are discussed in the following sections.

2.3.2. PBET/FAA Testing Procedures. The PBET procedure used for this experiment was adapted from the Ruby and Davis et. al (1996) procedure as described below. The entire digestive process can take many hours, but researchers have found that the lead extracted during the intestinal phase of the PBET is significantly less than that of the stomach phase (Li and Zhang 2012), so this research focused on the stomach phase to provide an expedient and conservative model of lead bioavailability. Although a water bath was available to maintain stomach temperature during PBET testing, it would not fit in a fume hood. The PBET apparatus therefore consisted of a 15-gallon glass tank in a fume hood with Tygon tubing connected to a water pump submerged in a heated water bath outside the fume hood to pump heated water into the tank, and a gravity siphon system allowing water to circulate from the tank back to the heated water bath. The tank in the fume hood was wrapped in bubble wrap to prevent heat loss. An image of this system can be seen in Figure 2.2.

Figure 2.2: PBET Setup – View of PBET Tank and External Heating Tank

This system allowed for the water to be circulated and heated to 37°C (human body temperature). Nalgene separatory funnels (each 1 L) were used as the artificial stomachs, and were held in the glass tank with ring stands so that the mouth of the Nalgene separatory funnel was above water but the majority of the funnel was submerged in the water. Four separatory funnels could be run at the same time, and each funnel had a Tygon tube attached to the bottom of the funnel to allow nitrogen to be pumped in and provide mixing.

Each 0.4 g soil sample was collected and dried as described in Section 2.3.1, then mixed with 40 mL of gastric solution in the separatory funnel. The gastric solution was prepared by adjusting 1 L of DI water to the selected pH of 1.8 with HCl, then mixing it with 1.25 g pepsin, 0.5 g citrate, 0.5 g malate, 420 µL lactic acid, and 500 µL of acetic acid. The pH value of 1.8 was selected based on average pediatric pH in a fasting stomach (Ruby et al. 1996). Figure 2.3 shows the tare cans of oven-dried soil for each of the buckets, the sieve used to obtain the >250 micron soil samples, and four of the soil samples inside the separatory funnels containing the prepared gastric acid solution.

Figure 2.3: Soil Preparation for PBET

The separatory funnels containing the soil samples in stomach acid were then attached to the ring stands in the temperature-controlled water tank. Figure 2.4 shows the PBET tank with the separatory funnels in place. In the figure the separatory funnels have been attached to the nitrogen lines and are held in the pre-heated water by ring stands. The beaker of water next to the main tank is part of the gravity-fed water return system that cycles the water between the tanks to maintain constant temperature.

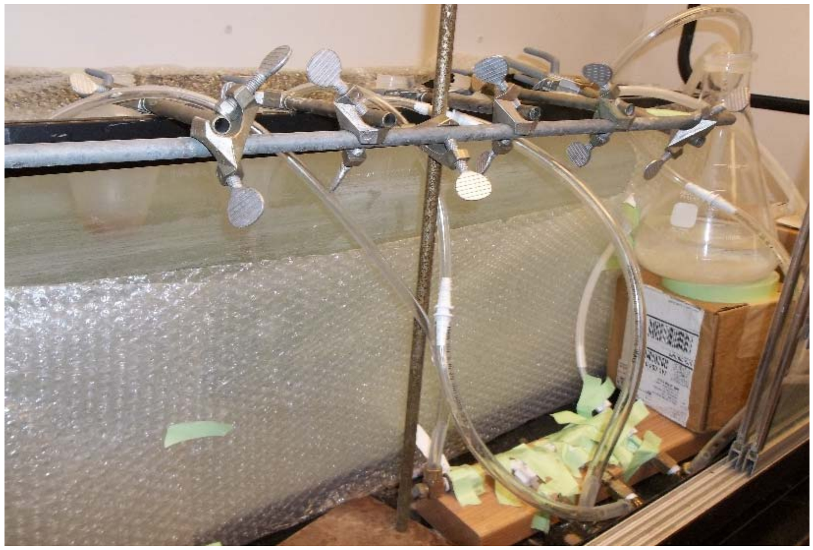

Figure 2.4: PBET Setup – View of PBET Tank with Separatory Funnels

The sample mixture was allowed to stand for 10 min, after which nitrogen gas was purged through the reaction vessel at 1 L/min to provide mixing. The pH was checked after 5 min, 10 min, and then every 15 min thereafter, and the pH was adjusted back to 1.8 with HCl and/or DI water as necessary. After one hour, the typical length of the stomach phase of digestion, the gas was turned off and the separatory funnels were disconnected from the system. The intestinal phase has been determined to be a low source of bioavailable lead due to higher pH, and thus not necessary for this experiment. The samples were collected in lidded glass containers and the liquid fraction removed using a vacuum filtration for analysis by FAA. Every time samples were run through the FAA, a calibration sample set was also run. The calibration samples consisted of 5, 10, 20, 50, and 100 ppm lead made by dilution from a lead standard solution. The gastric solution blank, 10% HCl, and the 10 ppm standard were run periodically to ensure consistent calibration, and 10% HCl was run between each sample in order to ensure the lines were clean. Three values were recorded for each sample run through the FAA, a high, low, and a middle value that represented the approximate average value of the readings given. A calibration curve was graphed based on the calibration standards, and this graph was then used to determine the lead concentrations based off the absorbance values given by the FAA (with a new calibration curve used for each sampling event). The data and accompanying calibration curves can be found in Appendix B. After each PBET analysis, all equipment that had come in contact with soil or gastric solution were washed in an acid bath (of 10% HCl at room temperature) for at least an hour (and allowed to sit in the bath overnight, if there was adequate time between PBET tests). The washed equipment was then rinsed thoroughly with distilled water, and allowed to air-dry.

2.3.3. Effects of Storage. A study was conducted on the impact of storage of post-PBET samples on sample quality/consistency. Four samples from a run of PBET sampling were left in in glass containers with plastic screw-on caps inside a fume hood during the trial period. It was found that two days of storage did not have a statistically significant impact on the lead concentration or appearance of the post-PBET sample, however after two days a mold-like substance, or what could have been a precipitate, began appearing within the containers, suggesting the samples would no longer produce reliable results. Data from the storage impact experiment can be found in Appendix A.

2.4. pH CONTROL TESTS

After a month of testing no statistically significant change in PBET lead concentration had occurred. A titration experiment was run to determine if pH was affecting the results. 0.5 mL of PA was added to a glass beaker of 500 mL of solution containing 100 ppm Pb and 5% HCl. The beaker was then put on a stir plate and continuously stirred and the pH monitored with the pH probe as the solution was gradually neutralized using 5 M NaOH. Samples were periodically collected, and later run through the FAA to determine dissolved lead concentration. A replicate titration experiment was run under similar conditions, but with a neutral starting pH and the addition of 10% HCl to bring the pH below 2. The data can be found in Appendix C.

3. RESULTS AND DISCUSSION

3.1. SOIL CHARACTERIZATION

Each bucket of soil was analyzed by FAA to determine an initial soil lead and calcium concentration. Table 3.1 presents the initial concentrations for the lead and calcium, which were used to calculate the lime and lye additions necessary for each sample bucket. The sample names indicate the bucket (B#), the intended amendment (PA/Lime/Lye), the day the amendment will be added (day#, ex: D20), and the component being analyzed for (Pb or Ca).

Table 3.1: Initial Pb and Ca Soil Concentrations from FAA Analysis

Sample	FAA Absorption	Std. Dev	Concentration of Pb or Ca in sample (mg/L)	Std. Dev	Concentration of Pb or Ca in soil (g/kg)	Std. Dev
B1None_Pb	0.024	0.002	10.1	1.26	1.82	0.227
B2PA_Pb	0.033	0.003	14.7	0.76	2.50	0.129
B3LimeD0_Pb	0.037	0.002	16.8	1.47	2.56	0.224
B4LimeD5_Pb	0.032	0.002	14.3	1.26	2.65	0.233
B5LimeD20_Pb	0.029	0.001	12.6	1.90	2.64	0.398
B6LyeD0_Pb	0.036	0.002	16.6	1.47	2.54	0.224
B7LyeD5_Pb	0.034	0.002	15.6	1.24	2.76	0.220
B8LyeD20_Pb	0.029	0.001	12.9	1.90	2.81	0.412
B1None_Ca	0.542	0.006	40.1	2.36	1.00	0.059
B2PA_Ca	0.730	0.004	55.0	2.46	1.37	0.061
B3LimeD0_Ca	0.757	0.052	57.1	1.29	1.43	0.032
B4LimeD5_Ca	0.762	0.054	57.5	1.44	1.44	0.036
B5LimeD20_Ca	0.731	0.039	55.1	0.27	1.38	0.006
B6LyeD0_Ca	0.724	0.059	54.5	1.84	1.36	0.046
B7LyeD5_Ca	0.752	0.006	56.7	2.34	1.42	0.058
B8LyeD20_Ca	0.668	0.008	50.0	2.16	1.25	0.054
Average/Overall_Pb	0.032	0.002	14.2	1.41	2.54	0.258
Average/Overall_Ca	0.708	0.028	53.3	1.77	1.33	0.044

The soil had an average starting lead concentration of approximately 2.54 ± 0.258 g/kg. The average calcium concentration of the soil was 1.33 ± 0.0443 g/kg, which is typical for soils with high calcium content, such as the Bonne Terre Missouri area, with reported pH range over 6.1 (Nathan et al 2007) due to the presence of dolomite and limestone. These starting concentrations were used to calculate the stoichiometric lime and lye additions. Table 3.2 shows the amendment addition amounts for each bucket of soil, along with a brief description of what amendments were added to each bucket and at what time the amendments were added. Initial calcium data, lead data, and calculations can be found in Table 3.2, and with greater detail in Appendix A.

Table 3.2: Amendment Additions

	PA needed (mL)	Amendment needed (lime or lye) (g)	Description
B1None	0.0	0.0	Control soil
B2PA	33.4	0.0	Control soil + PA
B3LimeD0	63.2	51.7	PA + immediate lime
B4LimeD5	61.0	49.9	PA + 5 day lime
B5LimeD20	67.1	54.9	PA + 20 day lime
B6LyeD0	32.8	29.9	PA + immediate lye
B7LyeD5	33.9	30.9	PA + 5 day lye
B8LyeD20	28.2	25.7	PA + 20 day lye

3.2. PBET LEAD EXPERIMENT

Initial PBET experiments showed an average bioaccessible soil lead concentration of 2.5 g/kg, as shown previously in Table 3.1. The data later collected from the experiment was not statistically significant as it fluctuated dramatically and did not follow any noticeable trends. A summary chart of the data is shown in Figure 3.1, on the next page, and all data and corresponding graphs are located in Appendix B. The

Day 0 data was collected at the start of the experiment period (immediately after amendments were added to the soil), after the initial sampling (initial sampling is represented by a "negative" day value and occurred before any amendments were added to the soil). The Day 0 data showed a decrease in lead concentration from the initial samples, however the samples on Day 1 showed an increase in lead concentration for most samples, and this up and down trend continued throughout the rest of the trial period with no observable overall increase or decrease in lead concentration. Some of the buckets were not sampled between days 0 and 4 because at this point the buckets in question had no amendments other than phosphoric acid, due to the schedule of amendment addition (which can be found in Table 3.2). These buckets would thus correspond with B2PA, the control bucket containing only phosphoric acid, until their amendments were scheduled to begin.

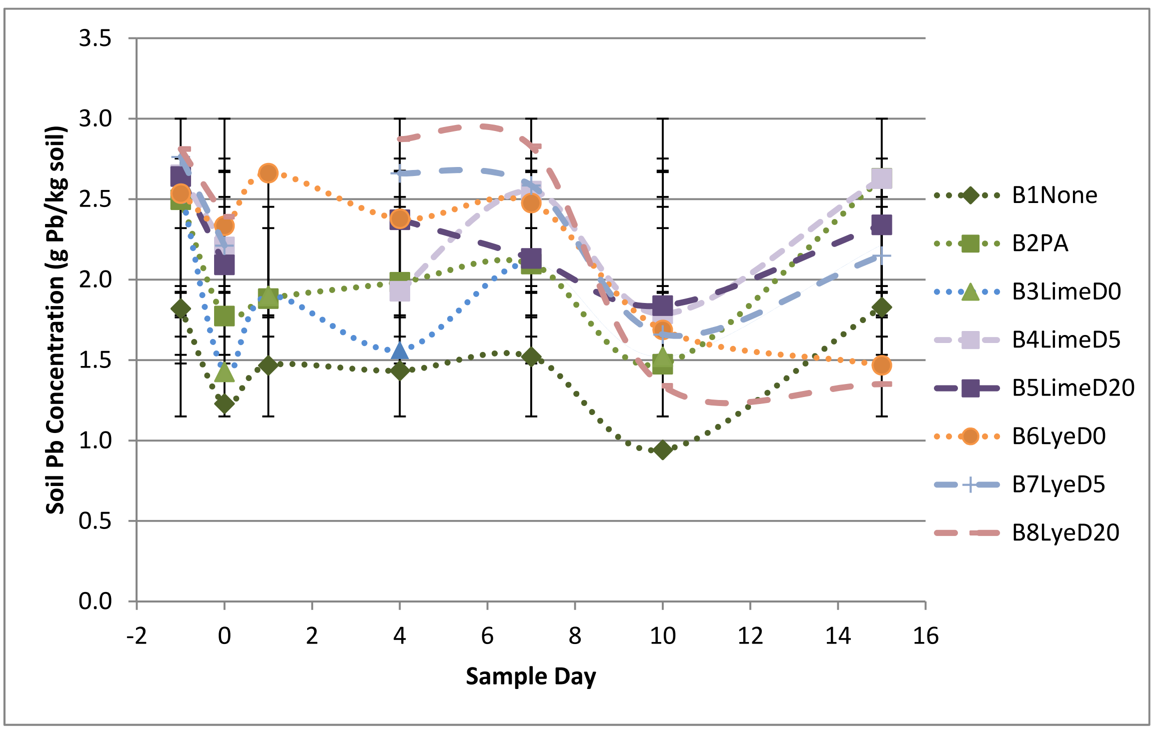

Figure 3.1: Bioaccessible Pb Over Time

The PBET extractions for days 7, 10, and 15 were frozen and later analyzed via graphite furnace atomic absorption spectroscopy (GFAA), which has a lower detection limit than the FAA. The data from the samples run through the GFAA, shown in Figure 3.2, also support the conclusion that the PBET data does not show a statistically significant change in lead concentration over time, and that there seems to be no observable decrease in bioaccessible lead concentration in the soil. All GFAA data and corresponding graphs can be found in Appendix B.

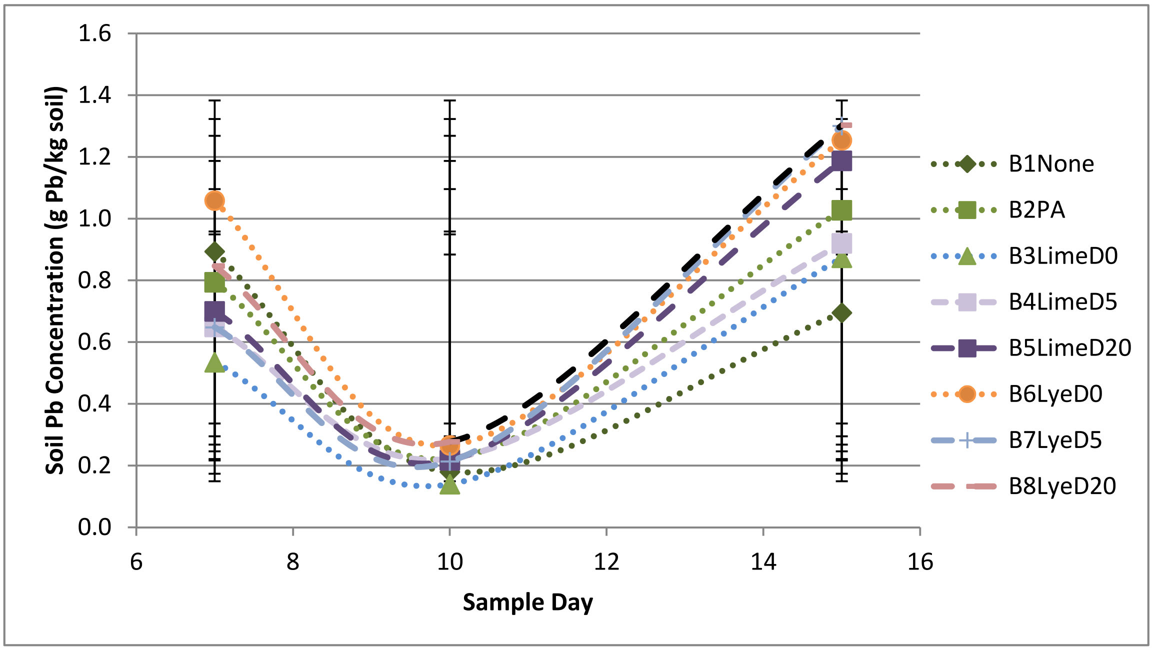

Figure 3.2: Bioaccessible Pb Over Time (Day 7 to Day 15)

Samples from Days 7, 10, and 15 were saved and run through the GFAA for verification of the initial FAA results. Sample day refers to the day the sample was collected. The actual concentration results between the FAA and GFAA analyses differ, most likely due to the effects of storage time; however, the trends in concentration are generally similar and allow for a general comparison.

These results echo the results in Mosby's research (2000), which showed that although the lead had transformed to chloropyromorphite the pig studies showed an increase in bioavailable lead, instead of the expected decrease. This result appears again in Tang et al's research (2009) where field tests indicated a decrease in lead bioavailability but *in-vivo* tests showed a greater amount of bioaccessible lead than expected. In both instances, it is possible that the lead was dissolved by the acidic gastric systems of the test animals, which allowed the animals to absorb more lead than the researchers accounted for, as they assumed the lead would be in a form that could withstand the acidity of a digestive system. One researcher stands out as having evidence that phosphate amendment can withstand the digestive system; Juhasz et al (2005) studied mice and their results supported the hypothesis that phosphate remediation worked in reducing lead bioavailability. This research relied on feeding the mice lead contaminated soil; however, feeding an animal results in an increased pH of the gastric system, which could yield very different results from the impact of the pH of a fasting animal (with a lower gastric pH). The research also focused on the mouse waste, not the concentration of lead present in the animals themselves, which could indicate that soluble lead decreases once the soil reaches the neutral intestinal phase, as theorized by Tang et al (2004).

The vast majority of researchers studying remediation of lead contaminated soils seem to accept the premise that pyromorphite is stable over a wide range of pH's, and indeed prove that phosphate amendment results in pyromorphite formation, without delving into whether that "wide range" includes a human child's digestive system. Those that do focus on a human child's digestive system tend to agree that the pH of the digestive system matters a great deal. Li et al. (2013) observed that the bioaccessibility of soil lead was pH-dependent and that lead became less bioaccessible after the pH drop in the intestinal phase. Wragg and Cave (2003) also agree that the small intestinal phase of the PBET can be ignored for lead bioaccessibility studies, as lead is relatively insoluble at pH values greater than 5.5 and would therefore be excreted with other solid matter.

3.3. pH CONTROL TESTS

The unexpected PBET results caused reexamination of the assumption of the formation of pyromorphite, specifically the question as to whether pyromorphite could form in the soil conditions, and if pyromorphite would be extracted by PBET. A bench-scale study was performed on various combinations of lead, phosphate, lime, and lye to determine why the soil concentration did not seem to be changing significantly. During this bench-scale study the observation was made that while adding lime or lye to a solution of lead nitrate and phosphoric acid did form a precipitate, the precipitate would re-dissolve if the pH was lowered to below 2. The unexpected stability of phosphate and lead in solution at low pH resulted in a shift of focus to the question of pyromorphite solubility.

Titration tests were performed to identify what the correlation was between lead dissolution and pH. A series of two sets of titration tests were performed, from low pH to neutral, then from neutral to low pH. Each test set was performed using a solution of lead nitrate in 1% hydrochloric acid, to which sodium hydroxide was gradually added while on a stir plate with a pH probe monitoring the change after each addition. Once the pH neared a pH of 7, hydrochloric acid was added to the solution until the pH dropped below 2. Figure 3.3 shows the results of the first part of the titration tests, which started with a solution of low pH that was gradually neutralized with the addition of NaOH.

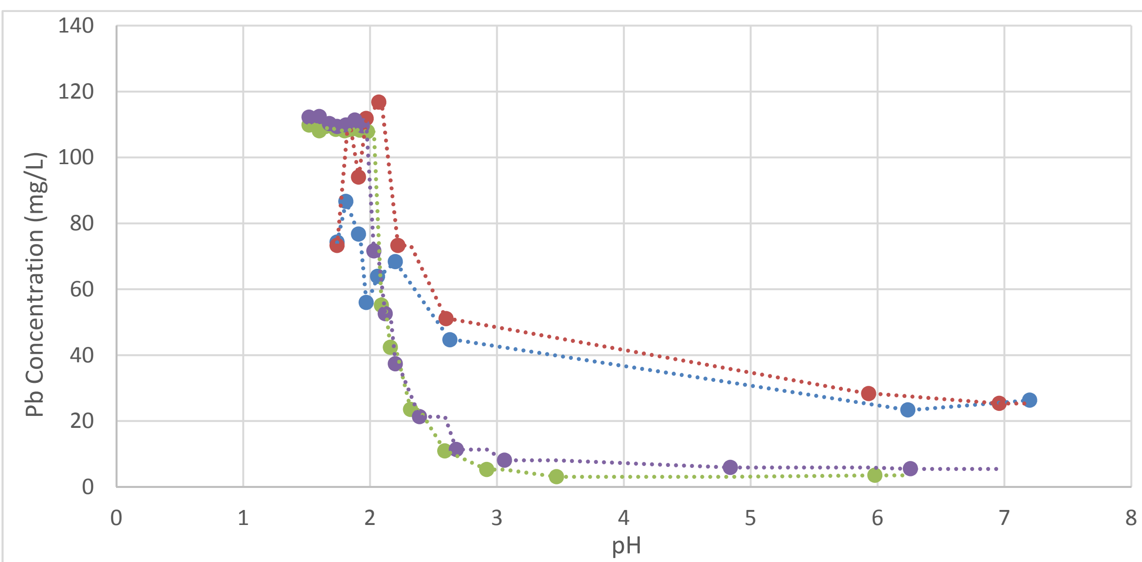

Figure 3.3: Concentration vs. pH: Low Starting pH

The data shows a high lead concentration at the starting pH values (around 1.5-1.7), followed by a decrease in lead concentration of approximately 90 mg/L between pH 2 and pH 3, and finally a relatively constant lead concentration of approximately 5 to 40 mg/L above pH 3. The titration tests showed a drastic change in lead concentration between pH 2 and 3. Each of the four data sets presented are duplicate runs of the same pH titration experiment and were performed in two sets of tests performed on two different days. The data and corresponding graphs for the titration tests can be found in Appendix C.

Figure 3.4 shows the results of the second part of the titration tests, which started with a higher (close to neutral) pH to which hydrochloric acid was added to lower the pH of the solution. The data shows a constant low lead concentration of approximately 5 to 20 mg/L above pH 3, with a rapid increase in lead concentration between pH 3 and 2, followed by a high lead concentration of approximately 60 to 90 mg/L below pH 2. Each of the four data sets presented are duplicate runs of the same pH titration experiment and were performed in two sets of tests performed on two different days.

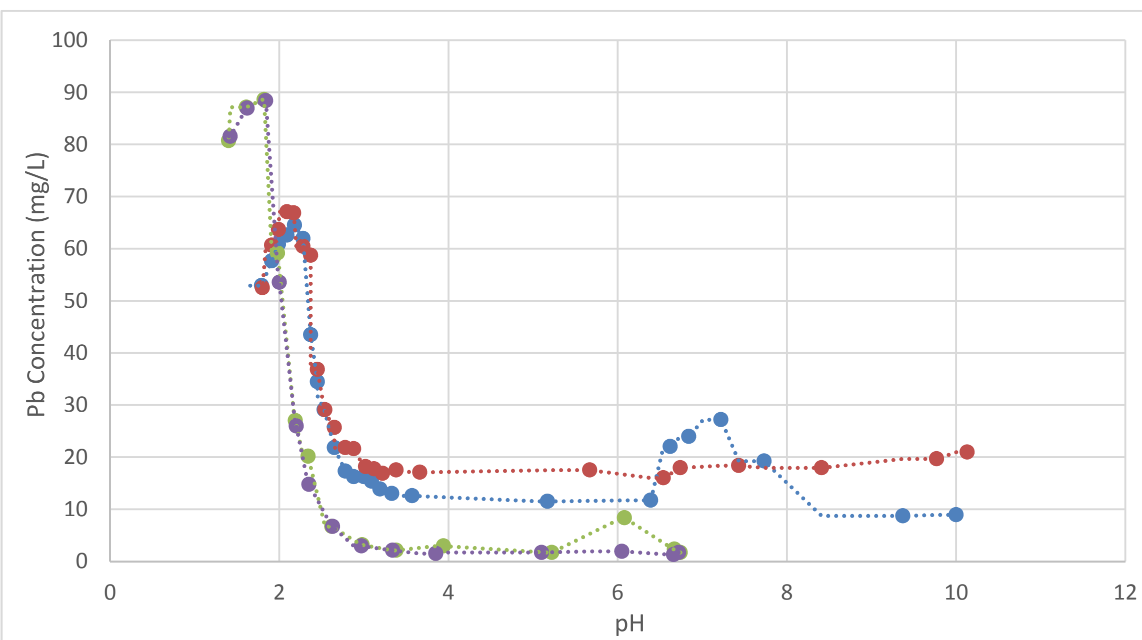

Figure 3.4: Concentration vs. pH: High/Neutral Starting pH

Observations from the titration tests were consistent with the theory that pyromorphite dissolution was occurring at a pH under 3, which would explain the unexpected PBET results. A series of PBET tests were then performed with the gastric solution held at pH 1.8 and pH 3, and extracted solution analyzed using the GFAA. Soil was used from the bucket of soil containing only phosphate amendment and no pH adjustment. The results in Table 3.3 showed a soil lead concentration of 0.24 ± 0.01 g/kg at pH 3, and 1.20 ± 0.02 g/kg at pH 1.8. The data for the PBET tests at pH 1.8 and 3 can be found in Appendix C.

Table 3.3: Soil Sample Comparison: PBET at pH 3 and pH 1.8

Sample	Sample volume (mL)	Soil wt (g)	Sample Conc. (mg/L)	Std. Dev	Soil Conc. (g/kg)	Std. Dev
1 at pH 3	42	0.4	2.12	0.07	0.22	0.01
2 at pH 3	53	0.4	1.56	0.06	0.21	0.01
3 at pH 3	84	0.4	1.17	0.07	0.25	0.01
4 at pH 3	69	0.4	1.64	0.07	0.28	0.01
1 at pH 1.8	60	0.4	7.80	0.13	1.17	0.02
2 at pH 1.8	61	0.4	8.11	0.13	1.24	0.02
3 at pH 1.8	70	0.4	7.09	0.13	1.24	0.02
4 at pH 1.8	59	0.4	7.85	0.14	1.16	0.02

Average, pH 3	0.24	0.01
Average, pH 1.8	1.20	0.02

The results of the initial PBET tests were expected to show a noticeable decrease in Pb concentration, based on the general consensus of past researchers that phosphate remediation of Pb contaminated soil is a relatively fast and effective way to decrease the Pb concentration of the soil. The results from this initial experiment did not support this hypothesis, so the experiment was refocused to the effect of pH on phosphate amendment in an attempt to explain the unexpected failure of phosphate amendment to show a noticeable decrease in Pb contaminated soil. Although most researcher in this field has focused on transforming lead into pyromorphite and stopped there, some researchers, such as Tang et al 2004 and Li et al 2003 took it a step further and when studying PBET systems noticed that there was significantly more bioavailable lead in the acidic stomach phase than in the relatively neutral intestinal phase. Scheckel et al 2005 noted that a PBET run at pH 1.5 resulted in a greater amount of bioavailable lead than the same sample run in a PBET at pH 2.5. The results of the PBET presented in this paper (run at pH 1.8) and the follow-up pH titration tests, supports the observation from the Tang, Li, and Scheckel papers that a PBET run at conditions simulating a fasting human child do not result in a significant reduction of bioavailable lead.

4. POSSIBLE SOURCES OF ERROR

Potential cross-contamination from trowel, sieve, tare can, separatory funnels, or glass sample containers is possible, which could impact the lead concentration of the final sample. To minimize the potential for cross-contamination from these sources, each was put through an acid bath, then rinsed (except for trowels and tare cans, which were washed with soapy water and rinsed before use, then were used for only one bucket and never came in contact with other soils). Cross-contamination could also have occurred while the samples from the PBET were being vacuum extracted. To minimize the effects of cross-contamination, the vacuum extraction equipment was thoroughly rinsed between every sample with distilled water (further cleaning was deemed impractical, as there was insufficient time for extensive acid bath cleaning of the vacuum extraction device between PBET analyses). Cross-contamination could also have occurred during FAA or GFAA sampling. To prevent this, diluted HCl (of 10% HCl) was run through the sampling hose between each sample, until the instrument was reading near zero concentration (due to drift over analysis time, absolute zero was not always achieved). The absorption values given by the FAA tended to drift (gradually increase) over time each time an analysis was performed, so the longer it took to run a sample set through the FAA, the more inaccurate the FAA readings became. This drift could cause some error in calculating the final lead concentration of the samples, which the combination of MDL and standard deviation should account for. The FAA also had to be recalibrated a couple times as the lamp had been taken out of the machine for various reasons over the course of this study, which could have caused some inconsistencies in the lead concentrations given by the machine (although the recalibration each time, and the calibration curves run at the beginning of each FAA analysis should correct this). The GFAA had many sources of error, including the possibility of a dirty or old graphite furnace, unexplained error messages given by the computer, and inconsistent calibration curves. Diluted hydrochloric acid (HCl) or nitric acid (HNO_3) blanks were run through the GFAA multiple times before any samples were run, in order to clean the graphite furnace to minimize the effects of leftover samples from other research groups, and the unexplained error messages seemed to go away if the GFAA and computer were both restarted (although this sometimes had to be done multiple times). To minimize the impact of the inconsistent calibration curves, the calibration samples were run a number of times until a data set with a low relative standard deviation (RSD) was attained. The calibration curves tended to have lower RSD values for concentrations above 100 μg/L (RSD < 2), and most of the data was above 100 μg/L, so this was deemed acceptable. Other sources of error could include human error in measuring, timing, calculations, etc. which could lead to an error in final lead concentration values, in addition to inconsistencies between sample sets. The possibility also exists that chemicals went bad due to age or improper storage, as most chemicals used were found in the lab and were either close to expiration, or had been opened and used before (and thus presented an unknown for potential contamination from improper past use or handling of the chemicals). Only the gastric enzymes were purchased immediately before or during this research. As with any research involving soil, there is also the possibility that the given data does not accurately represent the true average value for each bucket of soil, due to the heterogeneous nature of soil. Another source of error would be the ratio of soil to gastric solution (0.4 g of soil to 40 mL of gastric solution). After the PBET procedure, the soil had been diluted enough that the FAA could no longer reliably read changes in the lead concentration of the samples; for example 1800 mg/kg of lead in soil completely extracted by PBET would result in 14.9 mg/L in solution, which would result in an FAA absorption value of 0.010, which is just below the MDL (determined to be 0.011). The GFAA looks at values in the low ppb range (μg/L), whereas the FAA detected concentrations only in the ppm (mg/L) range, so dilution of the samples was required before going through the GFAA (typically 10x dilution), and the GFAA was used to verify reliability of FAA data. While attempts were made to minimize any and all identified sources of error, there is always the possibility that the lead concentrations could be slightly over or under their actual values due to cross-contamination, machine or human error, or non-homogeneous soil.

5. CONCLUSIONS

The PBET data did not indicate a statistically significant change in lead concentration. A large amount of error could be attributed to the ratio of soil to gastric solution, which resulted in a solution that was diluted enough that the reliability of the FAA to detect the small amount of lead-containing soil was questionable. The high concentrations of lead throughout the experiment made this concern a non-issue, and the GFAA analysis of the same solutions supported the trends of the FAA data. The GFAA was computer-run and calculated its own MDL for each run of samples, flagging any samples which were below the MDL or too high in lead concentration for an accurate reading, and these flagged samples were then run again after the dilution amount was tweaked. The GFAA results further supported the conclusion that any observed change in soil lead concentration was statistically insignificant. After a couple weeks of no observable change in concentration, the author refocused the subject of the research to explain this lack of change in lead concentration. A bench-scale study was performed on various combinations of lead, phosphate, lime, and lye to determine why the soil concentration did not seem to be changing significantly. During this bench-scale study the observation was made that while adding phosphate to lead nitrate formed a precipitate, the precipitate would re-dissolve if the pH was lowered to below 2. A more in-depth titration study was then performed in order to test the hypothesis that lead becomes soluble at low pH. The validation of this hypothesis would then explain why the soil experiment was not yielding concrete results, as the PBET procedure requires the pH to be below 2 to simulate the pH of a resting/empty child’s stomach. The titration tests clearly showed that lead is soluble below a pH of 2 despite the presence of phosphate. To further confirm that the gastric pH in the PBET was causing the lead to become soluble, the PBET was run on four samples at pH 1.8 (average fasting child gastric pH) and at pH 3 (slightly above the pH at which pyromorphite was hypothesized to become soluble). The results of this PBET pH experiment indicated that there was about five times more lead present at pH 1.8 than at pH 3, again proving the theory that gastric pH has a large impact on lead bioaccessibility.

This likely explains the differing results presented in Mosby's thesis paper (2000) and presented earlier in this paper. Mosby's paper found that the soil amendments successfully transformed the lead into pyromorphite; however, during the pig portion of the study they discovered that the bioavailability of lead increased after phosphate amendment. The digestive system of a pig is very similar to that of a human child, and as such the pH's are comparable and the acidic environment of a fasting pig's digestive system would be enough to dissolve lead compounds such as pyromorphite into forms that are bioavailable to the pig.

Of the articles that look at pyromorphite formation over various pH ranges, few look at a pH less than 2 (Scheckel et al. 2005; Li et al. 2013; Zhang and Ryan 1998; Zhang and Ryan 1999; Scheckel and Ryan 2002). The lack of information on pyromorphite solubility below pH 2 suggests that previous research in the field has been based on either the assumption that pyromorphite will remain stable below pH 2, or that a fasting child's gastric system will not drop below pH 2. The research contained in this paper strongly suggests that phosphate amendment is not as effective as previously believed for lead soil remediation. Even if the phosphate amendment successfully forms pyromorphite, it will just become soluble (and bioaccessible) once ingested if the child has a gastric pH below 2 (a pH of 1.8 is the average gastric pH for a fasting child, as discussed previously). More in-depth research on the effects of low pH solubility on lead compounds, and the effect of stomach acids on pyromorphite and other lead compounds, are recommended. Continuing and expanding the titration tests to look at dissolution and precipitation of various lead compounds over pH ranges found in a human body could also be beneficial and lead to a more detailed understanding of the reduction of bioaccessible lead in soils.

APPENDIX A.

SOIL CHARACTERIZATION DATA

Table A1.1: Soil pH Test

	DI H2O	$CaCl_2$ soln	Average pH
B1None	7.44	7.33	7.39
B2PA	7.48	7.34	7.41
B3LimeD0	7.44	7.34	7.39
B4LimeD5	7.25	7.34	7.30
B5LimeD20	7.47	7.33	7.40
B6LyeD0	7.46	7.34	7.40
B7LyeD5	7.47	7.34	7.41
B8LyeD20	7.50	7.35	7.43
Average Soil pH			7.39

Soil pH was determined using the average of a soil slurry in both DI water and a $CaCl_2$ solution to account for seasonal variations, as shown in Table A1.1.

Table A1.2: Soil Weight Test

	soil + bucket[1] wt (lbs)	"wet soil"[2] wt (lbs)	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Wet soil (g)	Dry soil (g)	Water % by mass	wt. for bucket of dry soil (lbs)
B1None	44.00	42.00	21.06	53.49	51.41	32.43	30.35	6.85	39.12
B2PA	42.75	40.75	20.93	61.22	59.52	40.29	38.59	4.41	38.95
B3LimeD0	42.88	40.88	20.90	55.43	54.03	34.53	33.13	4.23	39.15
B4LimeD5	41.75	39.75	20.86	58.97	56.72	38.11	35.86	6.27	37.26
B5LimeD20	46.38	44.38	21.02	66.07	64.16	45.05	43.14	4.43	42.41
B6LyeD0	42.00	40.00	20.99	66.22	64.41	45.23	43.42	4.17	38.33
B7LyeD5	41.50	39.50	20.96	63.92	61.99	42.96	41.03	4.70	37.64
B8LyeD20	38.75	36.75	20.75	57.35	54.84	36.60	34.09	7.36	34.04

[1]Empty buckets weighed approximately 2 lbs

[2]"wet soil" weight is soil mass after subtracting the bucket weight

Note: Scale had ¼ mark increments (the two measurements that are not in ¼ increments were almost entirely between the marks, and recorded accordingly)

Table A1.2 is a record of the soil weight found for each bucket so that these values could later be used in the overall lead, calcium, and the amendment addition calculations.

Table A1.3: Phosphoric Acid Addition Stoichiometrically Required for Pb and Ca Concentrations in the Soil

	dry soil wt. (lbs)	Pb conc. (ppm)	Pb in soil (lb)	Pb in soil (mol)	Pb/P ratio	Ca conc. (ppm)	Ca in soil (lb)	Ca in soil (mol)	Ca/P ratio	PA needed (mol)	PA needed without Ca fix (g)	PA fix	PA needed (g)
B1None	39.12	1820.66	0.07	0.16	1.67	1003.00	0.04	0.44	1.67	0.36	35.34	0.85	41.58
B2PA	38.95	2497.22	0.10	0.21	1.67	1374.28	0.05	0.61	1.67	0.49	48.23	0.85	56.74
B3LimeD0	39.15	2562.14	0.10	0.22	1.67	1428.36	0.06	0.63	1.67	0.51	50.21	0.85	59.07
B4LimeD5	37.26	2652.46	0.10	0.22	1.67	1438.25	0.05	0.61	1.67	0.49	48.46	0.85	57.01
B5LimeD20	42.41	2641.40	0.11	0.25	1.67	1376.26	0.06	0.66	1.67	0.54	53.35	0.85	62.77
B6LyeD0	38.33	2535.30	0.10	0.21	1.67	1361.75	0.05	0.59	1.67	0.48	47.33	0.85	55.68
B7LyeD5	37.64	2763.54	0.10	0.23	1.67	1417.15	0.05	0.60	1.67	0.50	48.97	0.85	57.61
B8LyeD20	34.04	2812.28	0.10	0.21	1.67	1250.96	0.04	0.48	1.67	0.42	40.73	0.85	47.92

Note: Phosphoric acid is 85% by weight, which is corrected for in the PA fix column

PA Information

molar mass	98
mol/L	14.7
g solute/L	1445
% by mass	85
g/mL	1.7

Pb molar mass =	207	g/mol
Ca molar mass =	40	g/mol
P molar mass =	31	g/mol
Ca(OH)2 (lime) =	74	g/mol
NaOH (lye) =	40	g/mol
H3PO4 (PA) =	98	g/mol

Note: double-checked by weighing 5mL of PA (measured 8.545g PA)

The A1.3 table set shows the calculations for the phosphoric acid required to react with the Pb and calcium in the soil, along with all values used in these calculations.

Table A1.4: Lime Addition and Calcium Increase Calculation

	PA for soil (mol)	OH- for soil (mol)	Total PA needed (mol)	Lime needed (mol)	Ca/P ratio	PA for Ca in lime (mol)	Lime for added PA (mol)	PO4 almost total (mol)	delta	Final Total PA (mol)	Total PA (g)	PA fix (by mass)	Final Total PA (g)	PA needed (mL)	Final Total lime (mol)	Final Total lime (g)
B1None	0.36	0.54	0.66	0.49	1.67	0.30	0.22	0.66	0.00	0.66	64.26	0.85	75.60	44.47	0.49	36.39
B2PA	0.49	0.74	0.89	0.67	1.67	0.40	0.30	0.89	0.00	0.89	87.69	0.85	103.17	60.69	0.67	49.66
B3LimeD0	0.51	0.77	0.93	0.70	1.67	0.42	0.31	0.93	0.00	0.93	91.29	0.85	107.40	63.17	0.70	51.70
B4LimeD5	0.49	0.74	0.90	0.67	1.67	0.40	0.30	0.90	0.00	0.90	88.11	0.85	103.66	60.98	0.67	49.90
B5LimeD20	0.54	0.82	0.99	0.74	1.67	0.45	0.33	0.99	0.00	0.99	97.00	0.85	114.12	67.13	0.74	54.94
B6LyeD0	0.48	0.72	0.88	0.66	1.67	0.40	0.30	0.88	0.00	0.88	86.05	0.85	101.24	59.55	0.66	48.73
B7LyeD5	0.50	0.75	0.91	0.68	1.67	0.41	0.31	0.91	0.00	0.91	89.04	0.85	104.75	61.62	0.68	50.43
B8LyeD20	0.42	0.62	0.76	0.57	1.67	0.34	0.26	0.76	0.00	0.76	74.06	0.85	87.13	51.25	0.57	41.94

*random value initially, solver changes this to true value

*make this 0 in solver

Enough phosphoric acid must be added to react with the lead and calcium in the soil, and using lime as a pH neutralizer requires extra phosphoric acid addition to counteract the calcium in the lime. Table A1.4 shows the calculations involved in obtaining a final phosphoric acid and lime addition value.

Table A1.5: Lye Addition

	PA needed (mol)	OH- needed (mol)	Lye needed (mol)	Lye needed (g)	Lye correction	Lye needed (g)	PA needed (g)	PA needed (mL)
B1None	0.361	0.541	0.541	21.637	0.97	22.30666329	41.578	24.458
B2PA	0.492	0.738	0.738	29.529	0.97	30.44185483	56.741	33.377
B3LimeD0	0.512	0.768	0.768	30.740	0.97	31.69020891	59.068	34.746
B4LimeD5	0.494	0.742	0.742	29.670	0.97	30.58755388	57.013	33.537
B5LimeD20	0.544	0.817	0.817	32.665	0.97	33.67480259	62.767	36.922
B6LyeD0	0.483	0.724	0.724	28.976	0.97	29.87216707	55.679	32.753
B7LyeD5	0.500	0.750	0.750	29.983	0.97	30.91024973	57.614	33.891
B8LyeD20	0.416	0.623	0.623	24.938	0.97	25.70970223	47.921	28.189

Note: PA needed (g and mL) are from calculations in PA addition table

Table A1.5 shows the lye addition required to neutralize the pH change caused by the phosphoric acid soil addition.

Table A1.6: Overall Amendment Chart

	PA needed (mL)	Amendment needed (g)	Description
B1None	0.000	0.000	Control soil
B2PA	33.377	0.000	Control soil + PA
B3LimeD0	63.174	51.698	PA + immediate lime
B4LimeD5	60.976	49.899	PA + 5 day lime
B5LimeD20	67.131	54.936	PA + 20 day lime
B6LyeD0	32.753	29.872	PA + immediate lye
B7LyeD5	33.891	30.910	PA + 5 day lye
B8LyeD20	28.189	25.710	PA + 20 day lye

Table A1.6 is a tabulated form of what amendment is added to which bucket on which day.

Table A1.7: Water Addition Calculations

rainfall yearly average	46.6	in/yr
bucket diameter	11.5	in
soil surface area	103.9	in^2
volume of rainfall	4843.4	in^3/yr per bucket
1 L =	61.0	in^3
volume of rainfall	79.4	L/yr per bucket
daily water addition	0.2	L per bucket
if watering every 3 days	0.7	L per bucket
if watering every 3 days	652.3	mL per bucket

Table A1.7 shows the calculations for the simulated rainfall water addition.

The following tables (A1.8 through A1.15) are all soil moisture tests to ensure the soil was brought to an acceptable soil moisture content before amendments were added, and that once amendments were added and testing begun the soil moisture stayed within an acceptable range throughout the study.

Table A1.8: Soil Moisture Test 11/5/14

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	21.04	60.19	59.77	1.09
B2PA	20.92	77.61	75.87	3.16
B3LimeD0	20.88	66.33	65.16	2.62
B4LimeD5	20.83	69.51	67.91	3.38
B5LimeD20	21.00	67.81	66.14	3.71
B6LyeD0	20.96	71.91	70.39	3.06
B7LyeD5	20.93	66.27	64.80	3.36
B8LyeD20	20.73	68.91	67.24	3.59

Soil has been sitting in the lab with little to no water addition for a while, so the low water % is to be expected.

Table A1.9: Soil Moisture Test 2/15/15

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	21.053	46.000	44.141	8.052
B2PA	20.917	49.668	46.877	10.751
B3LimeD0	20.891	47.352	45.778	6.325
B4LimeD5	20.843	48.722	47.186	5.831
B5LimeD20	21.010	48.184	46.676	5.875
B6LyeD0	20.970	49.476	47.866	5.986
B7LyeD5	20.943	51.490	49.154	8.280
B8LyeD20	20.739	49.550	47.865	6.212

Soil has been watered every 2 or 3 days with 450 mL to each bucket

Table A1.10: Soil Moisture Test 2/25/15

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	21.048	43.209	38.393	27.766
B2PA	20.916	46.425	41.114	26.295
B3LimeD0	20.896	49.828	44.158	24.375
B4LimeD5	20.864	47.044	41.645	25.980
B5LimeD20	21.014	50.120	43.972	26.779
B6LyeD0	20.975	50.212	44.187	25.956
B7LyeD5	20.949	47.766	42.335	25.395
B8LyeD20	20.745	52.176	45.792	25.488

Soil has been watered every 2 or 3 days with 450 mL to each bucket

Table A1.11: Soil Moisture Test (Day 0 Samples)

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	21.217	63.424	54.532	26.691
B2PA	21.047	61.052	52.937	25.447
B3LimeD0	20.854	54.991	48.038	25.578
B4LimeD5	21.144	56.598	49.189	26.418
B5LimeD20	20.996	59.740	51.417	27.359
B6LyeD0	21.029	64.243	55.333	25.974
B7LyeD5	21.012	62.989	54.080	26.941
B8LyeD20	20.833	71.383	60.449	27.600

Soil has been watered every 3 days with 655 mL to each bucket.
Soil dried in oven for ~16.5hrs

Table A1.12: Soil Moisture Test (Day 1 Samples)

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	21.122	57.763	55.089	7.872
B2PA	20.902	62.668	59.637	7.825
B3LimeD0	20.889	63.390	61.333	5.086
B4LimeD5	20.871	62.956	60.425	6.399
B5LimeD20	21.003	67.226	65.181	4.629
B6LyeD0	20.958	65.202	63.582	3.801
B7LyeD5	20.937	63.962	62.106	4.508
B8LyeD20	20.727	60.523	58.381	5.689

Soil has been watered every 3 days with 655 mL to each bucket.
Soil dried in oven for ~23hrs

It seems highly unlikely that the soil moisture could have decreased so drastically over the course of a day, and as this is the only set of data that is so low this data set was disregarded as an outlier and attributed to some unknown human or instrument error.

Table A1.13: Soil Moisture Test (Day 4 Samples)

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	13.914	58.200	48.850	26.763
B2PA	13.939	59.800	49.894	27.551
B3LimeD0	13.795	57.100	48.186	25.920
B4LimeD5	13.958	53.200	44.971	26.534
B5LimeD20	13.777	55.100	46.651	25.701
B6LyeD0	13.806	55.700	46.776	27.067
B7LyeD5	13.809	55.400	46.778	26.152
B8LyeD20	13.650	54.500	45.836	26.919

Soil has been watered every 3 days with 655 mL to each bucket.
Soil put in oven at 6:00pm on 3/3, taken out approximately 36hrs later.

Table A1.14: Soil Moisture Test (Day 7 Samples)

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	14.060	62.20	51.522	28.504
B2PA	14.050	63.73	52.504	29.193
B3LimeD0	13.900	57.94	48.407	27.626
B4LimeD5	14.060	55.63	46.149	29.546
B5LimeD20	13.900	59.51	49.622	27.680
B6LyeD0	13.930	58.20	47.934	30.191
B7LyeD5	13.910	62.67	50.647	32.727
B8LyeD20	13.760	62.57	51.752	28.474

Soil has been watered every 3 days with 655 mL to each bucket. Soil put in oven at 6:00pm on 3/6, taken out on 3/10 at 5am, approximately 83 hrs later.

Table A1.15: Soil Moisture Test (Day 10 Samples)

	Tare Can (g)	Wet soil + tare can (g)	Dry soil + tare can (g)	Water % by mass (g)
B1None	13.751	65.13	54.015	27.605
B2PA	13.768	64.96	53.585	28.568
B3LimeD0	13.883	61.30	51.270	26.828
B4LimeD5	13.803	61.10	50.363	29.368
B5LimeD20	13.967	60.64	50.491	27.787
B6LyeD0	13.910	68.63	56.266	29.191
B7LyeD5	13.755	66.55	53.757	31.981
B8LyeD20	13.672	71.19	58.587	28.060

Soil has been watered every 3 days with 655 mL to each bucket. Soil put in oven at 7:20pm on 3/9, taken out on 3/12 at 4:30am, approximately 57 hrs later.

Table A1.16: Initial Pb Samples Data (Before Amendments Added)

Sample	FAA Absorbance			Average	Std. Dev
	Trial 1	Trial 2	Trial 3		
HCl	0.000	0.000	0.000	0.000	0.000
1ppm	0.004	0.004	0.004	0.004	0.000
2ppm	0.008	0.008	0.008	0.008	0.000
3ppm	0.014	0.013	0.013	0.013	0.000
4ppm	0.019	0.019	0.019	0.019	0.000
5ppm	0.023	0.023	0.023	0.023	0.000
10ppm	0.040	0.040	0.040	0.040	0.000
20ppm	0.075	0.075	0.075	0.075	0.000
50ppm	0.196	0.195	0.196	0.196	0.000
100ppm	0.374	0.373	0.372	0.373	0.001
HCl	0.006	0.006	0.006	0.006	0.000
10ppm	0.043	0.043	0.043	0.043	0.000
HCl	0.006	0.006	0.006	0.006	0.000
Soil 1	0.056	0.056	0.056	0.056	0.000
Soil 2	0.083	0.083	0.084	0.083	0.000
Soil 3	0.079	0.080	0.079	0.079	0.000
Soil 4	0.095	0.095	0.096	0.095	0.000
HCl	0.011	0.011	0.011	0.011	0.000
10ppm	0.048	0.048	0.048	0.048	0.000
HCl	0.011	0.011	0.011	0.011	0.000
Soil 5	0.080	0.080	0.080	0.080	0.000
Soil 6	0.077	0.076	0.077	0.077	0.000
Soil 7	0.089	0.089	0.089	0.089	0.000
Soil 8	0.080	0.081	0.080	0.080	0.000
Soil Mix	0.080	0.080	0.080	0.080	0.000
HCl	0.011	0.011	0.011	0.011	0.000
10ppm	0.048	0.048	0.048	0.048	0.000

Note: Concentration of sample was calculated by multiplying the absorption value and the starting solution soil concentration (50 mL solution/0.5 g soil)

The data shown in table A1.16.1 gives the FAA absorbance values (three values were recorded for each sample), along with a calculated average and standard deviation value.

Table A1.16.2

Sample	Absorption	Conc of sample (mg/L)	Conc of soil (mg/kg)
Soil 1	0.056	14.3	1434.0
Soil 2	0.083	21.7	2166.7
Soil 3	0.079	20.6	2059.5
Soil 4	0.095	24.9	2488.3
Soil 5	0.080	20.8	2077.3
Soil 6	0.077	19.9	1988.0
Soil 7	0.089	23.2	2318.5
Soil 8	0.080	20.9	2086.3
Soil Mix	0.080	20.8	2077.3

Table A1.16.3

	Sample Conc (mg/L)	Soil Conc (mg/kg)
Soil Mix	20.7732	2077.32
Soil Mix - Average	20.7732	2077.32

Tables A1.16.2 and A1.16.3 show the sample concentration and soil concentration, respectively, that were calculated from the FAA absorbance data. A soil sample from each bucket was assayed, along with an additional sample composed of an equal mix of soil from each of the buckets.

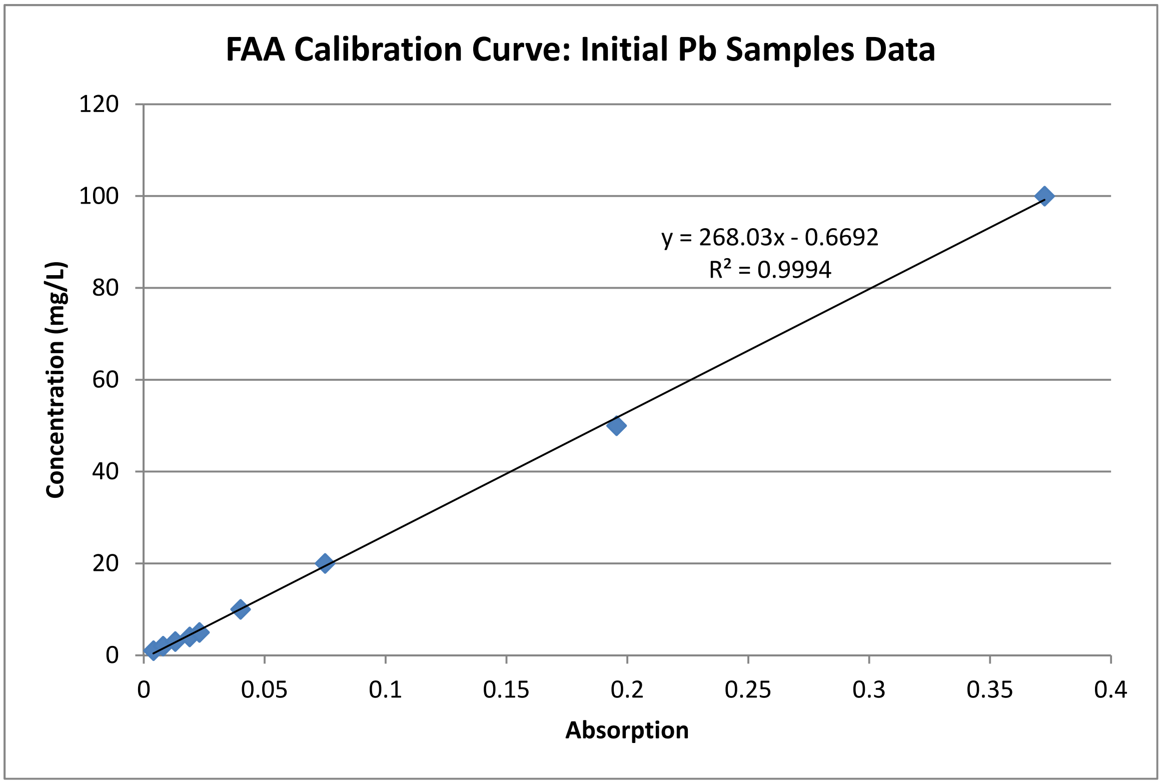

Figure A1.1: FAA Calibration Curve: Initial Pb Samples Data

Figure A1.1 shows the calibration curve for the FAA calibration data in table A1.16.1 on the previous page.

MDL Graph: Calibration Curve	
Absorption	Concentration (mg/L)
0.004	1
0.008	2
0.013	3
0.019	4
0.023	5

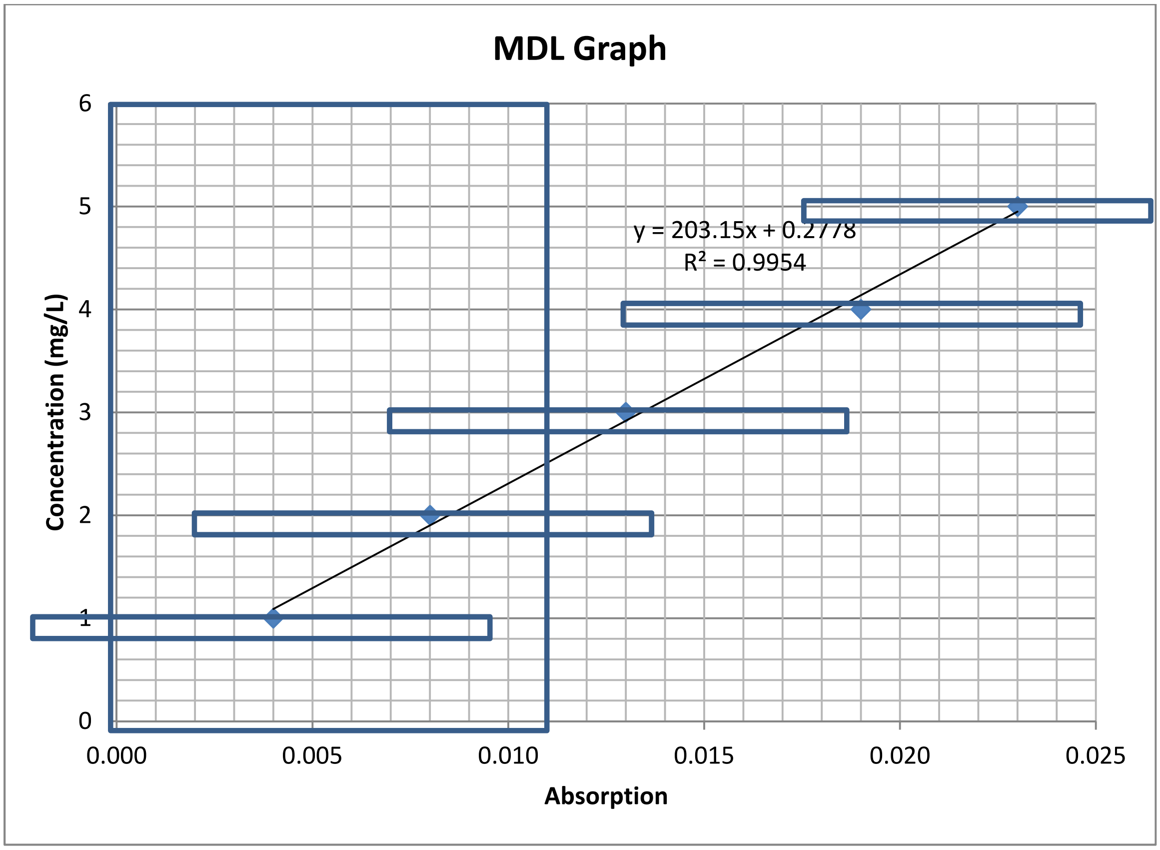

Figure A1.2: MDL Graph

Figure A1.2 is the low end of the calibration curve from the calibration samples used in the initial lead content test (see Table A1.16 for the complete table of absorbance values). The large box covering the absorption range of 0.000 to 0.011 was determined to be the MDL based on the largest recorded value for the control (HCl, assumed to be 0 mg/L lead). The horizontal boxes indicate an error range based on the MDL. From this data we can see that any concentration below 4 mg/L cannot be assumed to be detected by the FAA as a non-zero value.

Table A1.17: Storage Impact: Initial Values (Day 0)

FAA values

Sample	Absorbance			Average	Std. Dev
	Trial 1	Trial 2	Trial 3		
Gastric	0.000	0.000	0.000	0.000	0.000
5ppm	0.004	0.005	0.010	0.006	0.003
10ppm	0.009	0.020	0.016	0.015	0.005
20ppm	0.031	0.038	0.032	0.034	0.003
50ppm	0.085	0.093	0.094	0.091	0.004
100ppm	0.174	0.185	0.177	0.179	0.005
Gastric	0.000	0.000	0.000	0.000	0.000
10ppm	0.016	0.020	0.018	0.018	0.002
Gastric	0.000	0.000	0.000	0.000	0.000
Sample 1	0.024	0.018	0.021	0.021	0.002
Sample 2	0.007	0.014	0.017	0.013	0.004
Sample 3	0.013	0.018	0.015	0.015	0.002
Sample 4	0.017	0.021	0.018	0.019	0.002
Gastric	0.000	0.005	0.000	0.002	0.002
10ppm	0.018	0.023	0.017	0.019	0.003

Table A1.17 shows the initial FAA values obtained for a set of samples after being run through the PBET. The samples were then allowed so sit in closed containers under a fume hood as changes in concentration and appearance were observed.

Calibration Curve	
Absorption	Conc (mg/L)
0.006	5
0.015	10
0.034	20
0.091	50
0.179	100

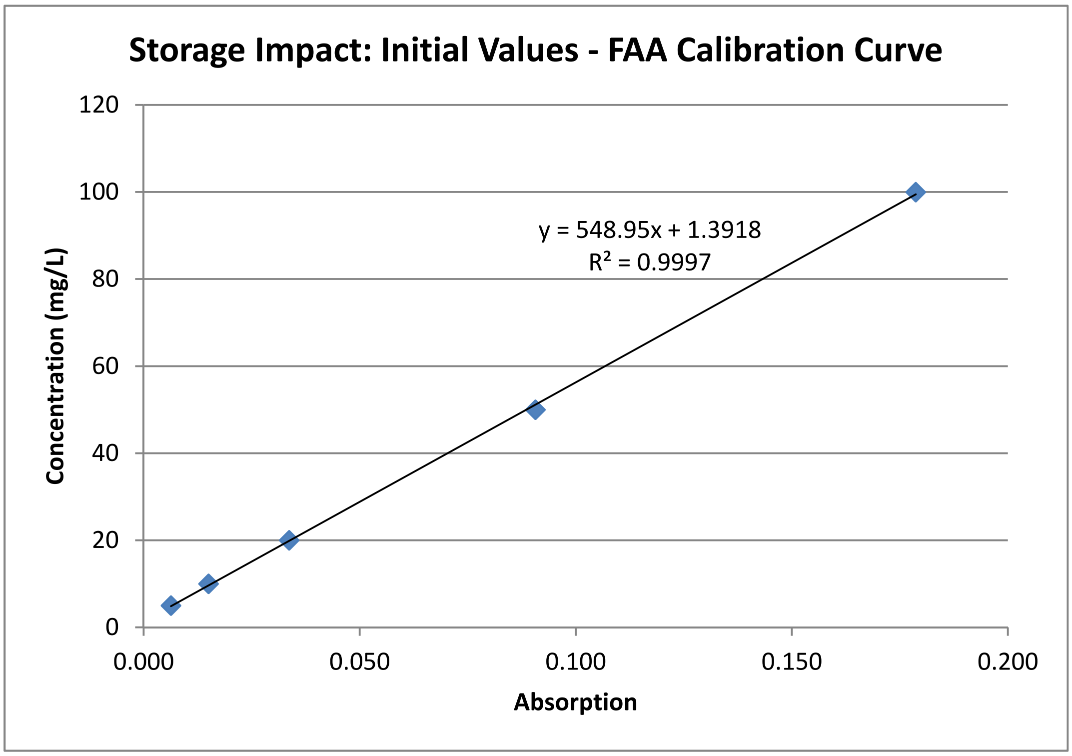

Figure A1.3: Storage Impact: Initial Values – FAA Calibration Curve

Figure A1.3 is the calibration curve obtained from the calibration data given in Table A1.17 (and copied/simplified onto this page, above the figure).

Table A1.18: Storage Impact (Day 2)

Storage Impact: Day 2 (approximately 42 hours later)

Sample	Absorbance			Average	Std. Dev
	Trial 1	Trial 2	Trial 3		
Gastric	0	-0.011	-0.007	-0.006	0.005
5ppm	0.004	0.006	0.001	0.004	0.002
10ppm	0.012	0.007	0.019	0.013	0.005
20ppm	0.026	0.031	0.033	0.030	0.003
50ppm	0.089	0.091	0.085	0.088	0.002
100ppm	0.17	0.178	0.0174	0.122	0.074
Gastric	-0.007	-0.004	-0.002	-0.004	0.002
10ppm	0.016	0.02	0.018	0.018	0.002
Gastric	0	-0.006	-0.001	-0.002	0.003
Sample 1	0.018	0.021	0.02	0.020	0.001
Sample 2	0.014	0.009	0.013	0.012	0.002
Sample 3	0.015	0.012	0.018	0.015	0.002
Sample 4	0.014	0.016	0.019	0.016	0.002
Gastric	-0.002	0	-0.002	-0.001	0.001
10ppm	0.02	0.017	0.025	0.021	0.003

Table A1.18 shows the FAA values for the same samples as those in Table 1.17 after they have been left closed in a fume hood for approximately 2 days. At this point there has been no observable change in solution appearance.

Calibration Curve	
Absorption	Concentration (mg/L)
0.004	5
0.013	10
0.030	20
0.088	50
0.122	100

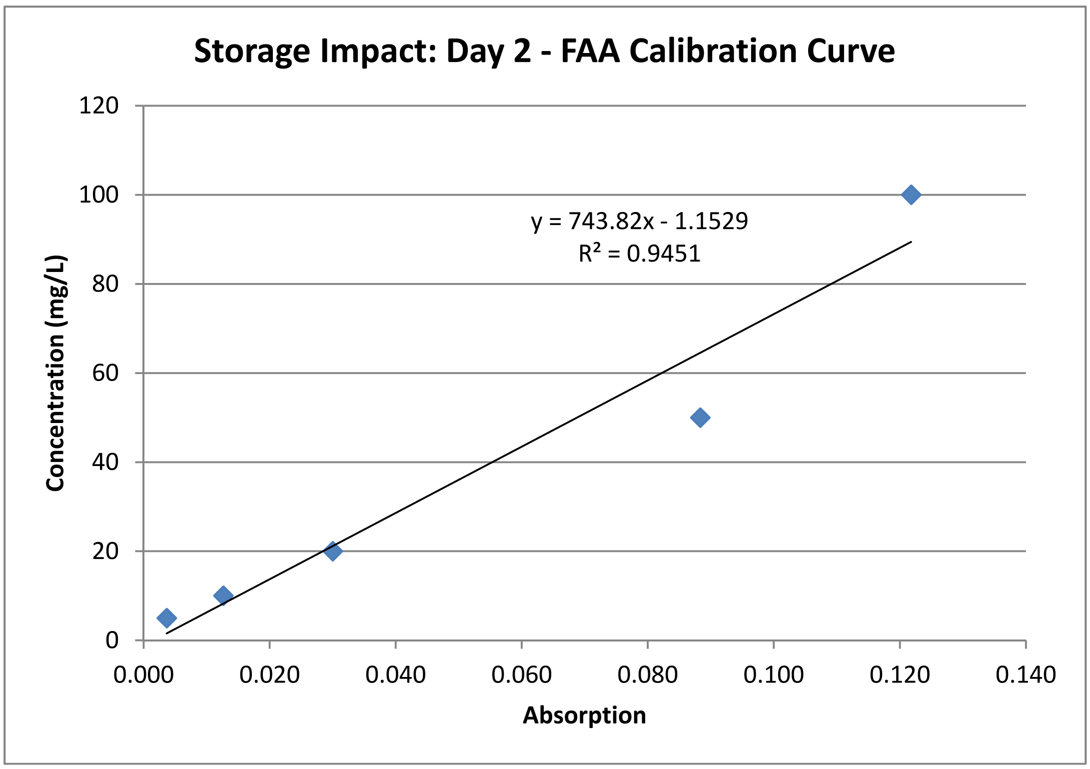

Figure A1.4: Storage Impact (Day 2) – FAA Calibration Curve

Figure A1.4 is the calibration curve obtained from the calibration data given in Table A1.18 (and copied/simplified onto this page, above).

Table A1.19: Storage Impact Results

Sample	Absorption	Std. Dev	Concentration of sample (mg/L)	Std. Dev	Concentration of soil (g/kg)	Std. Dev
Soil 1 - Day 0	0.021	0.002	12.9	2.7	1.6	0.3
Soil 2 - Day 0	0.013	0.004	8.3	3.7	1.6	0.7
Soil 3 - Day 0	0.015	0.002	9.8	2.5	1.8	0.5
Soil 4 - Day 0	0.019	0.002	11.6	2.3	2.0	0.4
Soil 1 - Day 2	0.020	0.001	13.5	2.1	1.7	0.3
Soil 2 - Day 2	0.012	0.002	7.8	2.6	1.5	0.5
Soil 3 - Day 2	0.015	0.002	10.0	2.7	1.8	0.5
Soil 4 - Day 2	0.016	0.002	11.0	2.5	1.9	0.4

*Note: for concentration of soil, the 50/0.5 is the 50 mL solution and 0.5g soil

Sample	Difference in soil Concentration (mg/kg)	Std. Dev (+/-)
Soil 1	69.5	0.0
Soil 2	-107.3	0.0
Soil 3	35.7	0.0
Soil 4	-112.5	0.0

Table A1.20: Initial Pb PBET Data

Initial Pb PBET Data (Before Amendments Added)

	Sample	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	Gastric	0.001	-0.001	0.000	0.000	0.001
	5 ppm	0.008	0.011	0.014	0.011	0.002
	10 ppm	0.021	0.024	0.023	0.023	0.001
	20 ppm	0.044	0.046	0.042	0.044	0.002
	50 ppm	0.104	0.107	0.109	0.107	0.002
	100 ppm	0.190	0.188	0.193	0.190	0.002
	Gastric	0.007	0.005	0.011	0.008	0.002
Soil Samples	Soil 1	0.027	0.021	0.024	0.024	0.002
	Soil 2	0.036	0.028	0.034	0.033	0.003
	Soil 3	0.034	0.037	0.039	0.037	0.002
	Soil 4	0.035	0.029	0.032	0.032	0.002
Control	Gastric	0.009	0.011	0.010	0.010	0.001
	10 ppm	0.031	0.033	0.035	0.033	0.002
Soil Samples	Soil 5	0.027	0.029	0.030	0.029	0.001
	Soil 6	0.034	0.039	0.036	0.036	0.002
	Soil 7	0.037	0.035	0.031	0.034	0.002
	Soil 8	0.028	0.031	0.029	0.029	0.001
Control	Gastric	0.011	0.013	0.009	0.011	0.002
	10 ppm	0.036	0.034	0.035	0.035	0.001

Sample	Absorp	Std. Dev	Concentration of sample (mg/L)	Std. Dev	Concentration of soil (g/kg)	Std. Dev
B1None_Pb	0.024	0.002	10.1	1.26	1.82	0.23
B2PA_Pb	0.033	0.003	14.7	0.76	2.50	0.13
B3LimeD0_Pb	0.037	0.002	16.8	1.47	2.56	0.22
B4LimeD5_Pb	0.032	0.002	14.3	1.26	2.65	0.23
B5LimeD20_Pb	0.029	0.001	12.6	1.90	2.64	0.40
B6LyeD0_Pb	0.036	0.002	16.6	1.47	2.54	0.22
B7LyeD5_Pb	0.034	0.002	15.6	1.24	2.76	0.22
B8LyeD20_Pb	0.029	0.001	12.9	1.90	2.81	0.41
Average/Overall_Pb	0.032	0.002	14.2	1.41	2.54	0.26

Calibration Curve: Initial Pb PBET Data	
Concentration (mg/L)	Absorption
5	0.011
10	0.023
20	0.044
50	0.107
100	0.190

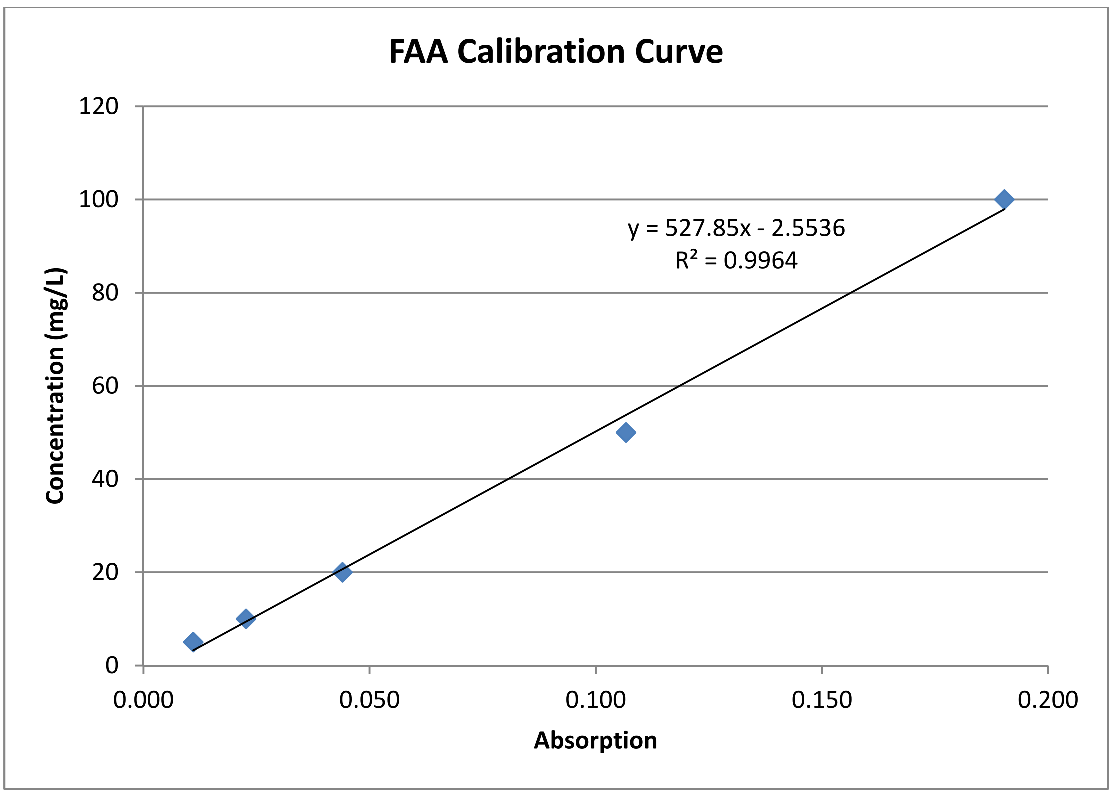

Figure A1.5: FAA Calibration Curve – Initial Pb PBET

Table A1.21: Soil Calcium Content Data

	Sample	FAA Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	HCl	0.002	-0.001	0.010		
	1 ppm	0.056	0.011	0.041	0.036	0.019
	2 ppm	0.078	0.055	0.046	0.060	0.013
	3 ppm	0.058	0.050	0.057	0.055	0.004
	4 ppm	0.083	0.080	0.074	0.079	0.004
	5 ppm	0.090	0.089	0.087	0.089	0.001
	10 ppm	0.163	0.171	0.165	0.166	0.003
	20 ppm	0.302	0.299	0.307	0.303	0.003
	50 ppm	0.740	0.734	0.753	0.742	0.008
	100 ppm	1.230	1.254	1.274	1.253	0.018
Control	HCl	0.029	0.007	0.000	0.012	0.012
	10 ppm	0.170	0.161	0.159	0.163	0.005
Soil Samples	Soil B1	0.550	0.540	0.537	0.542	0.006
	Soil B2	0.736	0.726	0.728	0.730	0.004
	Soil B3	0.750	0.698	0.824	0.757	0.052
	Soil B4	0.836	0.741	0.710	0.762	0.054
	Soil B5	0.785	0.696	0.712	0.731	0.039
	Soil B6	0.674	0.806	0.691	0.724	0.059
	Soil B7	0.753	0.744	0.758	0.752	0.006
	Soil B8	0.663	0.679	0.661	0.668	0.008
Control	HCl	-0.021	-0.023	-0.020	-0.021	0.001
	10 ppm	0.157	0.155	0.154	0.155	0.001

Sample	Absorption	Concentration of sample (mg/L)	Concentration of soil (mg/kg)
Soil 1	0.542	40.120	1003.002
Soil 2	0.730	54.971	1374.282
Soil 3	0.757	57.134	1428.358
Soil 4	0.762	57.530	1438.250
Soil 5	0.731	55.050	1376.260
Soil 6	0.724	54.470	1361.752
Soil 7	0.752	56.686	1417.147
Soil 8	0.668	50.038	1250.962
Average/Overall	0.708	53.250	1331.252

Calibration Curve: Ca Content	
Concentration (mg/L)	Absorption
1	0.036
2	0.060
3	0.055
4	0.079
5	0.089
10	0.166
20	0.303
50	0.742
100	1.253

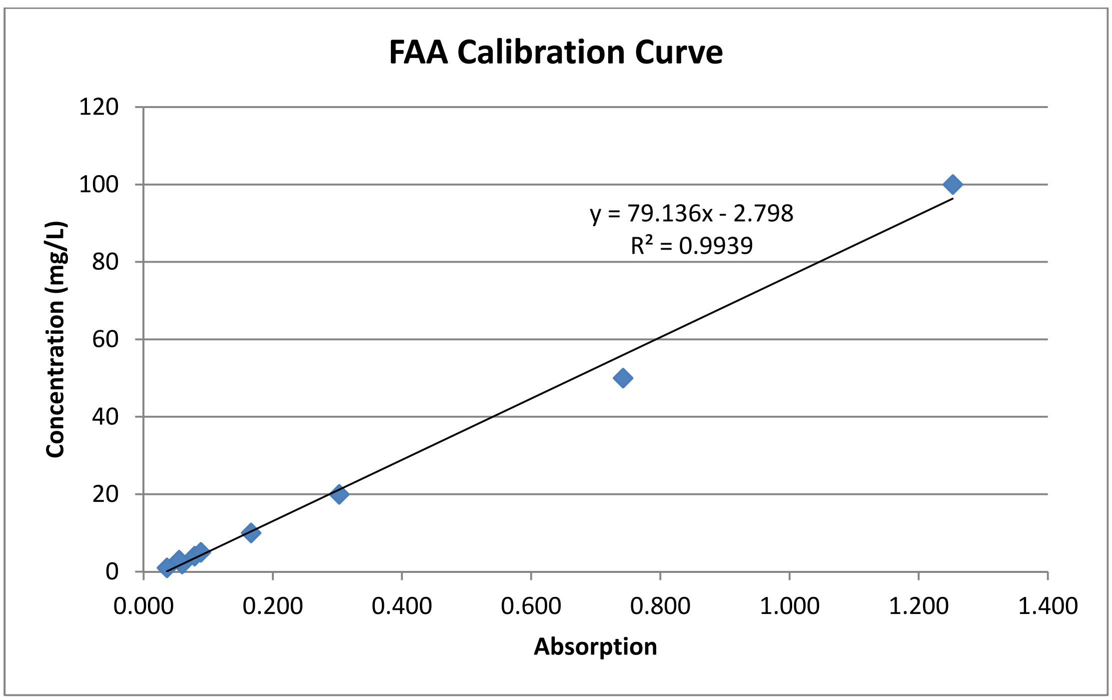

Figure A1.6: FAA Calibration Curve – Soil Calcium Content

APPENDIX B.

PBET LEAD EXPERIMENT DATA

Table A2.1: FAA Results (Day 0 Samples)

	3/1 Samples (Day 0)	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	Gastric	-0.008	-0.005	0.000	-0.004	0.003
	5 ppm	0.007	0.006	0.005	0.006	0.001
	10 ppm	0.006	0.013	0.016	0.012	0.004
	20 ppm	0.026	0.018	0.024	0.023	0.003
	50 ppm	0.058	0.061	0.054	0.058	0.003
	100 ppm	0.106	0.112	0.117	0.112	0.004
Control	Gastric	-0.002	-0.007	0.000	-0.003	0.003
	10 ppm	0.008	0.015	0.018	0.014	0.004
Soil Samples	B1.1None	0.014	0.008	0.012	0.011	0.002
	B1.2None	0.015	0.017	0.007	0.013	0.004
	B1.3None	0.011	0.012	0.006	0.010	0.003
	B2.1PA	0.018	0.006	0.021	0.015	0.006
	B2.2PA	0.019	0.015	0.009	0.014	0.004
	B2.3PA	0.015	0.018	0.019	0.017	0.002
	B3.1LimeD0	0.014	0.008	0.017	0.013	0.004
	B3.2LimeD0	0.011	0.014	0.018	0.014	0.003
	B3.3LimeD0	0.009	0.017	0.014	0.013	0.003
Control	Gastric	0.002	0.007	0.010	0.006	0.003
	10 ppm	0.018	0.021	0.016	0.018	0.002
Soil Samples	B4.1LimeD5	0.019	0.014	0.021	0.018	0.003
	B4.2LimeD5	0.014	0.024	0.018	0.019	0.004
	B4.3LimeD5	0.019	0.026	0.014	0.020	0.005
	B5.1LimeD20	0.011	0.022	0.014	0.016	0.005
	B5.2LimeD20	0.017	0.020	0.014	0.017	0.002
	B5.3LimeD20	0.014	0.017	0.021	0.017	0.003
	B6.1LyeD0	0.015	0.024	0.019	0.019	0.004
	B6.2LyeD0	0.019	0.024	0.022	0.022	0.002
	B6.3LyeD0	0.017	0.022	0.016	0.018	0.003
Control	Gastric	0.002	0.011	0.005	0.006	0.004
	10 ppm	0.018	0.025	0.015	0.019	0.004
Soil Samples	B7.1LyeD5	0.014	0.017	0.022	0.018	0.003
	B7.2LyeD5	0.012	0.022	0.018	0.017	0.004
	B7.3LyeD5	0.016	0.023	0.018	0.019	0.003
	B8.1LyeD20	0.014	0.026	0.017	0.019	0.005
	B8.2LyeD20	0.020	0.014	0.018	0.017	0.002
	B8.3LyeD20	0.016	0.021	0.017	0.018	0.002
Control	Gastric	0.009	0.005	0.008	0.007	0.002
	10 ppm	0.011	0.024	0.017	0.017	0.005

Note: gastric control average value of 0.007 used as MDL

Table A2.2: Day 0 Sample Information and Concentration Results

Day 0 Soil sample info		
Sample	FAA sample volume (mL)	Soil wt (g)
B1.1None	50	0.4
B1.2None	50	0.4
B1.3None	55	0.4
B2.1PA	61	0.4
B2.2PA	50	0.4
B2.3PA	49	0.4
B3.1LimeD0	51	0.4
B3.2LimeD0	52	0.4
B3.3LimeD0	45	0.4
B4.1LimeD5	56	0.4
B4.2LimeD5	54	0.4
B4.3LimeD5	53	0.4
B5.1LimeD20	58	0.4
B5.2LimeD20	59	0.4
B5.3LimeD20	58	0.4
B6.1LyeD0	56	0.4
B6.2LyeD0	57	0.4
B6.3LyeD0	50	0.4
B7.1LyeD5	56	0.4
B7.2LyeD5	60	0.4
B7.3LyeD5	55	0.4
B8.1LyeD20	68	0.4
B8.2LyeD20	60	0.4
B8.3LyeD20	55	0.4

Day 0 Sample Results				
Sample	Absorp	Conc of sample (mg/L)	Conc of soil (g/kg)	
B1.1None	0.011	9.570	1.20	*below MDL
B1.2None	0.013	11.064	1.38	
B1.3None	0.010	8.076	1.11	*below MDL
B2.1PA	0.015	12.857	1.96	
B2.2PA	0.014	12.259	1.53	
B2.3PA	0.017	14.948	1.83	
B3.1LimeD0	0.013	11.064	1.41	
B3.2LimeD0	0.014	12.259	1.59	
B3.3LimeD0	0.013	11.362	1.28	
B4.1LimeD5	0.018	15.546	2.18	
B4.2LimeD5	0.019	16.143	2.18	
B4.3LimeD5	0.020	17.040	2.26	
B5.1LimeD20	0.016	13.454	1.95	
B5.2LimeD20	0.017	14.649	2.16	
B5.3LimeD20	0.017	14.948	2.17	
B6.1LyeD0	0.019	16.741	2.34	
B6.2LyeD0	0.022	18.833	2.68	
B6.3LyeD0	0.018	15.845	1.98	
B7.1LyeD5	0.018	15.247	2.13	
B7.2LyeD5	0.017	14.948	2.24	
B7.3LyeD5	0.019	16.442	2.26	
B8.1LyeD20	0.019	16.442	2.80	
B8.2LyeD20	0.017	14.948	2.24	
B8.3LyeD20	0.018	15.546	2.14	

Calibration Curve	
Absorption	Concentration (mg/L)
0.006	5
0.012	10
0.023	20
0.058	50
0.112	100

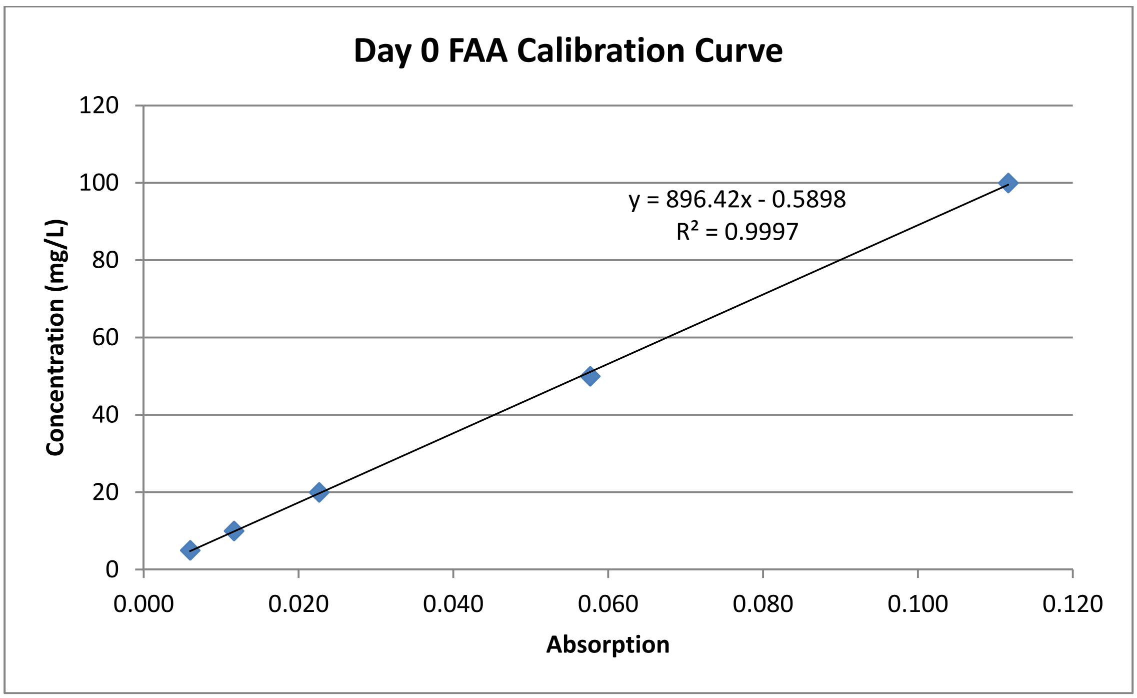

Figure A2.1: FAA Calibration Curve – Day 0 PBET Samples

Table A2.3: FAA Results (Day 1 Samples)

	3/2 Samples (Day 1)	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	Gastric	-0.001	-0.007	-0.003	-0.004	0.002
	5 ppm	0.006	-0.001	0.009	0.005	0.004
	10 ppm	0.012	0.009	0.010	0.010	0.001
	20 ppm	0.026	0.015	0.023	0.021	0.005
	50 ppm	0.064	0.057	0.054	0.058	0.004
	100 ppm	0.110	0.102	0.107	0.106	0.003
Control	Gastric	-0.001	-0.007	-0.001	-0.003	0.003
	10 ppm	0.007	0.015	0.012	0.011	0.003
Soil Samples	B1.1None	0.009	0.013	0.008	0.010	0.002
	B1.2None	0.006	0.012	0.008	0.009	0.002
	B1.3None	0.008	0.012	0.007	0.009	0.002
	B2.1PA	0.017	0.008	0.013	0.013	0.004
	B2.2PA	0.011	0.016	0.023	0.017	0.005
	B2.3PA	0.017	0.013	0.021	0.017	0.003
Control	Gastric	0.007	0.009	-0.001	0.005	0.004
	10 ppm	0.017	0.020	0.018	0.018	0.001
Soil Samples	B3.1LimeD0	0.018	0.008	0.015	0.014	0.004
	B3.2LimeD0	0.020	0.009	0.017	0.015	0.005
	B3.3LimeD0	0.014	0.019	0.017	0.017	0.002
	B6.1LyeD0	0.013	0.017	0.022	0.017	0.004
	B6.2LyeD0	0.018	0.014	0.020	0.017	0.002
	B6.3LyeD0	0.016	0.014	0.020	0.017	0.002
Control	Gastric	0.006	0.002	0.010	0.006	0.003
	10 ppm	0.022	0.015	0.018	0.018	0.003

Note: gastric control average value of 0.006 used as MDL

Note: Buckets 2, 4, 5, 7, and 8 should all behave the same, as they are all soil + PA, so only one bucket (bucket 2) was sampled to represent all of these similar buckets, in the interest of saving time and not being in the lab for 20+ hours for the second day in a row.

Table A2.4: Day 1 Sample Information and Concentration Results

Day 1 Soil sample info		
Sample	FAA sample volume (mL)	Soil wt (g)
B1.1None	65	0.4
B1.2None	71	0.4
B1.3None	75	0.4
B2.1PA	57	0.4
B2.2PA	54	0.4
B2.3PA	50	0.4
B3.1LimeD0	48	0.4
B3.2LimeD0	59	0.4
B3.3LimeD0	56	0.4
B6.1LyeD0	74	0.4
B6.2LyeD0	62	0.4
B6.3LyeD0	68	0.4

Day 1 Sample Results				
Sample	Absorp	Conc of sample (mg/L)	Conc of soil (g/kg)	
B1.1None	0.010	9.089	1.48	*below MDL
B1.2None	0.009	7.857	1.39	*below MDL
B1.3None	0.009	8.165	1.53	*below MDL
B2.1PA	0.013	11.553	1.65	
B2.2PA	0.017	15.250	2.06	
B2.3PA	0.017	15.558	1.94	
B3.1LimeD0	0.014	12.478	1.50	
B3.2LimeD0	0.015	14.018	2.07	
B3.3LimeD0	0.017	15.250	2.14	
B6.1LyeD0	0.017	15.866	2.94	
B6.2LyeD0	0.017	15.866	2.46	
B6.3LyeD0	0.017	15.250	2.59	

Calibration Curve	
Absorption	Concentration (mg/L)
0.005	5
0.010	10
0.021	20
0.058	50
0.106	100

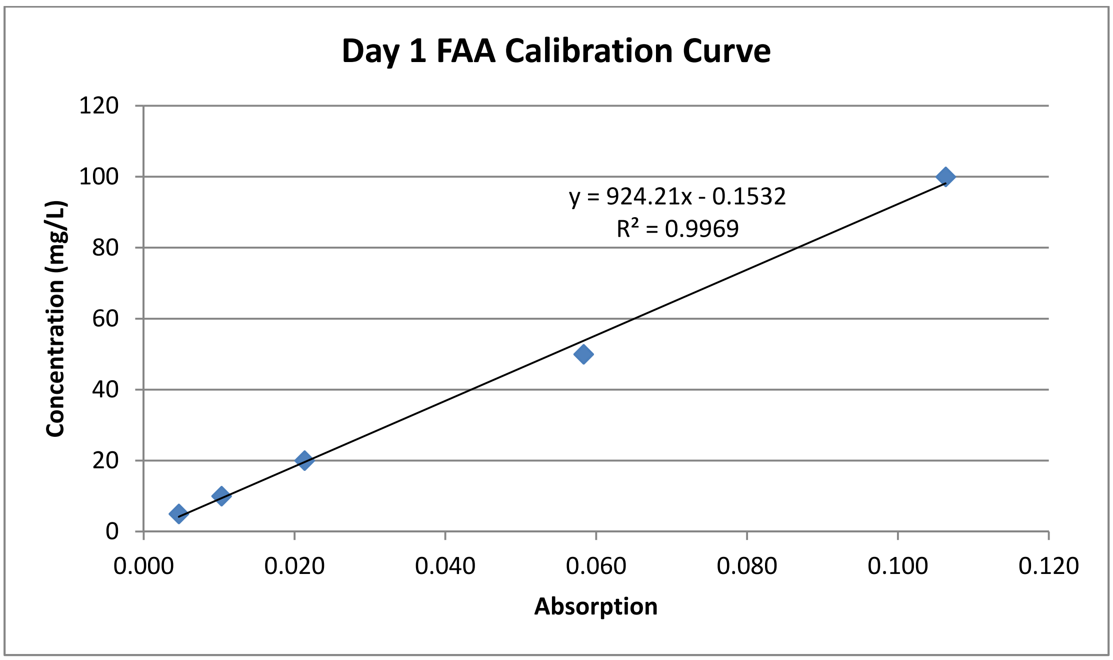

Figure A2.2: FAA Calibration Curve – Day 1 PBET Samples

Table A2.5: FAA Results (Day 4 Samples)

	3/6 Samples (Day 4)	Absorbance Trial 1	Absorbance Trial 2	Absorbance Trial 3	Average	Std. Dev
Calibration Standard	Gastric	-0.006	-0.003	-0.011	-0.007	0.003
	5 ppm	0.000	-0.003	0.004	0.000	0.003
	10 ppm	0.016	0.009	0.004	0.010	0.005
	20 ppm	0.014	0.026	0.019	0.020	0.005
	50 ppm	0.060	0.051	0.058	0.056	0.004
	100 ppm	0.113	0.004	0.114	0.077	0.052
Control	Gastric	-0.002	-0.001	-0.005	-0.003	0.002
	10 ppm	0.010	0.015	0.008	0.011	0.003
Soil Samples	B1.1None	0.012	0.003	0.007	0.007	0.004
	B1.2None	0.008	0.013	0.007	0.009	0.003
	B1.3None	0.009	0.012	0.017	0.013	0.003
	B2.1PA	0.007	0.011	0.015	0.011	0.003
	B2.2PA	0.009	0.015	0.020	0.015	0.004
	B2.3PA	0.021	0.014	0.008	0.014	0.005
	B3.1LimeD0	0.008	0.014	0.017	0.013	0.004
	B3.2LimeD0	0.007	-0.003	0.008	0.004	0.005
	B3.3LimeD0	0.009	0.017	0.007	0.011	0.004
Control	Gastric	0.003	0.014	-0.007	0.003	0.009
	10 ppm	0.012	0.011	0.021	0.015	0.004
Soil Samples	B4.1LimeD5	0.018	0.004	0.009	0.010	0.006
	B4.2LimeD5	0.006	0.020	0.014	0.013	0.006
	B4.3LimeD5	0.023	0.009	0.013	0.015	0.006
	B5.1LimeD20	0.023	0.018	0.009	0.017	0.006
	B5.2LimeD20	0.015	0.022	0.018	0.018	0.003
	B5.3LimeD20	0.012	0.025	0.017	0.018	0.005
	B6.1LyeD0	0.015	0.023	0.016	0.018	0.004
	B6.2LyeD0	0.020	0.014	0.011	0.015	0.004
	B6.3LyeD0	0.016	0.022	0.018	0.019	0.002
Control	Gastric	0.007	-0.002	0.002	0.002	0.004
	10 ppm	0.007	0.016	0.023	0.015	0.007
Soil Samples	B7.1LyeD5	0.013	0.018	0.023	0.018	0.004
	B7.2LyeD5	0.018	0.022	0.012	0.017	0.004
	B7.3LyeD5	0.018	0.023	0.014	0.018	0.004
	B8.1LyeD20	0.012	0.021	0.016	0.016	0.004
	B8.2LyeD20	0.013	0.017	0.026	0.019	0.005
	B8.3LyeD20	0.014	0.019	0.022	0.018	0.003
Control	Gastric	0.004	0.012	0.000	0.005	0.005
	10 ppm	0.018	0.013	0.021	0.017	0.003

*gastric control average value of 0.005 used as MDL

Table A2.6: Day 4 Sample Information and Concentration Results

Day 4 Soil sample info			Day 4 Sample Results				
Sample	FAA sample volume (mL)	Soil wt (g)	Sample	Absorp	Conc of sample (mg/L)	Conc of soil (g/kg)	
B1.1None	76	0.4	B1.1None	0.007	7.541	1.43	*below MDL
B1.2None	55	0.4	B1.2None	0.009	9.872	1.36	*below MDL
B1.3None	44	0.4	B1.3None	0.013	13.759	1.51	
B2.1PA	67	0.4	B2.1PA	0.011	11.816	1.98	*below MDL
B2.2PA	50	0.4	B2.2PA	0.015	16.091	2.01	
B2.3PA	50	0.4	B2.3PA	0.014	15.702	1.96	
B3.1LimeD0	54	0.4	B3.1LimeD0	0.013	14.147	1.91	
B3.2LimeD0	78	0.4	B3.2LimeD0	0.004	3.654	0.71	*below MDL
B3.3LimeD0	70	0.4	B3.3LimeD0	0.011	11.816	2.07	*below MDL
B4.1LimeD5	63	0.4	B4.1LimeD5	0.010	11.038	1.74	*below MDL
B4.2LimeD5	49	0.4	B4.2LimeD5	0.013	14.536	1.78	
B4.3LimeD5	55	0.4	B4.3LimeD5	0.015	16.479	2.27	
B5.1LimeD20	44	0.4	B5.1LimeD20	0.017	18.422	2.03	
B5.2LimeD20	52	0.4	B5.2LimeD20	0.018	20.366	2.65	
B5.3LimeD20	49	0.4	B5.3LimeD20	0.018	19.977	2.45	
B6.1LyeD0	51	0.4	B6.1LyeD0	0.018	19.977	2.55	
B6.2LyeD0	56	0.4	B6.2LyeD0	0.015	16.479	2.31	
B6.3LyeD0	44	0.4	B6.3LyeD0	0.019	20.754	2.28	
B7.1LyeD5	60	0.4	B7.1LyeD5	0.018	19.977	3.00	
B7.2LyeD5	53	0.4	B7.2LyeD5	0.017	19.200	2.54	
B7.3LyeD5	48	0.4	B7.3LyeD5	0.018	20.366	2.44	
B8.1LyeD20	60	0.4	B8.1LyeD20	0.016	18.034	2.71	
B8.2LyeD20	63	0.4	B8.2LyeD20	0.019	20.754	3.27	
B8.3LyeD20	52	0.4	B8.3LyeD20	0.018	20.366	2.65	

Concentration (mg/L)	
Absorption	Concentration (mg/L)
0.000	5
0.010	10
0.020	20
0.056	50
0.077	100

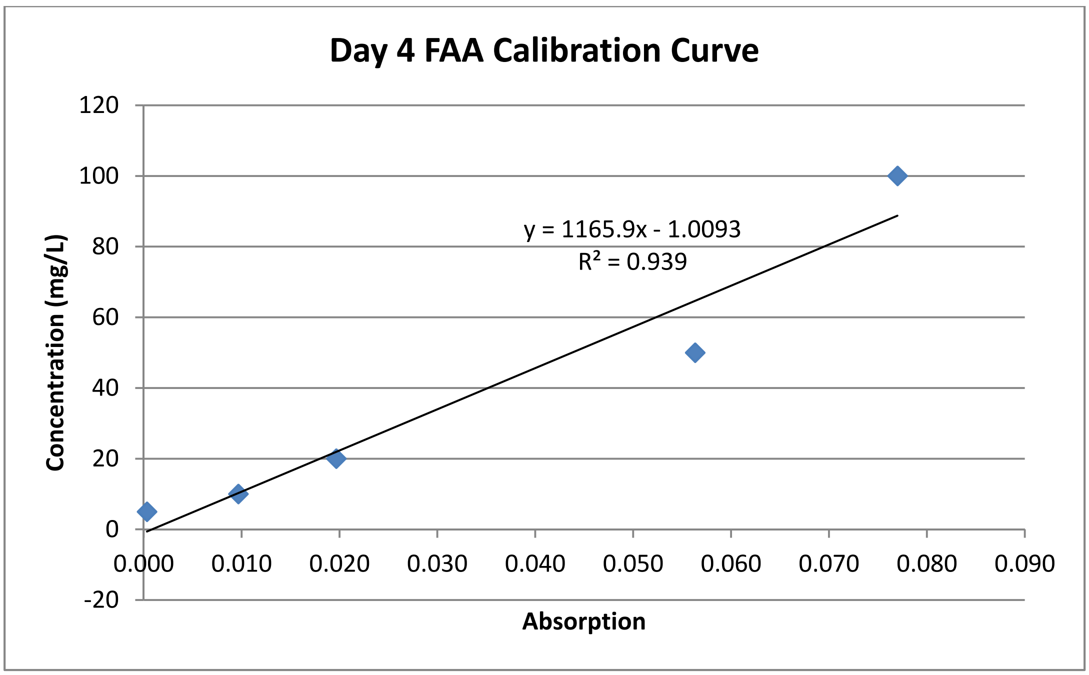

Figure A2.3: FAA Calibration Curve – Day 4 PBET Samples

Table A2.7: FAA Results (Day 7 Samples)

	3/11 Samples	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	Gastric	-0.002	-0.004	0.000	-0.002	0.002
	5 ppm	0.008	0.006	0.001	0.005	0.003
	10 ppm	0.007	0.010	0.013	0.010	0.002
	20 ppm	0.015	0.028	0.021	0.021	0.005
	50 ppm	0.047	0.056	0.059	0.054	0.005
	100 ppm	0.096	0.102	0.099	0.099	0.002
Control	Gastric	-0.002	0.004	0.000	0.001	0.002
	10 ppm	0.017	0.005	0.015	0.012	0.005
Soil Samples	B1.1None	0.009	0.020	0.012	0.014	0.005
	B1.2None	0.006	0.012	0.008	0.009	0.002
	B1.3None	0.003	0.011	0.008	0.007	0.003
	B2.1PA	0.010	0.024	0.016	0.017	0.006
	B2.2PA	0.011	0.020	0.018	0.016	0.004
	B2.3PA	0.013	0.019	0.015	0.016	0.002
	B3.1LimeD0	0.013	0.015	0.019	0.016	0.002
	B3.2LimeD0	0.007	0.019	0.014	0.013	0.005
	B3.3LimeD0	0.009	0.015	0.008	0.011	0.003
Control	Gastric	0.008	0.015	0.001	0.008	0.006
	10 ppm	0.015	0.023	0.017	0.018	0.003
Soil Samples	B4.1LimeD5	0.012	0.020	0.017	0.016	0.003
	B4.2LimeD5	0.012	0.021	0.015	0.016	0.004
	B4.3LimeD5	0.011	0.015	0.021	0.016	0.004
	B5.1LimeD20	0.015	0.025	0.018	0.019	0.004
	B5.2LimeD20	0.012	0.022	0.017	0.017	0.004
	B5.3LimeD20	0.013	0.021	0.016	0.017	0.003
	B6.1LyeD0	0.016	0.022	0.018	0.019	0.002
	B6.2LyeD0	0.014	0.020	0.018	0.017	0.002
	B6.3LyeD0	0.016	0.022	0.018	0.019	0.002
Control	Gastric	0.013	0.005	0.008	0.009	0.003
	10 ppm	0.014	0.023	0.018	0.018	0.004
Soil Samples	B7.1LyeD5	0.015	0.021	0.018	0.018	0.002
	B7.2LyeD5	0.015	0.024	0.020	0.020	0.004
	B7.3LyeD5	0.016	0.022	0.019	0.019	0.002
	B8.1LyeD20	0.014	0.024	0.018	0.019	0.004
	B8.2LyeD20	0.016	0.023	0.017	0.019	0.003
	B8.3LyeD20	0.015	0.024	0.019	0.019	0.004
Control	Gastric	0.006	0.014	0.010	0.010	0.003
	10 ppm	0.017	0.025	0.019	0.020	0.003

*gastric control average value of 0.010 used as MDL

Table A2.8: Day 7 Sample Information and Concentration Results

Day 7 Soil sample info		
Sample	FAA sample volume (mL)	Soil wt (g)
B1.1None	59	0.4
B1.2None	93	0.4
B1.3None	56	0.4
B2.1PA	53	0.4
B2.2PA	51	0.4
B2.3PA	61	0.4
B3.1LimeD0	53	0.4
B3.2LimeD0	56	0.4
B3.3LimeD0	108	0.4
B4.1LimeD5	53	0.4
B4.2LimeD5	64	0.4
B4.3LimeD5	87	0.4
B5.1LimeD20	44	0.4
B5.2LimeD20	54	0.4
B5.3LimeD20	56	0.4
B6.1LyeD0	58	0.4
B6.2LyeD0	59	0.4
B6.3LyeD0	55	0.4
B7.1LyeD5	60	0.4
B7.2LyeD5	54	0.4
B7.3LyeD5	59	0.4
B8.1LyeD20	65	0.4
B8.2LyeD20	62	0.4
B8.3LyeD20	62	0.4

Day 7 Sample Results				
Sample	Absorp	Conc of sample (mg/L)	Conc of soil (g/kg)	
B1.1None	0.014	12.726	1.88	
B1.2None	0.009	7.711	1.79	*below MDL
B1.3None	0.007	6.373	0.89	*below MDL
B2.1PA	0.017	15.735	2.08	
B2.2PA	0.016	15.400	1.96	
B2.3PA	0.016	14.732	2.25	
B3.1LimeD0	0.016	14.732	1.95	
B3.2LimeD0	0.013	12.391	1.73	
B3.3LimeD0	0.011	9.717	2.62	*below MDL
B4.1LimeD5	0.016	15.400	2.04	
B4.2LimeD5	0.016	15.066	2.41	
B4.3LimeD5	0.016	14.732	3.20	
B5.1LimeD20	0.019	18.409	2.03	
B5.2LimeD20	0.017	16.069	2.17	
B5.3LimeD20	0.017	15.735	2.20	
B6.1LyeD0	0.019	17.741	2.57	
B6.2LyeD0	0.017	16.403	2.42	
B6.3LyeD0	0.019	17.741	2.44	
B7.1LyeD5	0.018	17.072	2.56	
B7.2LyeD5	0.020	18.744	2.53	
B7.3LyeD5	0.019	18.075	2.67	
B8.1LyeD20	0.019	17.741	2.88	
B8.2LyeD20	0.019	17.741	2.75	
B8.3LyeD20	0.019	18.409	2.85	

Calibration Curve	
Absorption	Concentration (mg/L)
0.005	5
0.010	10
0.021	20
0.054	50
0.099	100

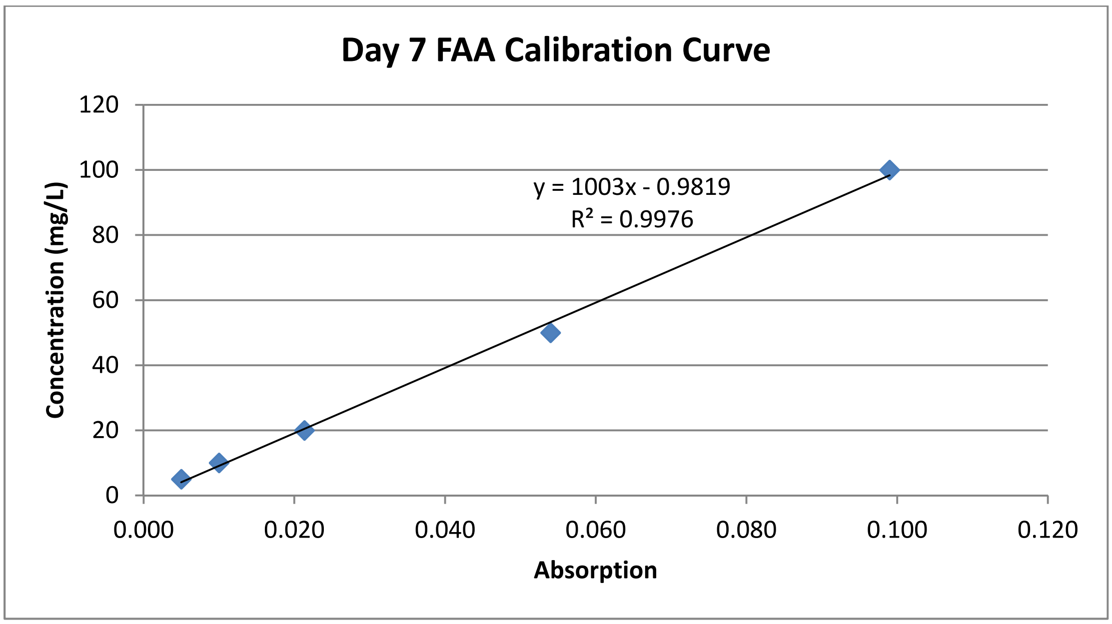

Figure A2.4: FAA Calibration Curve – Day 7 PBET Samples

Table A2.9: FAA Results (Day 10 Samples)

	3/13 Samples (Day 10)	Absorbance Trial 1	Absorbance Trial 2	Absorbance Trial 3	Average	Std. Dev
Calibration Standard	Gastric	-0.007	-0.002	-0.014	-0.008	0.005
	5 ppm	-0.010	0.003	-0.002	-0.003	0.005
	10 ppm	-0.003	0.010	0.004	0.004	0.005
	20 ppm	0.009	0.021	0.016	0.015	0.005
	50 ppm	0.037	0.050	0.041	0.043	0.005
	100 ppm	0.074	0.089	0.083	0.082	0.006
Control	Gastric	-0.002	-0.014	0.008	-0.003	0.009
	10 ppm	-0.006	0.010	0.004	0.003	0.007
Soil Samples	B1.1None	-0.004	0.010	0.003	0.003	0.006
	B1.2None	-0.001	0.008	0.001	0.003	0.004
	B1.3None	-0.001	0.011	0.005	0.005	0.005
	B2.1PA	-0.001	0.006	0.014	0.006	0.006
	B2.2PA	-0.004	0.009	0.004	0.003	0.005
	B2.3PA	-0.004	0.019	0.007	0.007	0.009
	B3.1LimeD0	-0.002	0.007	0.016	0.007	0.007
	B3.2LimeD0	-0.004	0.009	0.015	0.007	0.008
	B3.3LimeD0	-0.003	0.020	0.007	0.008	0.009
Control	Gastric	-0.004	-0.012	0.000	-0.005	0.005
	10 ppm	-0.002	0.014	0.009	0.007	0.007
Soil Samples	B4.1LimeD5	-0.003	0.015	0.008	0.007	0.007
	B4.2LimeD5	-0.002	0.015	0.011	0.008	0.007
	B4.3LimeD5	-0.002	0.017	0.007	0.007	0.008
	B5.1LimeD20	-0.003	0.014	0.008	0.006	0.007
	B5.2LimeD20	-0.002	0.021	0.007	0.009	0.009
	B5.3LimeD20	-0.002	0.018	0.009	0.008	0.008
	B6.1LyeD0	-0.003	0.015	0.004	0.005	0.007
	B6.2LyeD0	-0.004	0.014	0.007	0.006	0.007
	B6.3LyeD0	-0.005	0.017	0.005	0.006	0.009
Control	Gastric	-0.017	-0.006	0.002	-0.007	0.008
	10 ppm	-0.005	0.012	0.008	0.005	0.007
Soil Samples	B7.1LyeD5	-0.003	0.016	0.008	0.007	0.008
	B7.2LyeD5	-0.003	0.012	0.007	0.005	0.006
	B7.3LyeD5	-0.004	0.016	0.006	0.006	0.008
	B8.1LyeD20	-0.004	0.015	0.007	0.006	0.008
	B8.2LyeD20	-0.008	0.001	0.014	0.002	0.009
	B8.3LyeD20	-0.006	0.012	0.001	0.002	0.007
Control	Gastric	-0.017	-0.009	0.001	-0.008	0.007
	10 ppm	-0.004	0.017	0.003	0.005	0.009

Note: gastric control average value of -0.003 used as MDL

Table A2.10: Day 10 Sample Information and Concentration Results

Day 10 Soil sample info			Day 10 Sample Results				
Sample	FAA sample volume (mL)	Soil wt (g)	Sample	Absorp	Conc of sample (mg/L)	Conc of soil (g/kg)	
B1.1None	48	0.4	B1.1None	0.003	8.689	1.04	*below MDL
B1.2None	45	0.4	B1.2None	0.003	8.314	0.94	*below MDL
B1.3None	31	0.4	B1.3None	0.005	10.942	0.85	*below MDL
B2.1PA	59	0.4	B2.1PA	0.006	12.444	1.84	*below MDL
B2.2PA	49	0.4	B2.2PA	0.003	8.689	1.06	*below MDL
B2.3PA	45	0.4	B2.3PA	0.007	13.570	1.53	*below MDL
B3.1LimeD0	50	0.4	B3.1LimeD0	0.007	13.195	1.65	*below MDL
B3.2LimeD0	44	0.4	B3.2LimeD0	0.007	12.820	1.41	*below MDL
B3.3LimeD0	42	0.4	B3.3LimeD0	0.008	14.321	1.50	*below MDL
B4.1LimeD5	61	0.4	B4.1LimeD5	0.007	12.820	1.95	*below MDL
B4.2LimeD5	46	0.4	B4.2LimeD5	0.008	14.321	1.65	*below MDL
B4.3LimeD5	52	0.4	B4.3LimeD5	0.007	13.570	1.76	*below MDL
B5.1LimeD20	53	0.4	B5.1LimeD20	0.006	12.444	1.65	*below MDL
B5.2LimeD20	50	0.4	B5.2LimeD20	0.009	15.072	1.88	*below MDL
B5.3LimeD20	54	0.4	B5.3LimeD20	0.008	14.697	1.98	*below MDL
B6.1LyeD0	53	0.4	B6.1LyeD0	0.005	11.318	1.50	*below MDL
B6.2LyeD0	58	0.4	B6.2LyeD0	0.006	11.693	1.70	*below MDL
B6.3LyeD0	64	0.4	B6.3LyeD0	0.006	11.693	1.87	*below MDL
B7.1LyeD5	57	0.4	B7.1LyeD5	0.007	13.195	1.88	*below MDL
B7.2LyeD5	53	0.4	B7.2LyeD5	0.005	11.318	1.50	*below MDL
B7.3LyeD5	53	0.4	B7.3LyeD5	0.006	12.069	1.60	*below MDL
B8.1LyeD20	53	0.4	B8.1LyeD20	0.006	12.069	1.60	*below MDL
B8.2LyeD20	58	0.4	B8.2LyeD20	0.002	7.938	1.15	*below MDL
B8.3LyeD20	64	0.4	B8.3LyeD20	0.002	7.938	1.27	*below MDL

Calibration Curve	
Absorption	Concentration (mg/L)
-0.003	5
0.004	10
0.015	20
0.043	50
0.082	100

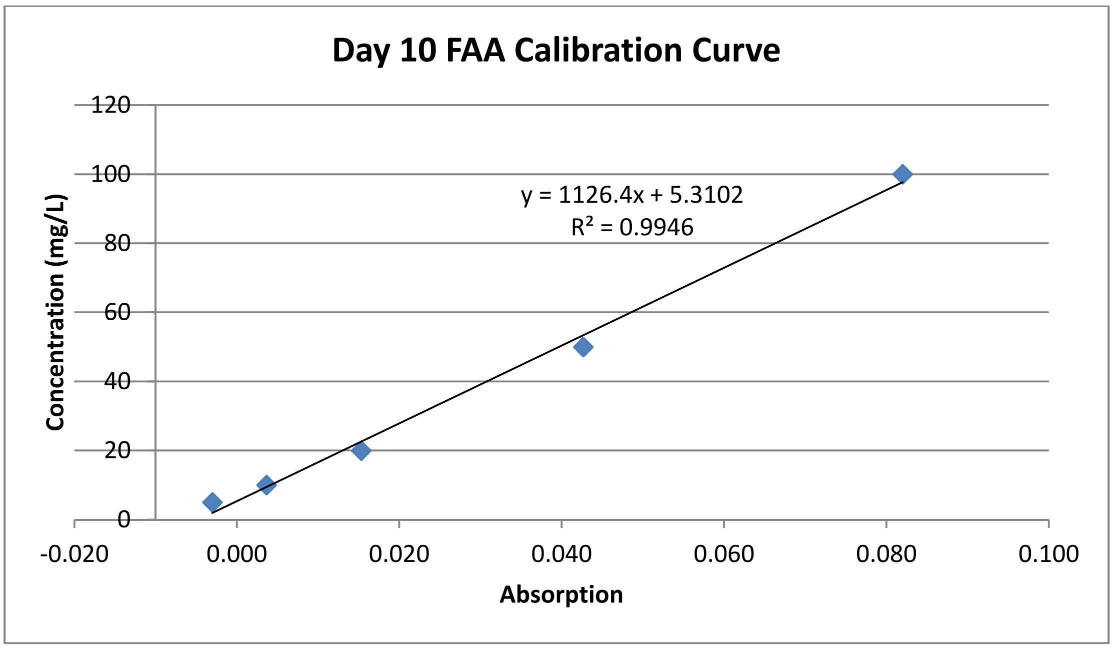

Figure A2.5: FAA Calibration Curve – Day 10 PBET Samples

Table A2.11: FAA Results (Day 15 Samples)

	3/17 Samples (Day 15)	Absorbance Trial 1	Absorbance Trial 2	Absorbance Trial 3	Average	Std. Dev
Calibration Standard	Gastric	-0.011	0.000	-0.005	-0.005	0.004
	5 ppm	0.009	-0.009	0.004	0.001	0.008
	10 ppm	0.015	-0.001	0.009	0.008	0.007
	20 ppm	0.009	0.024	0.018	0.017	0.006
	50 ppm	0.055	0.038	0.048	0.047	0.007
	100 ppm	0.085	0.104	0.091	0.093	0.008
Control	Gastric	0.004	-0.014	-0.004	-0.005	0.007
	10 ppm	0.004	0.017	0.009	0.010	0.005
Soil Samples	B1.1None	0.002	0.017	0.014	0.011	0.006
	B1.2None	0.005	0.017	0.011	0.011	0.005
	B1.3None	-0.001	0.021	0.009	0.010	0.009
	B2.1PA	0.007	0.024	0.014	0.015	0.007
	B2.2PA	0.006	0.023	0.015	0.015	0.007
	B2.3PA	0.004	0.021	0.016	0.014	0.007
	B3.1LimeD0	0.002	0.021	0.014	0.012	0.008
	B3.2LimeD0	0.005	0.020	0.016	0.014	0.006
	B3.3LimeD0	0.007	0.022	0.014	0.014	0.006
Control	Gastric	0.013	-0.008	0.002	0.002	0.009
	10 ppm	0.006	0.024	0.017	0.016	0.007
Soil Samples	B4.1LimeD5	0.005	0.024	0.016	0.015	0.008
	B4.2LimeD5	0.006	0.026	0.018	0.017	0.008
	B4.3LimeD5	0.006	0.022	0.014	0.014	0.007
	B5.1LimeD20	0.004	0.025	0.019	0.016	0.009
	B5.2LimeD20				0.000	0.000
	B5.3LimeD20	0.007	0.022	0.017	0.015	0.006
	B6.1LyeD0	-0.003	0.014	0.006	0.006	0.007
	B6.2LyeD0	-0.001	0.014	0.007	0.007	0.006
	B6.3LyeD0	0.002	0.021	0.010	0.011	0.008
Control	Gastric	-0.005	0.014	0.006	0.005	0.008
	10 ppm	0.002	0.020	0.016	0.013	0.008
Soil Samples	B7.1LyeD5	0.005	0.020	0.014	0.013	0.006
	B7.2LyeD5	0.002	0.023	0.014	0.013	0.009
	B7.3LyeD5	0.002	0.015	0.009	0.009	0.005
	B8.1LyeD20	-0.005	0.015	0.006	0.005	0.008
	B8.2LyeD20	-0.002	0.016	0.005	0.006	0.007
	B8.3LyeD20	-0.005	0.016	0.007	0.006	0.009
Control	Gastric	-0.005	0.016	0.004	0.005	0.009
	10 ppm	-0.003	0.017	0.007	0.007	0.008

Note: gastric control average value of 0.005 used as MDL
Note: sample 5.2 was spilled during PBET, no sample collected

Table A2.12: Day 15 Sample Information and Concentration Results

Day 15 Soil sample info		
Sample	FAA sample volume (mL)	Soil wt (g)
B1.1None	52	0.4
B1.2None	52	0.4
B1.3None	60	0.4
B2.1PA	56	0.4
B2.2PA	61	0.4
B2.3PA	64	0.4
B3.1LimeD0	55	0.4
B3.2LimeD0	52	0.4
B3.3LimeD0	55	0.4
B4.1LimeD5	53	0.4
B4.2LimeD5	57	0.4
B4.3LimeD5	63	0.4
B5.1LimeD20	49	0.4
B5.2LimeD20	spill	0.4
B5.3LimeD20	51	0.4
B6.1LyeD0	53	0.4
B6.2LyeD0	62	0.4
B6.3LyeD0	53	0.4
B7.1LyeD5	61	0.4
B7.2LyeD5	61	0.4
B7.3LyeD5	55	0.4
B8.1LyeD20	57	0.4
B8.2LyeD20	69	0.4
B8.3LyeD20	62	0.4

Day 15 Sample Results				
Sample	Absorp	Conc of sample (mg/L)	Conc of soil (g/kg)	
B1.1None	0.011	13.895	1.81	*below MDL
B1.2None	0.011	13.895	1.81	*below MDL
B1.3None	0.010	12.512	1.88	*below MDL
B2.1PA	0.015	18.046	2.53	
B2.2PA	0.015	17.700	2.70	
B2.3PA	0.014	16.663	2.67	
B3.1LimeD0	0.012	15.279	2.10	
B3.2LimeD0	0.014	16.663	2.17	
B3.3LimeD0	0.014	17.354	2.39	
B4.1LimeD5	0.015	18.046	2.39	
B4.2LimeD5	0.017	19.776	2.82	
B4.3LimeD5	0.014	17.008	2.68	
B5.1LimeD20	0.016	19.084	2.34	
B5.2LimeD20	0.000			
B5.3LimeD20	0.015	18.392	2.34	
B6.1LyeD0	0.006	8.361	1.11	*below MDL
B6.2LyeD0	0.007	9.399	1.46	*below MDL
B6.3LyeD0	0.011	13.895	1.84	*below MDL
B7.1LyeD5	0.013	15.971	2.44	
B7.2LyeD5	0.013	15.971	2.44	
B7.3LyeD5	0.009	11.474	1.58	*below MDL
B8.1LyeD20	0.005	8.015	1.14	*below MDL
B8.2LyeD20	0.006	9.053	1.56	*below MDL
B8.3LyeD20	0.006	8.707	1.35	*below MDL

Calibration Curve	
Absorption	Concentration (mg/L)
0.001	5
0.008	10
0.017	20
0.047	50
0.093	100

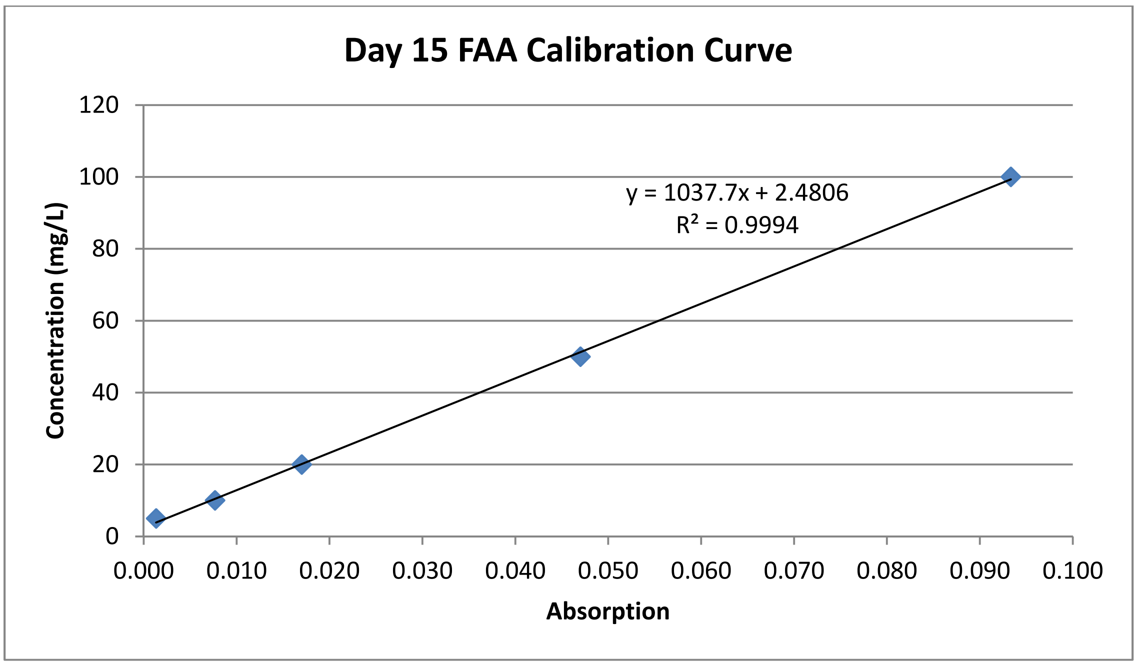

Figure A2.6: FAA Calibration Curve – Day 15 PBET Samples

Table 2.13: FAA Soil Pb Concentration Over Time

	Soil Pb Concentrations (g/kg)						
Sample	Initial Samples	Day 0 Samples	Day 1 Samples	Day 4 Samples	Day 7 Samples	Day 10 Samples	Day 15 Samples
Sample Day	-1	0	1	4	7	10	15
B1None	1.82	1.23	1.47	1.43	1.52	0.94	1.83
B2PA	2.50	1.77	1.88	1.98	2.10	1.48	2.63
B3LimeD0	2.56	1.43	1.90	1.56	2.10	1.52	2.22
B4LimeD5	2.65	2.20		1.93	2.55	1.79	2.63
B5LimeD20	2.64	2.09		2.37	2.13	1.84	2.34
B6LyeD0	2.54	2.34	2.66	2.38	2.48	1.69	1.47
B7LyeD5	2.76	2.21		2.66	2.59	1.66	2.15
B8LyeD20	2.81	2.39		2.87	2.83	1.34	1.35

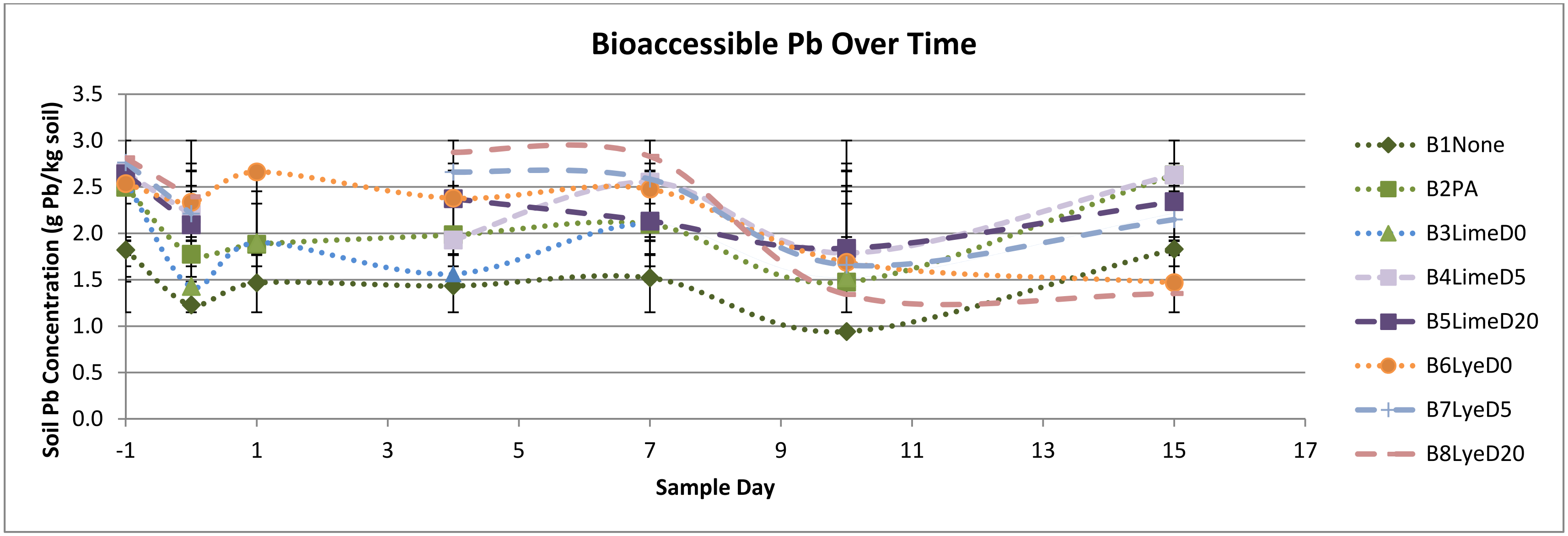

Figure A2.7: FAA Bioaccessible Pb Over Time

Table A2.14: Change in Bioaccessible Pb – FAA

Change of Pb Concentration in Soil (mg/L)

	Decrease in Pb conc (sample day - initial value)						Decrease in Pb conc (current day -previous day)				
Sample	Decrease in Pb conc (initial and day 0)	Decrease in Pb conc (initial and day 1)	Decrease in Pb conc (initial and day 4)	Decrease in Pb conc (initial and day 7)	Decrease in Pb conc (initial and day 10)	Decrease in Pb conc (initial and day 15)	Decrease in Pb conc (day 0 and day 1)	Decrease in Pb conc (day 1 and day 4)	Decrease in Pb conc (day 4 and day 7)	Decrease in Pb conc (day 7 and day 10)	Decrease in Pb conc (day 10 and day 15)
B1None	590.8	353.2	386.1	300.0	878.6	-9.2	-237.6	32.9	-86.1	578.7	-887.8
B2PA	722.5	613.9	512.8	398.9	1021.7	-133.4	-108.6	-101.1	-113.9	622.8	-1155.0
B3LimeD0	1134.6	662.1	998.7	458.7	1041.0	344.4	-472.5	336.6	-540.0	582.3	-696.6
B4LimeD5	447.9		724.1	100.7	863.8	23.1			-623.4	763.1	-840.6
B5LimeD20	548.4		267.7	509.0	802.4	300.0			241.3	293.4	-502.4
B6LyeD0	199.3	-127.1	156.3	58.2	846.6	1066.7	-326.4	283.3	-98.1	788.4	220.1
B7LyeD5	551.0		102.1	177.8	1103.9	614.0			75.7	926.1	-489.9
B8LyeD20	420.6		-61.5	-16.4	1472.2	1461.2			45.1	1488.6	-11.0

Note: green indicates lead lost, while red indicates lead gained

Table A2.15: GFAA Calibration Curve

Sample ID	Mean Signal (Abs)	Entered Conc. (ug/L)	Calculated Conc. (ug/L)	Standard Deviation	% RSD
Blank	0.0000	0.0	0.000	0.00	11.6
Calib Std 1	0.0386	20.0	16.735	0.01	19.3
Calib Std 2	0.1178	50.0	53.254	0.01	5.5
Calib Std 3	0.2258	100.0	108.482	0.00	1.7
Calib Std 4	0.3904	200.0	207.209	0.01	1.3
Calib Std 5	0.6306	400.0	385.203	0.00	0.1
Calib Std 6	0.7102	500.0	473.377	0.00	0.1

Correlation coeff: 0.997738 Slope: 0.00235 Intercept: 0.00000

The above values were all given by the GFAA. The graph below is derived from the data above. Any data with a signal above 0.7102 is marked as "out of range" to the right of the table (tables A2.8 through A2.29).

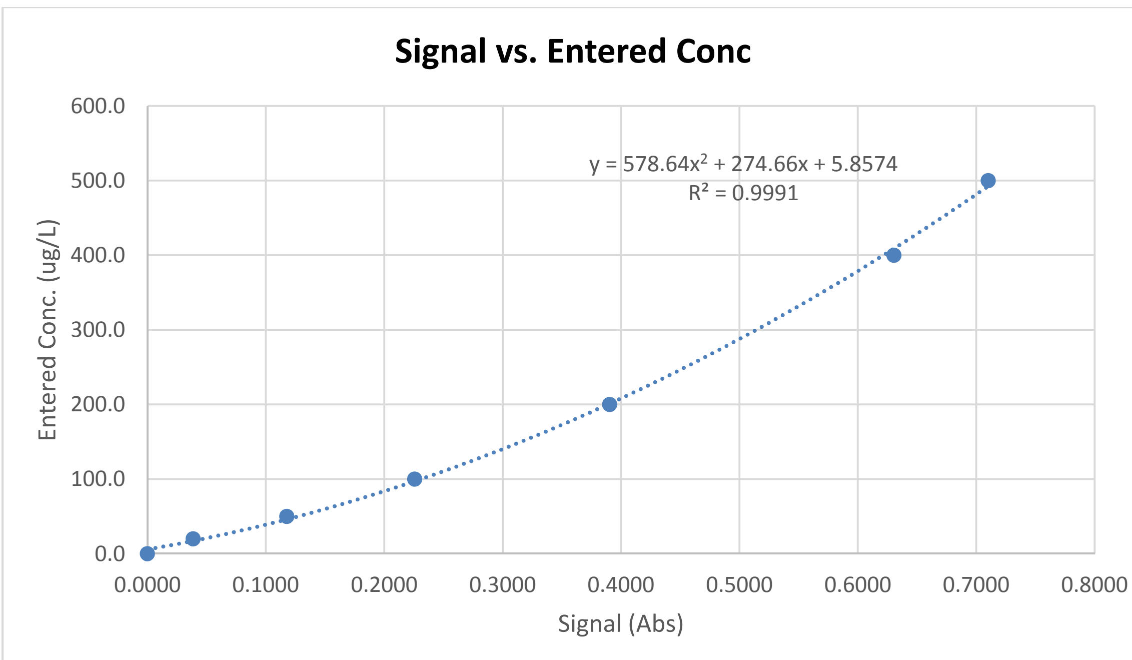

Figure A2.8: GFAA Calibration Curve - Signal vs. Entered Concentration

Table A2.16: GFAA Results – Day 7 Samples

Samples (Day 7)	Mean Abs	SD	Mean Conc (ug/L)	SD	
Blank	0.0120	0.0006	0.000	0.00	
Calib Std 1	0.0910	0.0050	23.964	0.00	
Calib Std 2	0.1946	0.0050	52.802	0.00	calibration samples not used. See GFAA calibration curve
Calib Std 3	0.3492	0.0009	100.126	0.00	
Calib Std 4	0.5926	0.0008	193.458	0.00	
Calib Std 5	0.8707	0.0050	383.515	0.00	
Calib Std 6	0.9747	0.0073	536.532	0.01	
standard	0.6926	0.0074	473.66	7.92	
control (GI soln)	0.8390	0.0061	643.61	7.55	
B1.1None	0.9893	0.0128	843.90	9.47	out of range
B1.2None	0.6453	0.0048	424.05	7.19	
B1.3None	0.5391	0.0030	322.10	6.69	
control (GI soln)	0.8392	0.0061	643.86	7.55	
B2.1PA	0.8739	0.0034	687.79	6.80	
B2.2PA	0.7646	0.0021	554.14	6.44	
B2.3PA	0.7178	0.0066	501.14	7.70	
control (GI soln)	0.8432	0.0041	648.86	6.99	
standard	0.6968	0.0040	478.19	6.97	
B3.1LimeD0	0.6290	0.0048	407.55	7.19	
B3.2LimeD0	0.6006	0.0062	379.55	7.58	
B3.3LimeD0	0.3858	0.0032	197.95	6.74	
control (GI soln)	0.8419	0.0107	647.23	8.86	
B4.1LimeD5	0.7294	0.0027	514.04	6.60	
B4.2LimeD5	0.5549	0.0013	336.44	6.22	
B4.3LimeD5	0.5536	0.0068	335.25	7.75	
control (GI soln)	0.8364	0.0024	640.38	6.52	
B5.1LimeD20	0.9561	0.0094	797.41	8.49	
standard	0.6974	0.0042	478.84	7.02	
B5.2LimeD20	0.5550	0.0063	336.53	7.61	
B5.3LimeD20	0.7602	0.0045	549.05	7.11	
control (GI soln)	0.8369	0.0050	641.00	7.25	
B6.1LyeD0	0.8544	0.0064	662.93	7.64	
B6.2LyeD0	0.9111	0.0063	736.43	7.61	
B6.3LyeD0	0.9729	0.0056	820.78	7.41	
control (GI soln)	0.8409	0.0034	645.98	6.80	
B7.1LyeD5	0.7005	0.0022	482.20	6.46	
B7.2LyeD5	0.6748	0.0007	454.68	6.05	
standard	0.6919	0.0031	472.90	6.71	

B7.3LyeD5	0.6336	0.0025	412.18	6.55
control (GI soln)	0.8394	0.0038	644.11	6.91
B8.1LyeD20	0.7387	0.0030	524.50	6.69
B8.2LyeD20	0.8059	0.0032	603.02	6.74
B8.3LyeD20	0.7039	0.0061	485.89	7.55
control (GI soln)	0.8414	0.0024	646.61	6.52
standard	0.6995	0.0084	481.11	8.21

Table A2.17: Comparison of Standard and Control Samples – Day 7

Comparison: standard

Sample	Conc. Mean	SD
standard	473.66	7.92
standard	478.19	6.97
standard	478.84	7.02
standard	472.90	6.71
standard	481.11	8.21

Comparison: control (GI soln)

Sample	Conc. Mean	SD
control (GI soln)	643.61	7.55
control (GI soln)	643.86	7.55
control (GI soln)	648.86	6.99
control (GI soln)	647.23	8.86
control (GI soln)	640.38	6.52
control (GI soln)	641.00	7.25
control (GI soln)	645.98	6.80
control (GI soln)	644.11	6.91
control (GI soln)	646.61	6.52

Table A2.18: GFAA Samples – Day 10

Day 10 Samples	Mean	SD	Mean Conc	SD
Blank	0.0015	0.0004		
Calib Std 1	0.0924	0.0134		
Calib Std 2	0.1929	0.0084		
Calib Std 3	0.3582	0.0024		
Calib Std 4	0.5465	0.1035		
Calib Std 5	0.7141	0.1578		
Calib Std 6	0.6609	0.0036		
standard	0.453	0.0027	249.02	6.60
control (GI soln)	0.0468	0.0004	19.98	5.97
B1.1None	0.3482	0.0069	171.65	7.78
B1.2None	0.3306	0.0038	159.90	6.91
B1.3None	0.3871	0.0079	198.89	8.06
control (GI soln)	0.0485	0.0017	20.54	6.33
B2.1PA	0.3738	0.0009	189.38	6.11
B2.2PA	0.3563	0.0016	177.18	6.30
B2.3PA	0.3218	0.0026	154.16	6.58
control (GI soln)	0.0523	0.0005	21.80	5.99
standard	0.4413	0.007	239.75	7.81
B3.1LimeD0	0.2968	0.0066	138.35	7.70
B3.2LimeD0	0.1958	0.1709	81.82	69.70
B3.3LimeD0	0.3147	0.0062	149.60	7.58
control (GI soln)	0.0535	0.0014	22.21	6.24
B4.1LimeD5	0.3462	0.0071	170.30	7.84
B4.2LimeD5	0.3371	0.013	164.20	9.53
B4.3LimeD5	0.3449	0.0217	169.42	12.09
control (GI soln)	0.0521	0.0017	21.74	6.33
B5.1LimeD20	0.3561	0.0031	177.04	6.71
standard	0.4498	0.0021	246.47	6.44
B5.2LimeD20	0.3146	0.0075	149.54	7.95
B5.3LimeD20	0.3464	0.0091	170.43	8.40
control (GI soln)	0.0549	0.0021	22.68	6.44
B6.1LyeD0	0.3469	0.0131	170.77	9.55
B6.2LyeD0	0.3564	0.0082	177.25	8.15
B6.3LyeD0	0.3837	0.0089	196.44	8.35
control (GI soln)	0.056	0.0013	23.05	6.22
B7.1LyeD5	0.3033	0.0025	142.39	6.55
B7.2LyeD5	0.3362	0.0046	163.60	7.13
standard	0.4447	0.0108	242.43	8.89
B7.3LyeD5	0.3417	0.0102	167.27	8.72
control (GI soln)	0.0577	0.0034	23.63	6.80
B8.1LyeD20	0.4033	0.0329	210.74	15.52
B8.2LyeD20	0.3719	0.0072	188.03	7.86

*calibration samples not used. See GFAA calibration curve

B8.3LyeD20	0.3527	0.0036	174.71	6.85
control (GI soln)	0.0581	0.0008	23.77	6.08
DI+HCL pH 1.8	0.0079	0.0001	8.06	5.88
pepsin	0.0008	0.0002	6.08	5.91
malate	0.0098	0.0003	8.60	5.94
standard	0.4572	0.002	252.39	6.41
acetic acid	0.2273	0.0039	98.18	6.94
citrate	-0.0007	0.0001	5.67	5.88
lactic acid	0.0153	0.0006	10.20	6.02
standard	-0.0013	0.0002	5.50	5.91

Table A2.19: Comparison of Standard and Control Samples – Day 10

Comparison: standard

Sample	Conc. Mean	SD
standard	249.02	6.60
standard	239.75	7.81
standard	246.47	6.44
standard	242.43	8.89
standard	252.39	6.41
standard	5.50	5.91

Comparison: control (GI soln)

Sample	Conc. Mean	SD
control (GI soln)	19.98	5.97
control (GI soln)	20.54	6.33
control (GI soln)	21.80	5.99
control (GI soln)	22.21	6.24
control (GI soln)	21.74	6.33
control (GI soln)	22.68	6.44
control (GI soln)	23.05	6.22
control (GI soln)	23.63	6.80
control (GI soln)	23.77	6.08

Table A2.20: GFAA Samples – Day 15 (run on 6/4)

Day 15 Samples (6/4)	Mean Absorp	SD	Mean Conc (ug/L)	SD	
Blank	0.0048	0.0002			
Calib Std 1	1.1326	0.0417			
Calib Std 2	1.3940	0.0299			
Calib Std 3	1.6737	0.0332			calibration samples not used. See GFAA calibration curve
Calib Std 4	1.8669	0.0248			
Calib Std 5	1.8603	0.0138			
Calib Std 6	1.7513	0.0298			
standard	1.9115	0.0021	2645.12	6.44	out of range
control (GI soln)	0.7097	0.0173	492.23	10.78	
B1.1None	0.8289	0.0059	631.09	7.50	out of range
B1.2None	0.7184	0.0039	501.81	6.94	
B1.3None	0.6298	0.0062	408.35	7.58	
control (GI soln)	0.6759	0.0035	455.85	6.83	
B2.1PA	0.7948	0.0017	589.69	6.33	out of range
B2.2PA	0.9899	0.0120	844.75	9.24	out of range
B2.3PA	0.8083	0.0075	605.92	7.95	out of range
control (GI soln)	0.6658	0.0031	445.23	6.71	
standard	1.9150	0.0033	2653.83	6.77	out of range
B3.1LimeD0	0.8099	0.0103	607.86	8.75	out of range
B3.2LimeD0	0.7929	0.0056	587.42	7.41	out of range
B3.3LimeD0	0.9158	0.0019	742.69	6.38	out of range
control (GI soln)	0.6686	0.0015	448.16	6.27	
B4.1LimeD5	0.8627	0.0047	673.46	7.16	out of range
B4.2LimeD5	0.6980	0.0028	479.49	6.63	
B4.3LimeD5	0.9227	0.0064	751.93	7.64	out of range
control (GI soln)	0.6661	0.0027	445.54	6.60	
B5.1LimeD20	1.0531	0.0102	936.82	8.72	out of range
standard	1.9102	0.0176	2641.89	10.87	out of range
B5.2LimeD20	0.0449	0.0213	19.36	11.97	
B5.3LimeD20	1.0703	0.0114	962.68	9.06	out of range
control (GI soln)	0.6709	0.0008	450.58	6.08	
B6.1LyeD0	1.0399	0.0023	917.21	6.49	out of range
B6.2LyeD0	0.9841	0.0059	836.54	7.50	out of range
B6.3LyeD0	1.0579	0.0025	944.01	6.55	out of range
control (GI soln)	0.6659	0.0004	445.34	5.97	
B7.1LyeD5	0.9991	0.0060	857.87	7.53	out of range

B7.2LyeD5	0.9990	0.0018	857.73	6.35	out of range
standard	1.9395	0.0047	2715.21	7.16	out of range
B7.3LyeD5	1.0520	0.0063	935.18	7.61	out of range
control (GI soln)	0.6764	0.0027	456.38	6.60	
B8.1LyeD20	0.9809	0.0011	832.02	6.16	out of range
B8.2LyeD20	0.9703	0.0048	817.14	7.19	out of range
B8.3LyeD20	0.9912	0.0106	846.60	8.83	out of range
control (GI soln)	0.6705	0.0020	450.16	6.41	
1.1 from 3/11 (10%)	0.9838	0.0152	836.11	10.17	out of range
1.1 from 3/11 (1%)	0.2471	0.0013	109.06	6.22	
standard	1.9306	0.0215	2692.83	12.03	out of range

Table A2.21: Comparison of Standard and Control Samples – Day 15 (run on 6/4)

Comparison: standard

Sample	Conc. Mean	SD
standard	2645.12	6.44
standard	2653.83	6.77
standard	2641.89	10.87
standard	2715.21	7.16
standard	2692.83	12.03

Comparison: control (GI soln)

Sample	Conc. Mean	SD
control (GI soln)	492.23	10.78
control (GI soln)	455.85	6.83
control (GI soln)	445.23	6.71
control (GI soln)	448.16	6.27
control (GI soln)	445.54	6.60
control (GI soln)	450.58	6.08
control (GI soln)	445.34	5.97
control (GI soln)	456.38	6.60
control (GI soln)	450.16	6.41

Table A2.22: GFAA Samples – Day 15 (run on 6/10)

Day 15 Samples (6/10)	Mean Absorp	SD	Mean Conc (ug/L)	SD	
Blank	0.0012	0.0002			
Calib Std 1	0.0642	0.0068			
Calib Std 2	0.1330	0.0013			
Calib Std 3	0.2490	0.0010			calibration samples not used. See GFAA calibration curve
Calib Std 4	0.4319	0.0024			
Calib Std 5	0.6745	0.0038			
Calib Std 6	0.7494	0.0064			
standard	0.5012	0.0058	288.87	7.47	
control (GI soln)	0.6115	0.0036	390.18	6.85	
B1.1None	0.9156	0.0056	742.42	7.41	out of range
B1.2None	0.7835	0.0027	576.26	6.60	out of range
B1.3None	0.7791	0.003	571.08	6.69	out of range
control (GI soln)	0.6159	0.0018	394.52	6.35	
B2.1PA	0.8114	0.0021	609.68	6.44	out of range
B2.2PA	1.008	0.0116	870.65	9.12	out of range
B2.3PA	0.8769	0.0057	691.65	7.44	out of range
control (GI soln)	0.6211	0.0031	399.67	6.71	
standard	0.5128	0.0008	298.86	6.08	
B3.1LimeD0	0.8286	0.0033	630.72	6.77	out of range
B3.2LimeD0	0.8013	0.0024	597.48	6.52	out of range
B3.3LimeD0	0.944	0.0138	780.78	9.76	out of range
control (GI soln)	0.7045	0.1407	486.55	55.96	
B4.1LimeD5	0.9038	0.0017	726.76	6.33	out of range
B4.2LimeD5	0.7656	0.0055	555.30	7.39	
B4.3LimeD5	0.9782	0.0071	828.22	7.84	out of range
control (GI soln)	0.6203	0.0028	398.87	6.63	
B5.1LimeD20	1.0967	0.0035	1003.04	6.83	out of range
standard	0.5147	0.004	300.52	6.97	
B5.2LimeD20	-0.0004	0.0003	5.75	5.94	
B5.3LimeD20	1.0596	0.0078	946.56	8.03	out of range
control (GI soln)	0.6174	0.0038	396.00	6.91	
B6.1LyeD0	1.062	0.0154	950.16	10.22	out of range
B6.2LyeD0	1.0446	0.0688	924.17	27.49	out of range
B6.3LyeD0	1.0309	0.0089	903.96	8.35	out of range
control (GI soln)	0.6177	0.0065	396.30	7.67	
B7.1LyeD5	1.0211	0.0049	889.63	7.22	out of range
B7.2LyeD5	1.0116	0.0086	875.85	8.26	out of range

standard	0.5116	0.0049	297.82	7.22	
B7.3LyeD5	1.0187	0.002	886.14	6.41	out of range
control (GI soln)	0.6172	0.0002	395.80	5.91	
B8.1LyeD20	1.0439	0.0019	923.13	6.38	out of range
B8.2LyeD20	0.9831	0.0156	835.12	10.28	out of range
B8.3LyeD20	0.9938	0.0135	850.30	9.67	out of range
control (GI soln)	0.6084	0.0005	387.14	5.99	
standard	0.5089	0.0027	295.49	6.60	

Table A2.23: Comparison of Standard and Control Samples – Day 15 (run on 6/10)

Comparison: standard

Sample	Conc. Mean	SD
standard	288.87	7.47
standard	298.86	6.08
standard	300.52	6.97
standard	297.82	7.22
standard	295.49	6.60

Comparison: control (GI soln)

Sample	Conc. Mean	SD
control (GI soln)	390.18	6.85
control (GI soln)	394.52	6.35
control (GI soln)	399.67	6.71
control (GI soln)	486.55	55.96
control (GI soln)	398.87	6.63
control (GI soln)	396.00	6.91
control (GI soln)	396.30	7.67
control (GI soln)	395.80	5.91
control (GI soln)	387.14	5.99

Table A2.24: GFAA Bioaccessible Pb Over Time (Day 7 to Day 15)

	GFAA Results: Soil Pb Concentrations (g/kg)		
Sample	Day 7 Samples	Day 10 Samples	Day 15 Samples
Sample Day	7	10	15
B1None	0.9	0.2	0.7
B2PA	0.8	0.2	1.0
B3LimeD0	0.5	0.1	0.9
B4LimeD5	0.6	0.2	0.9
B5LimeD20	0.7	0.2	1.2
B6LyeD0	1.1	0.3	1.3
B7LyeD5	0.6	0.2	1.3
B8LyeD20	0.8	0.3	1.3

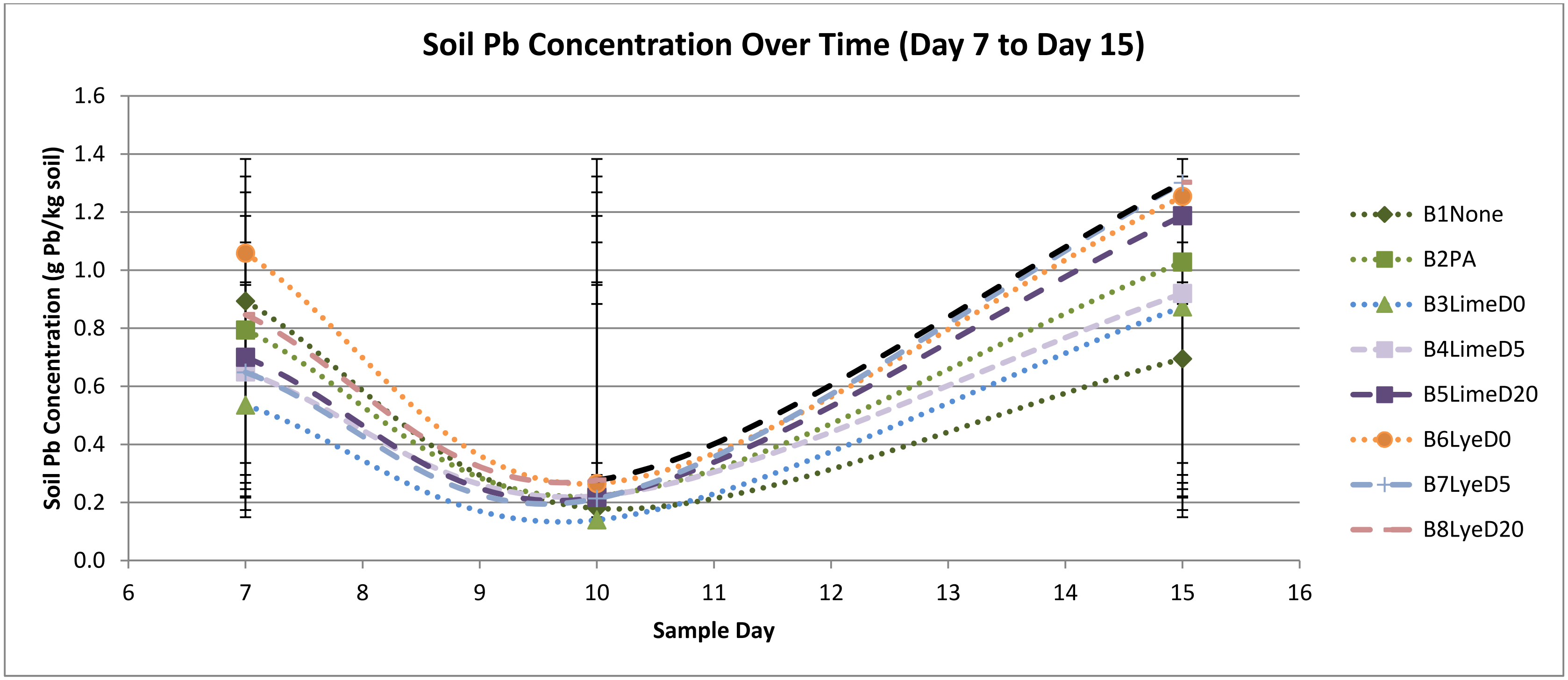

Figure A2.9: GFAA Bioaccessible Pb Over Time (Day 7 to Day 15)

Table A2.25: GFAA of PBET at pH 1.8 and pH 3 (Diluted: 10%)

PBET at pH 1.8 and 3 (Diluted to 10% initial concentration)

Sample	Mean Abs	SD	Mean Conc (ug/L)	SD	
Blank	0.0000	0.0010			
Calib Std 1	0.0344	0.0070			*calibration samples not used. See GFAA calibration curve
Calib Std 2	0.1031	0.0016			
Calib Std 3	0.2062	0.0004			
Calib Std 4	0.3704	0.0040			
Calib Std 5	0.6053	0.0072			
Calib Std 6	0.6751	0.0117			
standard	-0.0025	0.0001	5.17	5.88	
pH 3 GI soln	-0.0023	0.0002	5.23	5.91	
1 at pH 3	0.4053	0.0044	212.23	7.08	
2 at pH 3	0.3243	0.0010	155.79	6.13	
3 at pH 3	0.2616	0.0035	117.31	6.83	
4 at pH 3	0.3364	0.0036	163.73	6.85	
pH 3 GI soln	-0.0021	0.0003	5.28	5.94	
pH 1.8 GI soln	-0.0018	0.0003	5.36	5.94	
1 at pH 1.8	0.9013	0.0018	723.46	6.35	out of range
2 at pH 1.8	0.9169	0.0051	744.16	7.27	out of range
standard	-0.0020	0.0005	5.31	5.99	
3 at pH 1.8	0.8496	0.0055	656.88	7.39	out of range
4 at pH 1.8	0.9070	0.0013	730.99	6.22	out of range
pH 1.8 GI soln	-0.0017	0.0006	5.39	6.02	
standard	-0.0024	0.0003	5.20	5.94	

Table A2.26: Comparison of Standard and Control Samples – PBET at pH 1.8 and 3 (Diluted: 10%)

Comparison: standard

Sample	Conc. Mean	SD
standard	5.17	5.885
standard	5.31	5.99
standard	5.20	5.94

Comparison: control (GI soln)

Sample	Conc. Mean	SD
pH 3 GI soln	5.2	5.9
pH 3 GI soln	5.3	5.9
pH 1.8 GI soln	5.4	5.9
pH 1.8 GI soln	5.4	6.0

Table A2.27: GFAA of PBET at pH 1.8 (Diluted: 5%)

PBET at pH 1.8 (Diluted to 5% initial concentration)

Sample	Mean Abs	SD	Mean Conc (ug/L)	SD
Blank	0.0000	0.0010		
Calib Std 1	0.0362	0.0062		
Calib Std 2	0.1053	0.0031		
Calib Std 3	0.1967	0.0025		
Calib Std 4	0.3498	0.0006		
Calib Std 5	0.5748	0.0039		
Calib Std 6	0.6522	0.0051		
standard	-0.0021	0.0001	5.28	5.88
pH 1.8 GI soln	-0.0026	0.0004	5.15	5.97
1 at pH 1.8	0.6114	0.0030	390.09	6.69
2 at pH 1.8	0.6270	0.0015	405.55	6.27
3 at pH 1.8	0.5741	0.0028	354.25	6.63
4 at pH 1.8	0.6138	0.0038	392.45	6.91
pH 1.8 GI soln	-0.0020	0.0007	5.31	6.05
standard	-0.0022	0.0004	5.26	5.97

calibration samples not used. See GFAA calibration curve

Table A2.28: Comparison of Standard and Control Samples – PBET at pH 1.8 (Diluted: 5%)

Comparison: standard

Sample	Conc. Mean	SD
standard	5.28	5.88
standard	5.26	5.97

Comparison: control (GI soln)

Sample	Conc. Mean	SD
pH 1.8 GI soln	5.1	6.0
pH 1.8 GI soln	5.3	6.0

Table A2.29: GFAA of PBET at pH 1.8 (Diluted: 1%)

PBET at pH 1.8 (Diluted to 1% initial concentration)

Sample	Mean Abs	SD	Mean Conc (ug/L)	SD
Blank	0.0000	0.5235		
Calib Std 1	-0.2623	0.0015		
Calib Std 2	-0.2035	0.0008		
Calib Std 3	-0.1008	0.0011		
Calib Std 4	0.0608	0.0013		
Calib Std 5	0.2930	0.0008		
Calib Std 6	0.3819	0.0215		
standard	-0.3072	0.0005	-23.91	5.99
pH 1.8 GI soln	-0.3073	0.0003	-23.90	5.94
1 at pH 1.8	-0.1267	0.0003	-19.65	5.94
2 at pH 1.8	-0.1110	0.0016	-17.50	6.30
3 at pH 1.8	-0.1493	0.0005	-22.25	5.99
4 at pH 1.8	-0.1291	0.0023	-19.96	6.49
pH 1.8 GI soln	-0.3069	0.0003	-23.94	5.94
standard	-0.3076	0.0002	-23.88	5.91

*calibration samples not used. See GFAA calibration curve

Table A2.30: Comparison of Standard and Control Samples – PBET at pH 1.8 (Diluted: 1%)

Comparison: standard

Sample	Conc. Mean	SD
standard	-23.91	5.99
standard	-23.88	5.91

Comparison: control (GI soln)

Sample	Conc. Mean	SD
pH 1.8 GI soln	-23.9	5.9
pH 1.8 GI soln	-23.9	5.9

Table A2.31: GFAA PBET Bioaccessible Pb at pH 3 and pH 1.8

Sample	Sample volume (mL)	Soil wt (g)	Sample Conc. (mg/L)	Std. Dev	Soil Conc. (g/kg)	Std. Dev
1 at pH 3	42	0.4	2.12	0.07	0.22	0.01
2 at pH 3	53	0.4	1.56	0.06	0.21	0.01
3 at pH 3	84	0.4	1.17	0.07	0.25	0.01
4 at pH 3	69	0.4	1.64	0.07	0.28	0.01
1 at pH 1.8	60	0.4	7.80	0.13	1.17	0.02
2 at pH 1.8	61	0.4	8.11	0.13	1.24	0.02
3 at pH 1.8	70	0.4	7.09	0.13	1.24	0.02
4 at pH 1.8	59	0.4	7.85	0.14	1.16	0.02

Average pH 3	0.24	0.01
Average pH 1.8	1.20	0.02

APPENDIX C.

pH CONTROL DATA

Table A3.1: Titration Test 1 (pH 1 -> 7) - FAA

Titration Test 1 (pH 1 -> 7)

	Sample	Absorbance			Average	Std. Dev	
		Trial 1	Trial 2	Trial 3			
Calibration Standard	10% HCl	-0.003	0.000	-0.002	-0.002	0.001	
	5 ppm	0.001	0.004	0.002	0.002	0.001	
	10 ppm	0.004	0.007	0.006	0.006	0.001	
	20 ppm	0.010	0.015	0.012	0.012	0.002	
	50 ppm	0.030	0.036	0.032	0.033	0.002	
	100 ppm	0.056	0.064	0.061	0.060	0.003	
Control	10% HCl	0.004	0.000	0.002	0.002	0.002	
	10 ppm	0.008	0.012	0.010	0.010	0.002	
Samples	R1	0.046	0.051	0.048	0.048	0.002	
	R2	0.064	0.078	0.074	0.072	0.006	
	R3	0.057	0.067	0.063	0.062	0.004	
	R4	0.068	0.080	0.075	0.074	0.005	
	R5	0.077	0.081	0.075	0.078	0.002	
	R6	0.046	0.051	0.048	0.048	0.002	
	R7	0.031	0.036	0.033	0.033	0.002	
	R8	0.015	0.021	0.018	0.018	0.002	*white precipitate at bottom of vials
	R9	0.013	0.019	0.016	0.016	0.002	
Control	10% HCl	0.007	0.013	0.009	0.010	0.002	
	10 ppm	0.014	0.019	0.017	0.017	0.002	
Samples	B1	0.047	0.051	0.049	0.049	0.002	
	B2	0.053	0.061	0.058	0.057	0.003	
	B3	0.046	0.051	0.055	0.051	0.004	
	B4	0.032	0.040	0.038	0.037	0.003	
	B5	0.038	0.045	0.043	0.042	0.003	
	B6	0.041	0.049	0.045	0.045	0.003	
	B7	0.026	0.032	0.029	0.029	0.002	
	B8	0.012	0.017	0.015	0.015	0.002	*white precipitate at bottom of vials
	B9	0.014	0.019	0.017	0.017	0.002	
Control	10% HCl	0.007	0.013	0.011	0.010	0.002	
	10 ppm	0.014	0.020	0.017	0.017	0.002	

Table A3.1 (like most tables and graphs in Appendix C) have samples in the form of R# and B#, indicating that the two titration tests run that day can be distinguished by the red or black labels, respectively.

Calibration Curve	
Absorption	Conc (mg/L)
0.002	5
0.006	10
0.012	20
0.033	50
0.060	100

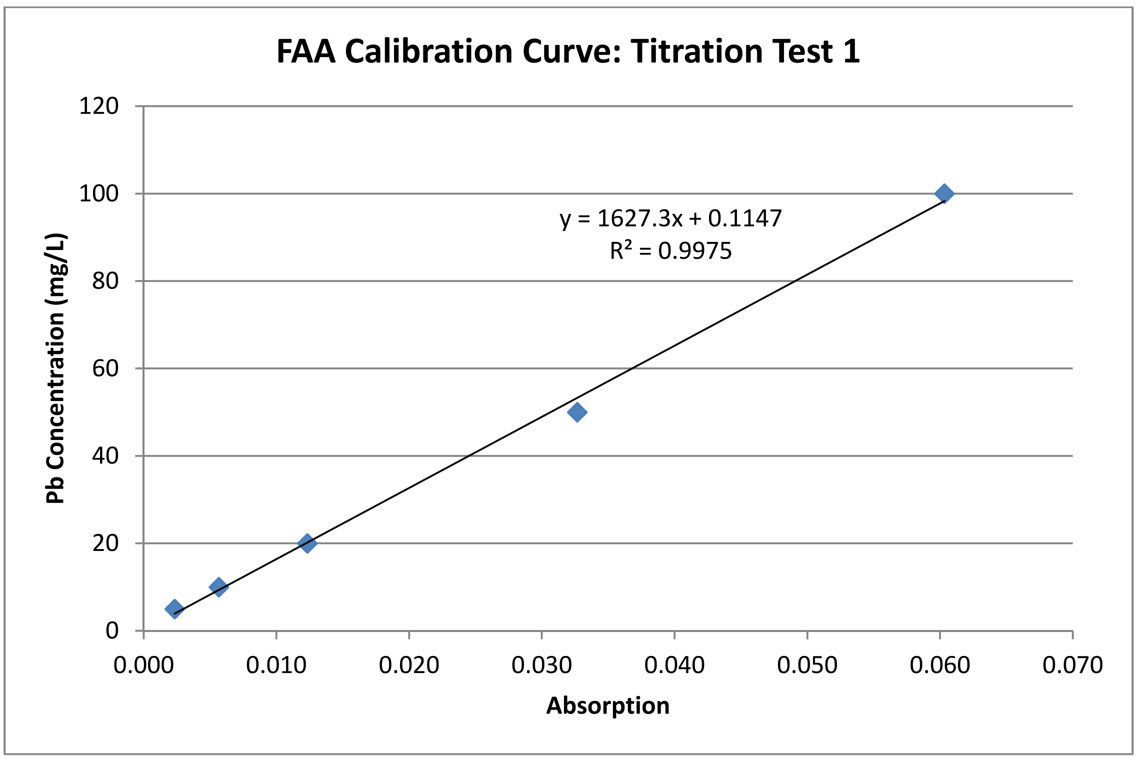

Figure A3.1: FAA Calibration Curve – Titration Test 1 (pH 1 -> 7)

Table A3.2: Volume addition and pH for Titration Test 1 (pH 1 -> 7)

Titration 1.1: Red labels

Sample	Add amt.	Δ V (mL)	Volume (mL)	pH
	0	0	500.5	1.74
	0.5	0.5	501.0	1.73
	0.5	0.5	501.5	1.73
	0.5	0.5	502.0	1.74
R1	0.5	0.5	502.5	1.74
	0.5	-9.5	493.0	1.75
	0.5	0.5	493.5	1.76
	0.5	0.5	494.0	1.78
	0.5	0.5	494.5	1.80
R2	0.5	0.5	495.0	1.82
	0.5	-9.5	485.5	1.83
	0.5	0.5	486.0	1.86
	0.2	0.2	486.2	1.88
	0.2	0.2	486.4	1.89
	0.2	0.2	486.6	1.90
R3	0.2	0.2	486.8	1.91
	0.2	-9.8	477.0	1.93
	0.2	0.2	477.2	1.94
	0.2	0.2	477.4	1.96
R4	0.2	0.2	477.6	1.97
	0.2	-9.8	467.8	1.99
	0.2	0.2	468.0	2.01
	0.2	0.2	468.2	2.03
	0.2	0.2	468.4	2.05
R5	0.2	0.2	468.6	2.07
	0.2	-9.8	458.8	2.09
	0.2	0.2	459.0	2.12
	0.2	0.2	459.2	2.15
	0.2	0.2	459.4	2.18
R6	0.2	0.2	459.6	2.22
	0.5	-9.5	450.1	2.32
	0.5	0.5	450.6	2.44
*precip	0.5	0.5	451.1	2.45
	0.2	0.2	451.3	2.52
R7	0.2	0.2	451.5	2.60
	0.2	-9.8	441.7	2.70

Titration 1.2: Black labels

Sample	Add amt.	Δ V (mL)	Volume (mL)	pH
	0	0	500.5	1.74
	0.5	0.5	501.0	1.73
	0.5	0.5	501.5	1.73
	0.5	0.5	502.0	1.74
B1	0.5	0.5	502.5	1.74
	0.5	-9.5	493.0	1.75
	0.5	0.5	493.5	1.76
	0.5	0.5	494.0	1.78
	0.5	0.5	494.5	0.79
B2	0.5	0.5	495.0	1.81
	0.5	-9.5	485.5	1.83
	0.5	0.5	486.0	1.86
	0.2	0.2	486.2	1.87
	0.2	0.2	486.4	1.89
	0.2	0.2	486.6	1.90
B3	0.2	0.2	486.8	1.91
	0.2	-9.8	477.0	1.93
	0.2	0.2	477.2	1.94
	0.2	0.2	477.4	1.96
B4	0.2	0.2	477.6	1.97
	0.2	-9.8	467.8	1.98
	0.2	0.2	468.0	2.00
	0.2	0.2	468.2	2.02
	0.2	0.2	468.4	2.04
B5	0.2	0.2	468.6	2.06
	0.2	-9.8	458.8	2.08
	0.2	0.2	459.0	2.11
	0.2	0.2	459.2	2.14
	0.2	0.2	459.4	2.17
B6	0.2	0.2	459.6	2.20
	0.5	-9.5	450.1	2.30
	0.5	0.5	450.6	2.41
*precip	0.2	0.2	450.8	2.48
	0.2	0.2	451.0	2.55
B7	0.2	0.2	451.2	2.63
	0.2	-9.8	441.4	2.76

	0.2	0.2	441.9	2.85			0.2	0.2	441.6	2.93
	0.2	0.2	442.1	2.09			0.2	0.2	441.8	3.21
	0.2	0.2	442.3	3.65			0.2	0.2	442.0	5.21
R8	0.2	0.2	442.5	5.93		B8	0.2	0.2	442.2	6.24
	0.1	-9.9	432.6	6.23			0.1	-9.9	432.3	6.44
	0.1	0.1	432.7	6.44			0.1	0.1	432.4	6.64
	0.1	0.1	432.8	6.64			0.1	0.1	432.5	6.80
	0.1	0.1	432.9	6.82			0.1	0.1	432.6	7.01
R9	0.1	0.1	433.0	6.96		B9	0.1	0.1	432.7	7.20
		-10	423.0					-10	422.7	

Note: The 10 mL decrease in sample is included in the row below each sample point (indicated by R# or B#) to indicate that the volume decreased by 10 mL after every sampling event (removal of 10 mL sample for later FAA testing)

Table A3.3: Pb Concentration Results for Titration Test 1 (pH 1 -> 7)

Sample Results for Titration Test 1 (pH 1 -> 7)

Sample	pH	FAA sample volume (mL)	Absorption	Std. Dev	Conc of sample (mg/L)	Std. Dev
R1	1.74	502.5	0.048	0.002	78.8	3.46
R2	1.82	495.0	0.072	0.006	117	9.70
R3	1.91	486.8	0.062	0.004	102	6.80
R4	1.97	477.6	0.074	0.005	121	8.12
R5	2.07	468.6	0.078	0.002	127	4.17
R6	2.22	459.6	0.048	0.002	78.8	3.46
R7	2.60	451.5	0.033	0.002	54.4	3.46
R8	5.93	442.5	0.018	0.002	29.4	4.10
R9	6.96	433.0	0.016	0.002	26.2	4.10
B1	1.74	502.5	0.049	0.002	79.9	2.77
B2	1.81	495.0	0.057	0.003	93.4	5.48
B3	1.91	486.8	0.051	0.004	82.6	6.11
B4	1.97	477.6	0.037	0.003	59.8	5.65
B5	2.06	468.6	0.042	0.003	68.5	4.91
B6	2.20	459.6	0.045	0.003	73.3	5.43
B7	2.63	451.2	0.029	0.002	47.3	4.10
B8	6.24	442.2	0.015	0.002	24.0	3.46
B9	7.20	432.7	0.017	0.002	27.2	3.46

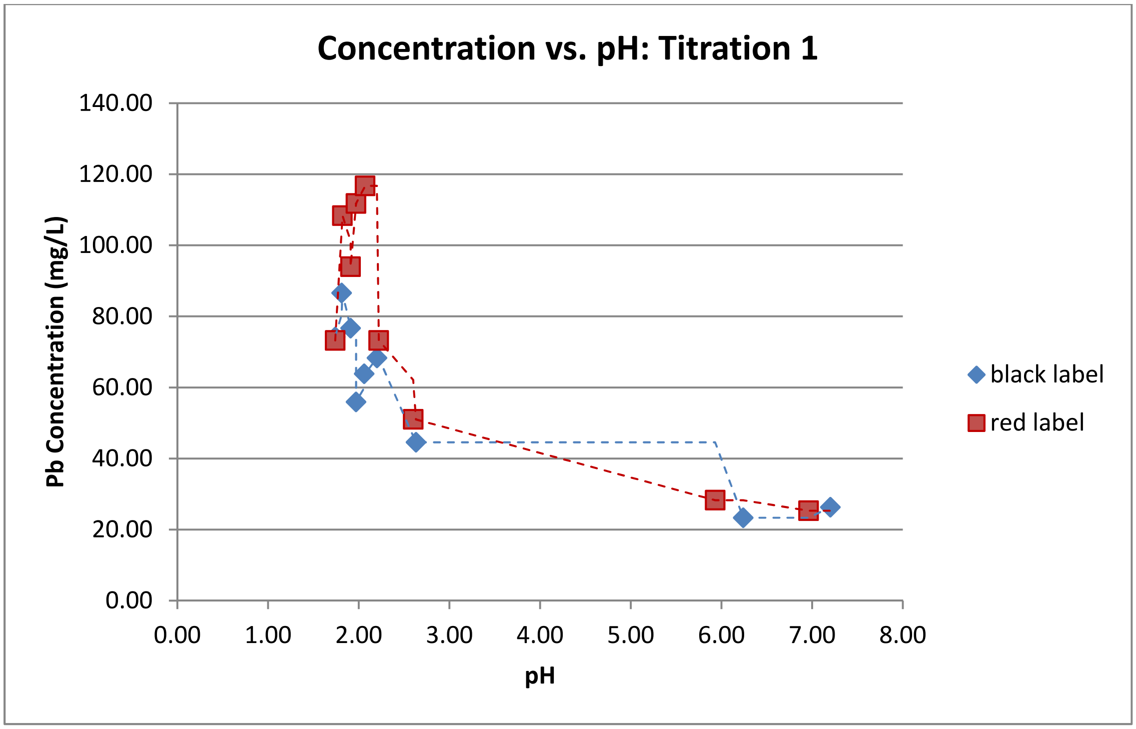

Figure A3.2: Change in Pb Concentration – Titration Test 1 (pH 1 -> 7)

Table A3.4: Titration Test 2 (pH 1 -> 7) - FAA

Titration Test 2 (pH 1 -> 7)

	Sample	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	1% HCl	-0.001	0.010	0.006	0.005	0.005
	5 ppm	0.007	0.021	0.016	0.015	0.006
	10 ppm	0.017	0.029	0.023	0.023	0.005
	20 ppm	0.033	0.047	0.040	0.040	0.006
	50 ppm	0.087	0.098	0.091	0.092	0.005
	100 ppm	0.150	0.170	0.158	0.159	0.008
Control	1% HCl	0.002	0.016	0.008	0.009	0.006
	10 ppm	0.024	0.031	0.026	0.027	0.003
Samples	R1	0.171	0.185	0.177	0.178	0.006
	R2	0.169	0.182	0.174	0.175	0.005
	R3	0.169	0.185	0.177	0.177	0.007
	R4	0.168	0.183	0.176	0.176	0.006
	R5	0.168	0.182	0.175	0.175	0.006
Control	1% HCl	0.004	0.014	0.009	0.009	0.004
	10 ppm	0.026	0.033	0.029	0.029	0.003
Samples	R6	0.168	0.183	0.176	0.176	0.006
	R7	0.169	0.181	0.176	0.175	0.005
	R8	0.167	0.181	0.176	0.175	0.006
	R9	0.086	0.100	0.095	0.094	0.006
	R10	0.067	0.080	0.075	0.074	0.005
Control	1% HCl	0.008	0.020	0.014	0.014	0.005
	10 ppm	0.026	0.039	0.030	0.032	0.005
Soil Samples	R11	0.038	0.051	0.046	0.045	0.005
	R12	0.017	0.034	0.026	0.026	0.007
	R13	0.011	0.023	0.017	0.017	0.005
	R14	0.003	0.022	0.016	0.014	0.008
	R15	0.008	0.020	0.015	0.014	0.005
Control	1% HCl	0.009	0.021	0.017	0.016	0.005
	10 ppm	0.036	0.047	0.040	0.041	0.005
Samples	B1	0.173	0.190	0.181	0.181	0.007
	B2	0.174	0.187	0.184	0.182	0.006
	B3	0.171	0.186	0.178	0.178	0.006
	B4	0.170	0.184	0.177	0.177	0.006
	B5	0.170	0.186	0.177	0.178	0.007
Control	1% HCl	0.010	0.021	0.016	0.016	0.004

	10 ppm	0.038	0.045	0.041	0.041	0.003
Samples	B6	0.171	0.190	0.179	0.180	0.008
	B7	0.172	0.184	0.176	0.177	0.005
	B8	0.115	0.124	0.118	0.119	0.004
	B9	0.084	0.096	0.089	0.090	0.005
	B10	0.060	0.072	0.067	0.066	0.005
Control	1% HCl	0.012	0.022	0.018	0.017	0.004
	10 ppm	0.034	0.049	0.039	0.041	0.006
Samples	B11	0.034	0.049	0.042	0.042	0.006
	B12	0.020	0.032	0.027	0.026	0.005
	B13	0.017	0.027	0.020	0.021	0.004
	B14	0.013	0.023	0.018	0.018	0.004
	B15	0.012	0.022	0.018	0.017	0.004
Control	1% HCl	0.015	0.022	0.017	0.018	0.003
	10 ppm	0.034	0.047	0.039	0.040	0.005

Calibration Curve	
Absorption	Concentration (mg/L)
0.015	5
0.023	10
0.040	20
0.092	50
0.159	100

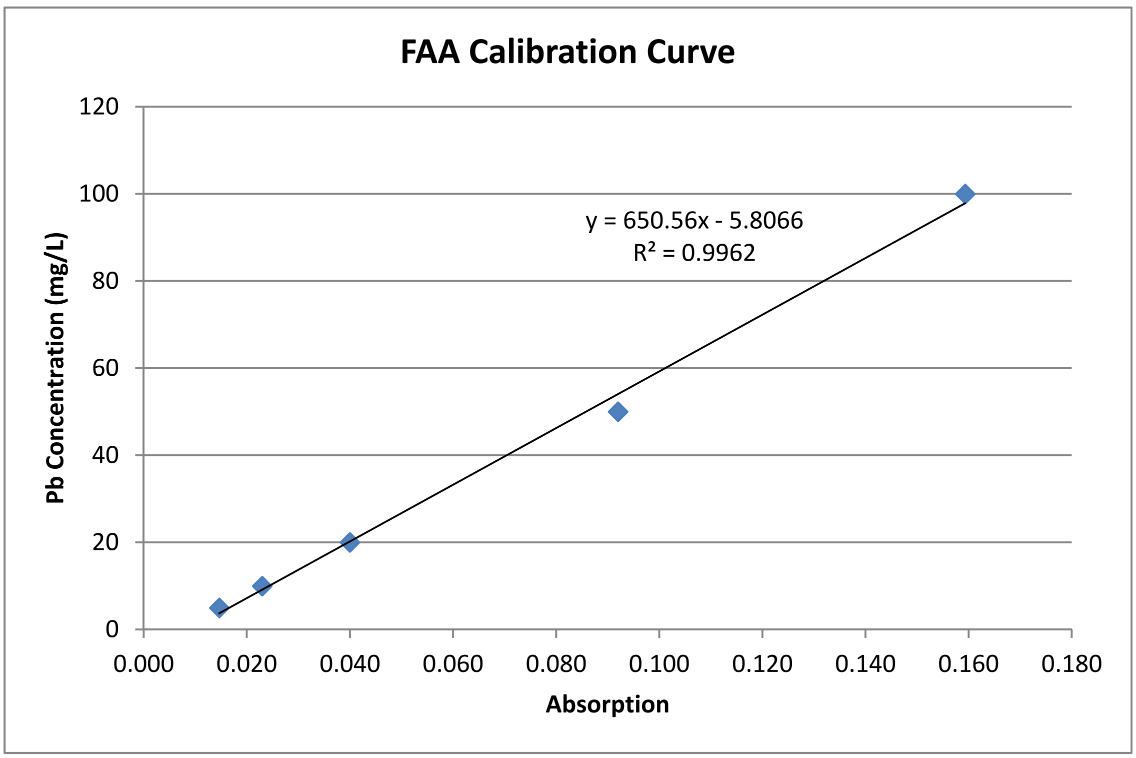

Figure A3.3: FAA Calibration Curve – Titration Test 2 (pH 1 -> 7)

Table A3.5: Volume addition and pH for Titration Test 2 (pH 1 -> 7)

Titration 1.1: Red labels				Titration 1.2: Black labels			
Sample	Δ V (mL)	Volume (mL)	pH	Sample	Δ V (mL)	Volume (mL)	pH
	0	491.0	1.55		0	491.0	1.55
R1	-9.5	481.5	1.52	B1	-9.5	481.5	1.52
	-8	473.5	1.55		-8	473.5	1.56
R2	1.5	475.0	1.60	B2	1.5	475.0	1.60
R3	-8.5	466.5	1.65	B3	-8.5	466.5	1.68
R4	-9	457.5	1.73	B4	-9	457.5	1.74
	-9.5	448.0	1.76		-9.5	448.0	1.79
R5	0.3	448.3	1.80	B5	0.3	448.3	1.81
R6	-9.4	438.9	1.85	B6	-9.4	438.9	1.88
R7	-9.5	429.4	1.92	B7	-9.5	429.4	1.94
R8	-9.5	419.9	1.98	B8	-9.5	419.9	2.03
R9	-9.5	410.4	2.09	B9	-9.5	410.4	2.12
R10	-9.7	400.7	2.16	B10	-9.7	400.7	2.20
R11	-9.5	391.2	2.32	B11	-9.5	391.2	2.39
R12	-9.5	381.7	2.59	B12	-9.5	381.7	2.68
R13	-9.7	372.0	2.92	B13	-9.7	372.0	3.06
R14	-9.8	362.2	3.47	B14	-9.8	362.2	4.84
R15	-9.8	352.4	5.98	B15	-9.8	352.4	6.26
	-10	342.4			-10	342.4	

* The 10 mL sample is included in the row below each sample point to indicate that the volume decreased after every sampling event

Table A3.6: Pb Concentration Results for Titration Test 2 (pH 1 -> 7)

Sample Results for Titration Test 2 (pH 1 -> 7)

Sample	pH	FAA sample volume (mL)	Absorption	Std. Dev	Conc of sample (mg/L)	Std. Dev
R1	1.52	481.50	0.178	0.006	110	2.08
R2	1.60	475.00	0.175	0.005	108	2.32
R3	1.65	466.50	0.177	0.007	109	1.56
R4	1.73	457.50	0.176	0.006	108	1.82
R5	1.80	448.30	0.175	0.006	108	2.09
R6	1.85	438.90	0.176	0.006	108	1.82
R7	1.92	429.40	0.175	0.005	108	2.60
R8	1.98	419.90	0.175	0.006	108	2.04
R9	2.09	410.40	0.094	0.006	55.1	2.04
R10	2.16	400.70	0.074	0.005	42.3	2.32
R11	2.32	391.20	0.045	0.005	23.5	2.32
R12	2.59	381.70	0.026	0.007	10.9	1.29
R13	2.92	372.00	0.017	0.005	5.25	2.62
R14	3.47	362.20	0.014	0.008	3.08	0.647
R15	5.98	352.40	0.014	0.005	3.52	2.60
B1	1.52	481.5	0.181	0.007	112	1.29
B2	1.60	475.0	0.182	0.006	112	2.19
B3	1.68	466.5	0.178	0.006	110	1.82
B4	1.74	457.5	0.177	0.006	109	2.09
B5	1.81	448.3	0.178	0.007	110	1.55
B6	1.88	438.9	0.180	0.008	111	0.739
B7	1.94	429.4	0.177	0.005	110	2.56
B8	2.03	419.9	0.119	0.004	71.6	3.37
B9	2.12	410.4	0.090	0.005	52.5	2.60
B10	2.20	400.7	0.066	0.005	37.3	2.60
B11	2.39	391.2	0.042	0.006	21.3	1.82
B12	2.68	381.7	0.026	0.005	11.3	2.60
B13	3.06	372.0	0.021	0.004	8.07	3.08
B14	4.84	362.2	0.018	0.004	5.90	3.15
B15	6.26	352.4	0.017	0.004	5.47	3.13

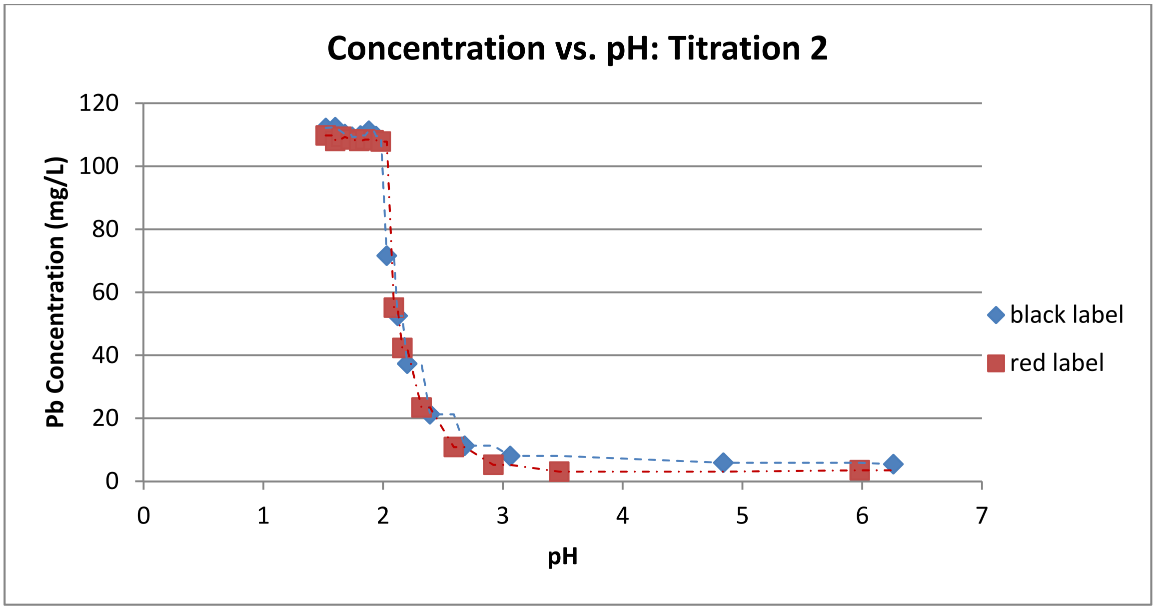

Figure A3.4: Change in Pb Concentration – Titration Test 2 (pH 1 -> 7)

Table A3.7: Titration Test 1 (pH 7 -> 1) - FAA

Titration Test 1 (pH 7 -> 1)

	Sample	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	10% HCl	0.000	-0.007	-0.004	-0.004	0.003
	5 ppm	-0.004	0.007	0.014	0.006	0.007
	10 ppm	0.007	0.024	0.018	0.016	0.007
	20 ppm	0.026	0.042	0.036	0.035	0.007
	50 ppm	0.071	0.089	0.079	0.080	0.007
	100 ppm	0.147	0.162	0.156	0.155	0.006
Control	10% HCl	0.003	0.018	0.012	0.011	0.006
	10 ppm	0.018	0.031	0.027	0.025	0.005
Samples	R1	0.006	0.022	0.016	0.015	0.007
	R2	0.004	0.024	0.015	0.014	0.008
	R3	0.025	0.039	0.028	0.031	0.006
	R4	0.036	0.050	0.043	0.043	0.006
	R5	0.031	0.048	0.035	0.038	0.007
	R6	0.025	0.042	0.038	0.035	0.007
	R7	0.012	0.028	0.017	0.019	0.007
	R8	0.011	0.026	0.019	0.019	0.006
	R9	0.014	0.027	0.020	0.020	0.005
	R10	0.014	0.027	0.022	0.021	0.005
	R11	0.017	0.029	0.021	0.022	0.005
	R12	0.017	0.031	0.026	0.025	0.006
	R13	0.018	0.032	0.028	0.026	0.006
Control	10% HCl	0.018	0.027	0.021	0.022	0.004
	10 ppm	0.030	0.043	0.038	0.037	0.005
Samples	R14	0.018	0.034	0.026	0.026	0.007
	R15	0.019	0.037	0.027	0.028	0.007
	R16	0.027	0.041	0.036	0.035	0.006
	R17	0.039	0.052	0.047	0.046	0.005
	R18	0.047	0.061	0.055	0.054	0.006
	R19	0.062	0.074	0.069	0.068	0.005
	R20	0.088	0.106	0.097	0.097	0.007
	R21	0.093	0.107	0.103	0.101	0.006
	R22	0.091	0.104	0.099	0.098	0.005
	R23	0.087	0.102	0.097	0.095	0.006
	R24	0.082	0.100	0.089	0.090	0.007
	R25	0.074	0.089	0.086	0.083	0.006

Control	10% HCl	0.016	0.034	0.028	0.026	0.007
	10 ppm	0.033	0.050	0.042	0.042	0.007
Samples	B1	0.024	0.041	0.035	0.033	0.007
	B2	0.024	0.040	0.030	0.031	0.007
	B3	0.021	0.038	0.027	0.029	0.007
	B4	0.021	0.039	0.028	0.029	0.007
	B5	cracked vial - lost sample				
	B6	0.021	0.037	0.028	0.029	0.007
	B7	0.017	0.034	0.026	0.026	0.007
	B8	0.018	0.038	0.028	0.028	0.008
	B9	0.018	0.038	0.026	0.027	0.008
	B10	0.021	0.037	0.026	0.028	0.007
	B11	0.018	0.035	0.028	0.027	0.007
	B12	0.019	0.039	0.027	0.028	0.008
	B13	0.017	0.041	0.029	0.029	0.010
Control	10% HCl	0.019	0.037	0.027	0.028	0.007
	10 ppm	0.037	0.053	0.046	0.045	0.007
Samples	B14	0.026	0.043	0.034	0.034	0.007
	B15	0.028	0.042	0.034	0.035	0.006
	B16	0.030	0.052	0.040	0.041	0.009
	B17	0.039	0.053	0.046	0.046	0.006
	B18	0.051	0.066	0.057	0.058	0.006
	B19	0.084	0.101	0.091	0.092	0.007
	B20	0.085	0.102	0.097	0.095	0.007
	B21	0.094	0.113	0.107	0.105	0.008
	B22	0.098	0.112	0.105	0.105	0.006
	B23	0.092	0.108	0.099	0.100	0.007
	B24	0.086	0.103	0.096	0.095	0.007
	B25	0.071	0.092	0.084	0.082	0.009
Control	10% HCl	0.019	0.033	0.026	0.026	0.006
	10 ppm	0.037	0.054	0.045	0.045	0.007

Calibration Curve	
Absorption	Concentration (mg/L)
0.006	5
0.016	10
0.035	20
0.080	50
0.155	100

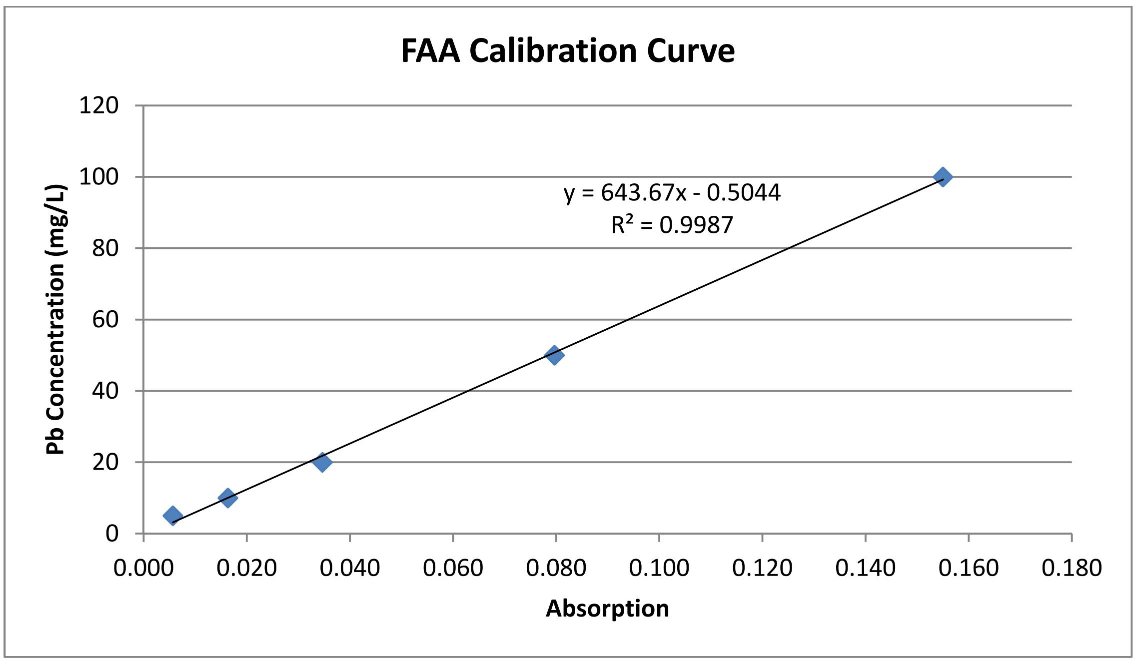

Figure A3.5: FAA Calibration Curve – Titration Test 1 (pH 7 -> 1)

Table A3.8: Volume addition and pH for Titration Test 1 (pH 7 -> 1)

Titration 1.1: Red labels					Titration 1.2: Black labels				
Sample	Add amt.	Δ V (mL)	Volume (mL)	pH	Sample	Add amt.	Δ V (mL)	Volume (mL)	pH
	0	0	500.6	10.21		0	0	500.7	10.30
	0.1	0.1	500.7	10.18		0.1	0.1	500.8	10.28
	0.1	0.1	500.8	10.11		0.1	0.1	500.9	10.22
	0.1	0.1	500.9	10.08		0.1	0.1	501.0	10.16
	0.1	0.1	501.0	10.03		0.1	0.1	501.1	10.17
R1	0.1	0.1	501.1	10.00	B1	0.1	0.1	501.2	10.13
	0.1	-9.9	491.2	9.92		0.1	-9.9	491.3	10.09
	0.1	0.1	491.3	9.84		0.1	0.1	491.4	10.03
	0.1	0.1	491.4	9.77		0.1	0.1	491.5	9.99
	0.1	0.1	491.5	9.66		0.1	0.1	491.6	9.94
	0.1	0.1	491.6	9.54		0.1	0.1	491.7	9.89
R2	0.1	0.1	491.7	9.37	B2	0.1	0.1	491.8	9.77
	0.1	-9.9	481.8	9.08		0.1	-9.9	481.9	9.66
	0.1	0.1	481.9	8.65		0.1	0.1	482.0	9.56
	0.1	0.1	482.0	8.28		0.1	0.1	482.1	9.42
	0.1	0.1	482.1	8.04		0.1	0.1	482.2	9.21
	0.1	0.1	482.2	7.86		0.1	0.1	482.3	8.90
R3	0.1	0.1	482.3	7.73	B3	0.1	0.1	482.4	8.41
	0.1	-9.9	472.4	7.61		0.1	-9.9	472.5	8.08
	0.1	0.1	472.5	7.49		0.1	0.1	472.6	7.83
	0.1	0.1	472.6	7.41		0.1	0.1	472.7	7.72
	0.1	0.1	472.7	7.33		0.1	0.1	472.8	7.63
	0.1	0.1	472.8	7.27		0.1	0.1	472.9	7.53
R4	0.1	0.1	472.9	7.22	B4	0.1	0.1	473.0	7.43
	0.1	-9.9	463.0	7.16		0.1	-9.9	463.1	7.34
	0.1	0.1	463.1	7.11		0.1	0.1	463.2	7.27
	0.1	0.1	463.2	7.03		0.1	0.1	463.3	7.21
	0.1	0.1	463.3	7.00		0.1	0.1	463.4	7.13
	0.1	0.1	463.4	6.94		0.1	0.1	463.5	7.11
	0.1	0.1	463.5	6.90		0.1	0.1	463.6	7.06
R5	0.1	0.1	463.6	6.84	B5	0.1	0.1	463.7	7.01
	0.1	-9.9	453.7	6.79		0.1	-9.9	453.8	6.95
	0.1	0.1	453.8	6.77		0.1	0.1	453.9	6.91
	0.1	0.1	453.9	6.72		0.1	0.1	454.0	6.85
	0.1	0.1	454.0	6.66		0.1	0.1	454.1	6.81
R6	0.1	0.1	454.1	6.62	B6	0.1	0.1	454.2	6.74

	0.1	-9.9	444.2	6.57		0.1	-9.9	444.3	6.70
	0.1	0.1	444.3	6.52		0.1	0.1	444.4	6.68
	0.1	0.1	444.4	6.49		0.1	0.1	444.5	6.61
	0.1	0.1	444.5	6.44		0.1	0.1	444.6	6.59
R7	0.1	0.1	444.6	6.39	B7	0.1	0.1	444.7	6.54
	0.2	-9.8	434.8	6.28		0.2	-9.8	434.9	6.42
	0.2	0.2	435.0	6.15		0.2	0.2	435.1	6.32
	0.2	0.2	435.2	6.03		0.2	0.2	435.3	6.22
	0.2	0.2	435.4	5.85		0.2	0.2	435.5	6.07
	0.2	0.2	435.6	5.61		0.2	0.2	435.7	5.92
R8	0.2	0.2	435.8	5.17	B8	0.2	0.2	435.9	5.67
	0.2	-9.8	426.0	4.36		0.2	-9.8	426.1	5.31
	0.2	0.2	426.2	4.01		0.2	0.2	426.3	4.55
	0.2	0.2	426.4	3.83		0.2	0.2	426.5	4.09
	0.2	0.2	426.6	3.71		0.2	0.2	426.7	3.88
	0.2	0.2	426.8	3.63		0.2	0.2	426.9	3.75
R9	0.2	0.2	427.0	3.57	B9	0.2	0.2	427.1	3.66
	0.2	-9.8	417.2	3.53		0.2	-9.8	417.3	3.59
	0.2	0.2	417.4	3.47		0.2	0.2	417.5	3.53
	0.2	0.2	417.6	3.43		0.2	0.2	417.7	3.48
	0.2	0.2	417.8	3.39		0.2	0.2	417.9	3.43
	0.2	0.2	418.0	3.36		0.2	0.2	418.1	3.40
R10	0.2	0.2	418.2	3.33	B10	0.2	0.2	418.3	3.38
	0.2	-9.8	408.4	3.30		0.2	-9.8	408.5	3.35
	0.2	0.2	408.6	3.28		0.2	0.2	408.7	3.31
	0.2	0.2	408.8	3.25		0.2	0.2	408.9	3.28
	0.2	0.2	409.0	3.23		0.2	0.2	409.1	3.26
	0.2	0.2	409.2	3.21		0.2	0.2	409.3	3.24
R11	0.2	0.2	409.4	3.19	B11	0.2	0.2	409.5	3.22
	0.2	-9.8	399.6	3.17		0.2	-9.8	399.7	3.20
	0.2	0.2	399.8	3.15		0.2	0.2	399.9	3.18
	0.2	0.2	400.0	3.14		0.2	0.2	400.1	3.16
	0.2	0.2	400.2	3.12		0.2	0.2	400.3	3.14
	0.2	0.2	400.4	3.10		0.2	0.2	400.5	3.12
R12	0.2	0.2	400.6	3.09	B12	0.2	0.2	400.7	3.12
	0.2	-9.8	390.8	3.06		0.2	-9.8	390.9	3.09
	0.2	0.2	391.0	3.05		0.2	0.2	391.1	3.07
	0.2	0.2	391.2	3.04		0.2	0.2	391.3	3.06
	0.2	0.2	391.4	3.03		0.2	0.2	391.5	3.04
	0.2	0.2	391.6	3.01		0.2	0.2	391.7	3.03

R13	0.2	0.2	391.8	3.00		B13	0.2	0.2	391.9	3.02
	0.5	-9.5	382.3	2.97			0.5	-9.5	382.4	3.00
	0.5	0.5	382.8	2.95			0.5	0.5	382.9	2.97
	0.5	0.5	383.3	2.93			0.5	0.5	383.4	2.94
	0.5	0.5	383.8	2.89			0.5	0.5	383.9	2.91
	0.5	0.5	384.3	2.90			0.5	0.5	384.4	2.90
R14	0.5	0.5	384.8	2.88		B14	0.5	0.5	384.9	2.88
	0.5	-9.5	375.3	2.86			0.5	-9.5	375.4	2.87
	0.5	0.5	375.8	2.84			0.5	0.5	375.9	2.84
	0.5	0.5	376.3	2.83			0.5	0.5	376.4	2.83
	0.5	0.5	376.8	2.81			0.5	0.5	376.9	2.81
	0.5	0.5	377.3	2.80			0.5	0.5	377.4	2.80
R15	0.5	0.5	377.8	2.78		B15	0.5	0.5	377.9	2.78
	0.8	-9.2	368.6	2.75			0.8	-9.2	368.7	2.75
	0.8	0.8	369.4	2.73			0.8	0.8	369.5	2.73
	0.8	0.8	370.2	2.71			0.8	0.8	370.3	2.71
	0.8	0.8	371.0	2.69			0.8	0.8	371.1	2.69
	0.8	0.8	371.8	2.67			0.8	0.8	371.9	2.67
R16	0.8	0.8	372.6	2.65		B16	0.8	0.8	372.7	2.65
	1	-9	363.6	2.63			1	-9	363.7	2.63
	1	1	364.6	2.61			1	1	364.7	2.61
	1	1	365.6	2.59			1	1	365.7	2.59
	1	1	366.6	2.57			1	1	366.7	2.58
	1	1	367.6	2.55			1	1	367.7	2.56
R17	1	1	368.6	2.53		B17	1	1	368.7	2.54
	1	-9	359.6	2.51			1	-9	359.7	2.52
	1	1	360.6	2.51			1	1	360.7	2.51
	1	1	361.6	2.49			1	1	361.7	2.49
	1	1	362.6	2.48			1	1	362.7	2.48
	1	1	363.6	2.46			1	1	363.7	2.47
R18	1	1	364.6	2.45		B18	1	1	364.7	2.45
	1.5	-8.5	356.1	2.43			1.5	-8.5	356.2	2.43
	1.5	1.5	357.6	2.41			1.5	1.5	357.7	2.42
	1.5	1.5	359.1	2.40			1.5	1.5	359.2	2.40
	1.5	1.5	360.6	2.38			1.5	1.5	360.7	2.38
R19	1.5	1.5	362.1	2.37		B19	1.5	1.5	362.2	2.37
	1.5	-8.5	353.6	2.34			1.5	-8.5	353.7	2.35
	1.5	1.5	355.1	2.33			1.5	1.5	355.2	2.34
	1.5	1.5	356.6	2.32			1.5	1.5	356.7	2.33
	1.5	1.5	358.1	2.31			1.5	1.5	358.2	2.31

	1.5	1.5	359.6	2.30		1.5	1.5	359.7	2.30
R20	1.5	1.5	361.1	2.28	B20	1.5	1.5	361.2	2.28
	2	-8	353.1	2.25		2	-8	353.2	2.26
	2	2	355.1	2.25		2	2	355.2	2.25
	3	3	358.1	2.23		3	3	358.2	2.23
	3	3	361.1	2.21		3	3	361.2	2.21
* clear	3	3	364.1	2.20	* clear	3	3	364.2	2.19
R21	3	3	367.1	2.18	B21	3	3	367.2	2.17
	3	-7	360.1	2.15		3	-7	360.2	2.17
	3	3	363.1	2.15		3	3	363.2	2.16
	3	3	366.1	2.13		3	3	366.2	2.14
	3	3	369.1	2.11		3	3	369.2	2.12
	3	3	372.1	2.10		3	3	372.2	2.10
R22	3	3	375.1	2.09	B22	3	3	375.2	2.09
	3.5	-6.5	368.6	2.05		3.5	-6.5	368.7	2.07
	3.5	3.5	372.1	2.05		3.5	3.5	372.2	2.05
	3.5	3.5	375.6	2.03		3.5	3.5	375.7	2.04
	4	4	379.6	2.01		4	4	379.7	2.02
	4	4	383.6	2.00		4	4	383.7	2.01
R23	4	4	387.6	1.99	B23	4	4	387.7	1.99
	4.5	-5.5	382.1	1.98		4.5	-5.5	382.2	1.98
	4.5	4.5	386.6	1.97		4.5	4.5	386.7	1.97
	5	5	391.6	1.95		5	5	391.7	1.96
	5	5	396.6	1.94		5	5	396.7	1.94
	5	5	401.6	1.93		5	5	401.7	1.93
R24	5	5	406.6	1.91	B24	5	5	406.7	1.91
	10	0	406.6	1.88		10	0	406.7	1.88
	10	10	416.6	1.86		10	10	416.7	1.87
	10	10	426.6	1.85		10	10	426.7	1.85
	10	10	436.6	1.83		10	10	436.7	1.83
	10	10	446.6	1.81		10	10	446.7	1.81
R25	10	10	456.6	1.79	B25	10	10	456.7	1.80
		-10	446.6				-10	446.7	

* The 10 mL sample is included in the row below each sample point to indicate that the volume decreased after every sampling event

Table A3.9: Pb Concentration Results for Titration Test 1 (pH 7 -> 1)

Sample Results for Titration Test 1 (pH 7 -> 1)

Sample	pH	FAA sample volume (mL)	Absorption	Std. Dev	Conc of sample (mg/L)	Std. Dev
R1	10.00	501.1	0.015	0.007	8.94	3.74
R2	9.37	491.7	0.014	0.008	8.72	4.76
R3	7.73	482.3	0.031	0.006	19.2	3.37
R4	7.22	472.9	0.043	0.006	27.2	3.17
R5	6.84	463.6	0.038	0.007	24.0	4.17
R6	6.62	454.1	0.035	0.007	22.0	4.17
R7	6.39	444.6	0.019	0.007	11.7	3.80
R8	5.17	435.8	0.019	0.006	11.5	3.44
R9	3.57	427.0	0.020	0.005	12.6	2.92
R10	3.33	418.2	0.021	0.005	13.0	2.94
R11	3.19	409.4	0.022	0.005	13.9	2.71
R12	3.09	400.6	0.025	0.006	15.4	3.22
R13	3.00	391.8	0.026	0.006	16.2	3.29
R14	2.88	384.8	0.026	0.007	16.2	3.70
R15	2.78	377.8	0.028	0.007	17.3	4.24
R16	2.65	372.6	0.035	0.006	21.8	3.22
R17	2.53	368.6	0.046	0.005	29.1	2.94
R18	2.45	364.6	0.054	0.006	34.5	3.19
R19	2.37	362.1	0.068	0.005	43.5	2.66
R20	2.28	361.1	0.097	0.007	61.9	4.23
R21	2.18	367.1	0.101	0.006	64.5	3.29
R22	2.09	375.1	0.098	0.005	62.6	2.94
R23	1.99	387.6	0.095	0.006	60.9	3.51
R24	1.91	406.6	0.090	0.007	57.6	4.26
R25	1.79	456.6	0.083	0.006	52.9	3.67
B1	10.13	501.2	0.033	0.007	21.0	4.03
B2	9.77	491.8	0.031	0.007	19.7	3.74
B3	8.41	482.4	0.029	0.007	17.9	4.03
B4	7.43	473.0	0.029	0.007	18.4	4.26
B5	7.01	463.7				
B6	6.74	454.2	0.029	0.007	17.9	3.71
B7	6.54	444.7	0.026	0.007	16.0	3.97
B8	5.67	435.9	0.028	0.008	17.5	4.75
B9	3.66	427.1	0.027	0.008	17.1	4.79

B10	3.38	418.3	0.028	0.007	17.5	3.80
B11	3.22	409.5	0.027	0.007	16.9	3.99
B12	3.12	400.7	0.028	0.008	17.7	4.79
B13	3.02	391.9	0.029	0.010	18.2	5.80
B14	2.88	384.9	0.034	0.007	21.6	3.97
B15	2.78	377.9	0.035	0.006	21.8	3.19
B16	2.65	372.7	0.041	0.009	25.7	5.28
B17	2.54	368.7	0.046	0.006	29.1	3.17
B18	2.45	364.7	0.058	0.006	36.8	3.46
B19	2.37	362.2	0.092	0.007	58.7	3.99
B20	2.28	361.2	0.095	0.007	60.4	4.09
B21	2.17	367.2	0.105	0.008	66.9	4.60
B22	2.09	375.2	0.105	0.006	67.1	3.17
B23	1.99	387.7	0.100	0.007	63.6	3.71
B24	1.91	406.7	0.095	0.007	60.6	3.99
B25	1.80	456.7	0.082	0.009	52.5	5.07

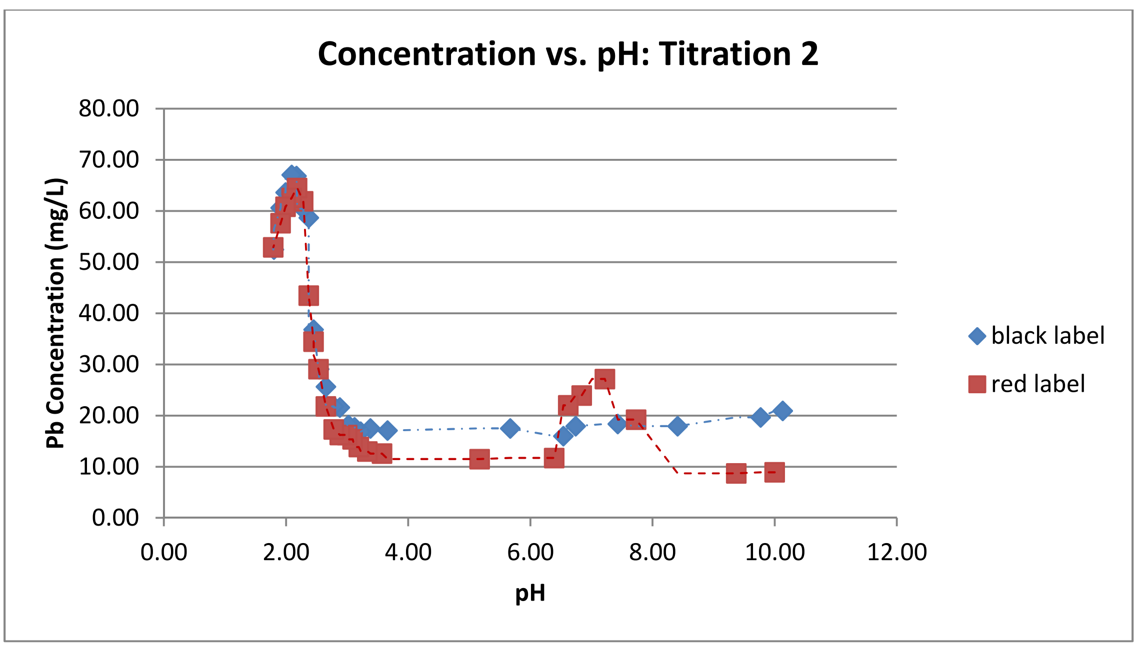

Figure A3.6: Change in Pb Concentration – Titration Test 1 (pH 7 -> 1)

Table A3.10: Titration Test 2 (pH 7 -> 1) - FAA

Titration Test 2 (pH 7 -> 1)

	Sample	Absorbance			Average	Std. Dev
		Trial 1	Trial 2	Trial 3		
Calibration Standard	1% HCl	0.000	0.009	0.005	0.005	0.004
	5 ppm	0.004	0.016	0.011	0.010	0.005
	10 ppm	0.015	0.024	0.020	0.020	0.004
	20 ppm	0.029	0.041	0.036	0.035	0.005
	50 ppm	0.079	0.091	0.085	0.085	0.005
	100 ppm	0.157	0.171	0.162	0.163	0.006
Control	1% HCl	0.000	0.009	0.004	0.004	0.004
	10 ppm	0.015	0.025	0.020	0.020	0.004
Samples	R1	0.000	0.012	0.006	0.006	0.005
	R2	0.000	0.014	0.007	0.007	0.006
	R3	0.013	0.021	0.016	0.017	0.003
	R4	0.000	0.012	0.006	0.006	0.005
	R5	0.000	0.015	0.009	0.008	0.006
Control	1% HCl	0.000	0.010	0.004	0.005	0.004
	10 ppm	0.015	0.024	0.019	0.019	0.004
Samples	R6	0.000	0.013	0.007	0.007	0.005
	R7	0.003	0.014	0.008	0.008	0.004
	R8	0.005	0.020	0.017	0.014	0.006
	R9	0.029	0.040	0.038	0.036	0.005
	R10	0.041	0.052	0.047	0.047	0.004
Control	1% HCl	0.000	0.010	0.004	0.005	0.004
	10 ppm	0.014	0.026	0.020	0.020	0.005
Samples	R11	0.091	0.106	0.098	0.098	0.006
	R12	0.138	0.152	0.147	0.146	0.006
	R13	0.133	0.153	0.144	0.143	0.008
	R14	0.125	0.138	0.136	0.133	0.006
Control	1% HCl	0.000	0.010	0.005	0.005	0.004
	10 ppm	0.013	0.024	0.019	0.019	0.004
Samples	B1	0.000	0.012	0.006	0.006	0.005
	B2	0.000	0.010	0.006	0.005	0.004
	B3	0.000	0.012	0.007	0.006	0.005
	B4	0.000	0.011	0.007	0.006	0.005
	B5	0.000	0.011	0.006	0.006	0.004
Control	1% HCl	0.000	0.009	0.005	0.005	0.004
	10 ppm	0.016	0.024	0.018	0.019	0.003

Samples	B6	0.000	0.013	0.007	0.007	0.005
	B7	0.001	0.015	0.008	0.008	0.006
	B8	0.006	0.022	0.014	0.014	0.007
	B9	0.021	0.033	0.027	0.027	0.005
	B10	0.038	0.051	0.046	0.045	0.005
Control	1% HCl	0.000	0.009	0.004	0.004	0.004
	10 ppm	0.014	0.023	0.019	0.019	0.004
Samples	B11	0.080	0.098	0.090	0.089	0.007
	B12	0.138	0.152	0.146	0.145	0.006
	B13	0.135	0.150	0.144	0.143	0.006
	B14	0.128	0.141	0.134	0.134	0.005
Control	1% HCl	0.000	0.012	0.004	0.005	0.005
	10 ppm	0.015	0.025	0.020	0.020	0.004

Calibration Curve	
Absorption	Concentration (mg/L)
0.010	5
0.020	10
0.035	20
0.085	50
0.163	100

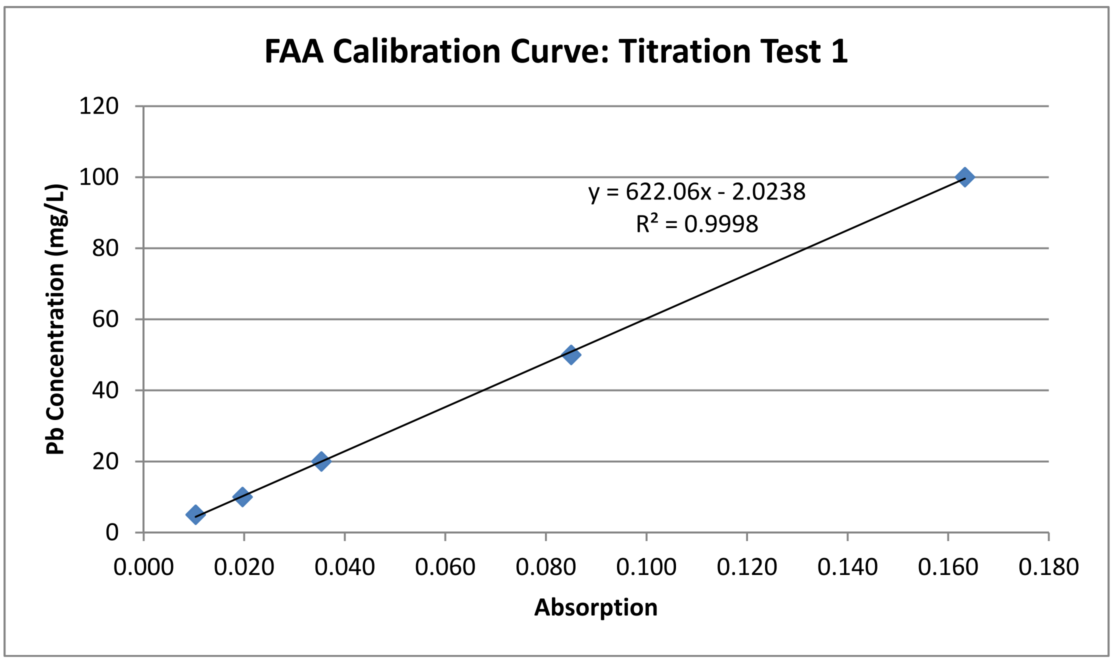

Figure A3.7: FAA Calibration Curve – Titration Test 1 (pH 1 -> 7)

Table A3.11: Volume addition and pH for Titration Test 2 (pH 7 -> 1)

Titration 1.1: Red labels					Titration 1.2: Black labels			
Sample	Δ V (mL)	Volume (mL)	pH		Sample	Δ V (mL)	Volume (mL)	pH
	0	342.4				0	342.4	
R1	3	345.4	6.74		B1	2	344.4	6.72
R2	-9.8	335.6	6.67		B2	-9.8	334.6	6.66
R3	-8.5	327.1	6.08		B3	-8.5	326.1	6.05
R4	-9.3	317.8	5.22		B4	-9.3	316.8	5.10
R5	-9.75	308.1	3.94		B5	-9.75	307.1	3.85
R6	-9.6	298.5	3.38		B6	-9.6	297.5	3.34
R7	-9.2	289.3	2.98		B7	-9.2	288.3	2.97
R8	-8.4	280.9	2.62		B8	-8.4	279.9	2.63
R9	-7.6	273.3	2.34		B9	-7.6	272.3	2.35
R10	-8	265.3	2.19		B10	-8	264.3	2.20
R11	-6	259.3	1.98		B11	-6	258.3	2.00
R12	-5	254.3	1.82		B12	-5	253.3	1.84
R13	0	254.3	1.61		B13	0	253.3	1.62
R14	6	260.3	1.40		B14	6	259.3	1.42
	-10	250.3				-10	249.3	

* The 10 mL sample is included in the row below each sample point to indicate that the volume decreased after every sampling event

Table A3.12: Pb Concentration Results for Titration Test 2 (pH 7 -> 1)

Sample Results for Titration Test 2 (pH 7 -> 1)

Sample	pH	FAA sample volume (mL)	Absorption	Std. Dev	Conc of sample (mg/L)	Std. Dev
R1	6.74	345.4	0.006	0.005	1.71	1.02
R2	6.67	335.6	0.007	0.006	2.33	1.53
R3	6.08	327.1	0.017	0.003	8.34	0.029
R4	5.22	317.8	0.006	0.005	1.71	1.02
R5	3.94	308.1	0.008	0.006	2.95	1.81
R6	3.38	298.5	0.007	0.005	2.12	1.28
R7	2.98	289.3	0.008	0.004	3.16	0.774
R8	2.62	280.9	0.014	0.006	6.69	2.01
R9	2.34	273.3	0.036	0.005	20.2	0.95
R10	2.19	265.3	0.047	0.004	27.0	0.774
R11	1.98	259.3	0.098	0.006	59.1	1.79
R12	1.82	254.3	0.146	0.006	88.6	1.58
R13	1.61	254.3	0.143	0.008	87.1	3.06
R14	1.40	260.3	0.133	0.006	80.7	1.53
B1	6.72	344.4	0.006	0.005	1.71	1.02
B2	6.66	334.6	0.005	0.004	1.29	0.533
B3	6.05	326.1	0.006	0.005	1.92	1.04
B4	5.10	316.8	0.006	0.005	1.71	0.804
B5	3.85	307.1	0.006	0.004	1.50	0.774
B6	3.34	297.5	0.007	0.005	2.12	1.28
B7	2.97	288.3	0.008	0.006	2.95	1.53
B8	2.63	279.9	0.014	0.007	6.69	2.04
B9	2.35	272.3	0.027	0.005	14.8	1.02
B10	2.20	264.3	0.045	0.005	26.0	1.31
B11	2.00	258.3	0.089	0.007	53.5	2.56
B12	1.84	253.3	0.145	0.006	88.4	1.54
B13	1.62	253.3	0.143	0.006	86.9	1.81
B14	1.42	259.3	0.134	0.005	81.5	1.28

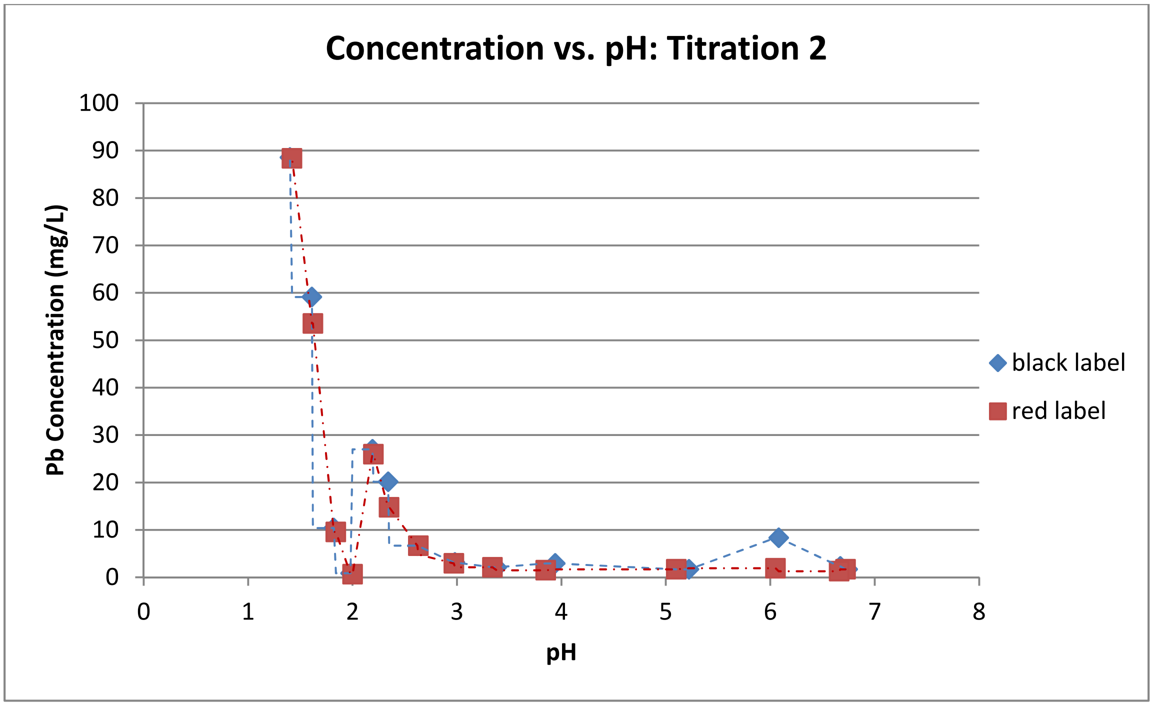

Figure A3.8: Change in Pb Concentration – Titration Test 2 (pH 1 -> 7)

Table A3.13: Titration Pb Concentration Data (pH 1 -> 7)

Titration Test pt 1 (low -> neutral)					
Sample	pH	Conc of sample (mg/L)			
B1 T2	1.52				112.16
R1 T2	1.52			109.78	
B2 T2	1.60				112.38
R2 T2	1.60			108.04	
R3 T2	1.65			109.34	
B3 T2	1.68				110.21
R4 T2	1.73			108.48	
R1 T1	1.74		73.26		
B1 T1	1.74	74.25			
B4 T2	1.74				109.34
R5 T2	1.80			108.04	
B2 T1	1.81	86.60			
B5 T2	1.81				109.78
R2 T1	1.82		108.35		
R6 T2	1.85			108.48	
B6 T2	1.88				111.29
R3 T1	1.91		94.02		
B3 T1	1.91	76.72			
R7 T2	1.92			108.26	
B7 T2	1.94				109.56
R4 T1	1.97		111.81		
B4 T1	1.97	55.97			
R8 T2	1.98			107.82	
B8 T2	2.03				71.61
B5 T1	2.06	63.87			
R5 T1	2.07		116.75		
R9 T2	2.09			55.13	
B9 T2	2.12				52.53
R10 T2	2.16			42.33	
B6 T1	2.20	68.32			
B10 T2	2.20				37.35
R6 T1	2.22		73.26		
R11 T2	2.32			23.47	
B11 T2	2.39				21.30
R12 T2	2.59			10.89	
R7 T1	2.60		51.02		
B7 T1	2.63	44.60			
B12 T2	2.68				11.32
R13 T2	2.92			5.25	

B13 T2	3.06				8.07
R14 T2	3.47			3.08	
B14 T2	4.84				5.90
R8 T1	5.93		28.29		
R15 T2	5.98			3.52	
B8 T1	6.24	23.35			
B15 T2	6.26				5.47
R9 T1	6.96		25.33		
B9 T1	7.20	26.32			

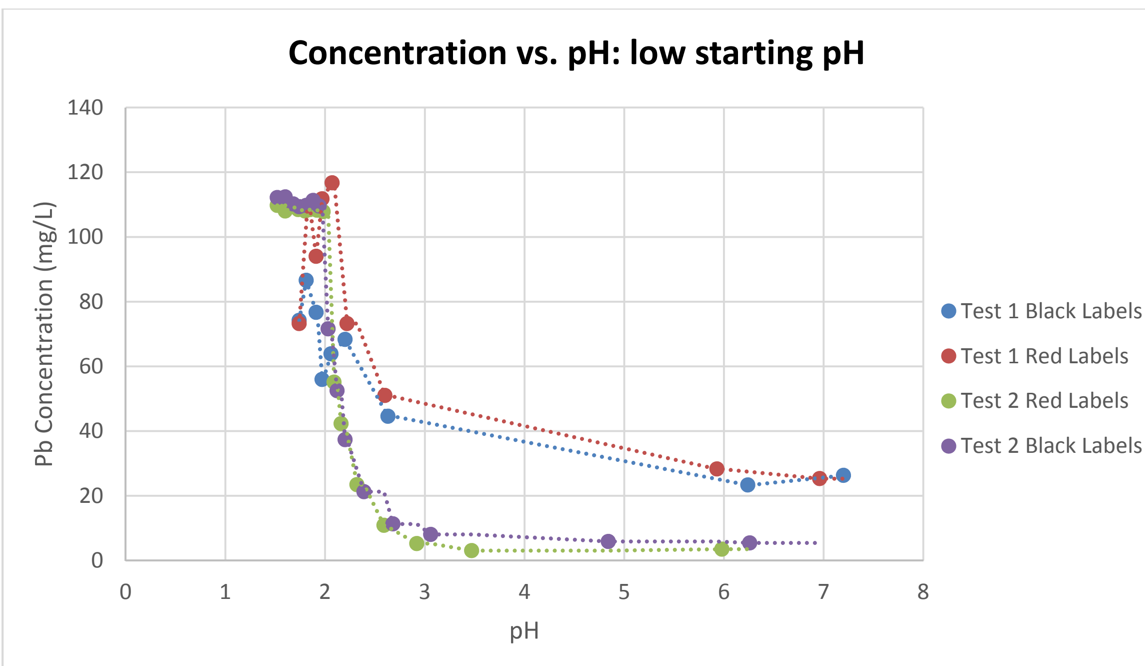

Figure A3.9: Pb Concentration – Titration Tests (pH 1 -> 7)

Table A3.14: Titration Pb Concentration Data (pH 7 -> 1)

Titration Test pt 2 (neutral -> low)					
Sample	pH	Conc of sample (mg/L)			
B1 T1	10.13		20.95		
R1 T1	10.00	8.94			
B2 T1	9.77		19.66		
R2 T1	9.37	8.72			
B3 T1	8.41		17.95		
R3 T1	7.73	19.23			
B4 T1	7.43		18.38		
R4 T1	7.22	27.17			
B5 T1	7.01				
R5 T1	6.84	23.96			
B6 T1	6.74		17.95		
R1 T2	6.74			1.71	
B1 T2	6.72				1.71
R2 T2	6.67			2.33	
B2 T2	6.66				1.29
R6 T1	6.62	22.02			
B7 T1	6.54		16.02		
R7 T1	6.39	11.73			
R3 T2	6.08			8.34	
B3 T2	6.05				1.92
B8 T1	5.67		17.52		
R4 T2	5.22			1.71	
R8 T1	5.17	11.51			
B4 T2	5.10				1.71
R5 T2	3.94			2.95	
B5 T2	3.85				1.50
B9 T1	3.66		17.09		
R9 T1	3.57	12.58			
B10 T1	3.38		17.52		
R6 T2	3.38			2.12	
B6 T2	3.34				2.12
R10 T1	3.33	13.01			
B11 T1	3.22		16.87		
R11 T1	3.19	13.87			
B12 T1	3.12		17.73		
R12 T1	3.09	15.37			
B13 T1	3.02		18.16		
R13 T1	3.00	16.23			
R7 T2	2.98			3.16	

B7 T2	2.97				2.95
R14 T1	2.88	16.23			
B14 T1	2.88		21.59		
R15 T1	2.78	17.30			
B15 T1	2.78		21.81		
R16 T1	2.65	21.81			
B16 T1	2.65		25.67		
B8 T2	2.63				6.69
R8 T2	2.62			6.69	
B17 T1	2.54		29.10		
R17 T1	2.53	29.10			
R18 T1	2.45	34.47			
B18 T1	2.45		36.83		
R19 T1	2.37	43.48			
B19 T1	2.37		58.71		
B9 T2	2.35				14.77
R9 T2	2.34			20.16	
R20 T1	2.28	61.93			
B20 T1	2.28		60.43		
B10 T2	2.20				25.97
R10 T2	2.19			27.01	
R21 T1	2.18	64.51			
B21 T1	2.17		66.87		
R22 T1	2.09	62.58			
B22 T1	2.09		67.08		
B11 T2	2.00				53.55
R23 T1	1.99	60.86			
B23 T1	1.99		63.65		
R11 T2	1.98			59.15	
R24 T1	1.91	57.64			
B24 T1	1.91		60.64		
B12 T2	1.84				88.38
R12 T2	1.82			88.59	
B25 T1	1.80		52.49		
R25 T1	1.79	52.92			
B13 T2	1.62				86.93
R13 T2	1.61			87.14	
B14 T2	1.42				81.54
R14 T2	1.40			80.71	

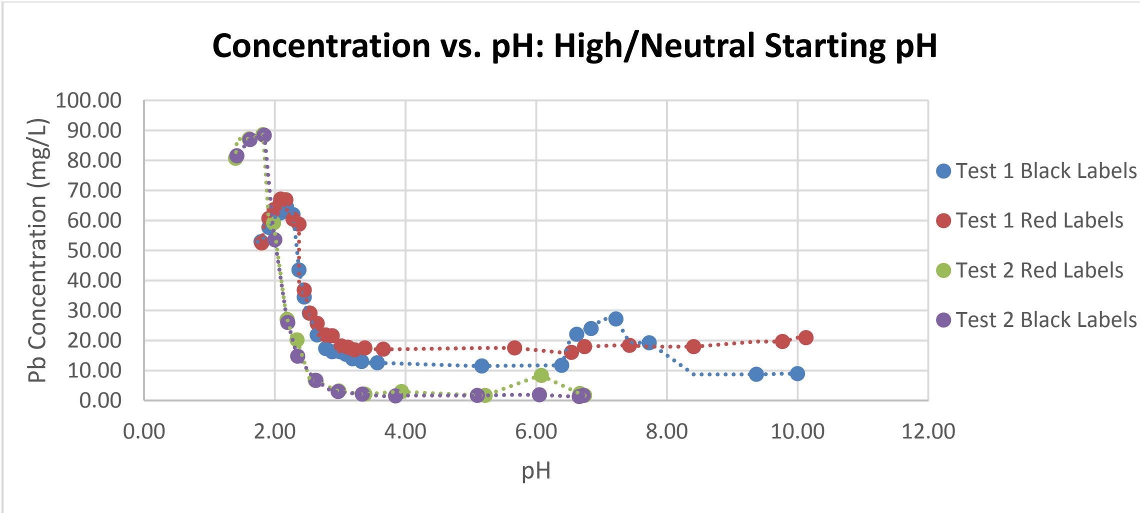

Figure A3.10: Pb Concentration – Titration Tests (pH 7 -> 1)

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