I: Introduction

This experiment involves the analysis of our lead samples in the Graphite Furnace Atomic Absorption (GFAA) apparatus. Each person's lead solution was diluted to 50 ml in the aa solution, and then injected into the graphite furnace. Initially, this involves raising the temperature to 120° for 25 seconds to the evaporate the water from each sample. Next the sample is ashed by raising the temperature to 700° for 25 seconds. Lastly, the temperature is raised to 2300° for 7 seconds to atomize the sample and acquire the absorbance. Each absorbance was acquired, and the data was to be graphed and analyzed.

II: Methods and Materials

The materials needed for the completion of this lab are each members lead samples, and the aa solution which consisted of HNO_3, $NH_4H_2PO_4$, and $Mg(NO_3)_2$. Each person in the group was to add a variable amount of their lead solution to be diluted to 50 ml with the aa solution. This was dependent on the relative amount of lead in each member's sample, and the amount added ranged from 1000 to 2000 µL. Each lead sample (five for each person, so a total of 25 for the whole group), was to be analyzed by the Perkin- Elmer HGA 400 Control Box with the Perkin- Elmer 5000 Atomic Absorption Spectrophotometer, with each requiring between 3 and 5 trials. These trials needed to be run until at least three absorbencies came in close proximity to each other. The absorbance found for each trial was then recorded , along with each blank run. The settings for these trials on the GFAA are as follows,

Lamp number 2

I= 10 mA

Wavelength= 283.3 nm

Time= 7 seconds

Energy= 62

Slit Width= H 0.7 nm

Program= 1A

III: Results and Data

The standard deviation of our injection technique of the blank and standards are:

Blank; 0.048 +/- 0.005

0.025 ppm Lead Standard; 0.158 +/- 0.003

0.050 ppm Lead Standard; 0.313 +/- 0.014

0.075 ppm Lead Standard; 0.394 +/- 0.022

0.100 ppm Lead Standard; 0.512 +/- 0.024

Sample Turn Around Time: Two minutes

The concentration of lead in each of our individual samples subtracting out the baseline (concentration of the blank) and using b= 2, ε= 597, 717 and the related absorbancies for each trial were found to be:

Sample Calculation using calibration curve I:

ACM #3 ave. absorbance = 0.1513 = 1.717x + 0.101. x = 0.0293 ppm.

[0.0293ppm x 5000(dilution factor for 1 in 50)]/1.0g soil = 146.5 ppm.

Nick Bretl:

Sample 4: 75.7 ppm

Kevin Hudziak:

Sample 4: 1171.5 ppm

Stacy Mraz:

Sample 3: 146.5 ppm

Dan DeMarah:

Sample 3: 0.0 ppm

[redacted]

Using calibration curve II:

Sample 1: 243.5 ppm

Sample 2: 168.6 ppm

Sample 3: 175.5 ppm

Sample 4: 473.0 ppm **spiked**

Sample 5: 555.0 ppm **spiked**

Mike's samples show a definite difference between spiked and unspiked soil samples. The other four group member's samples were redone and were all unspiked soil only samples. Our raw data is attached. All samples were 1 mL diluted to 50 mL in a volumetric flask.

LOD:

Limit of Detection= 0.51 +/ - 0.03 ppm

LOQ:

Limit of Quantification= 0.51 +/ - 0.09 ppm

R:

R= 0.9962

While analyzing the data we realized that there is a great deal of difference between the LOD corrected to the soil and the cutoff between what is considered to be contaminated versus uncontaminated soil. The EPA(Environmental Protection Agency) has deemed soil that is 250 ppm to be at the threshold of what is considered contaminated soil. Obviously 0.51 +/ - 0.03 ppm lead lies a great deal from 250 ppm. In fact the EPA determined threshold is almost 500 times greater than the Limit of Detection of GFAA. Yet the LOD depends on the injection technique into the graphite furnace. After a little analysis the numbers reveal that the injection technique has little effect on the Limit of Detection. Therefore we can say with confidence that the LOD is as stands.

During the analysis process we noticed an amount of variation in the amounts of lead being detected in each sample. For example, Kevins' concentration values differed greatly from one sample to the next. One

explanation for the variation lies in the inherent error of the GFAA process. However, another plausible explanation exists. The differing lead compounds in the soil give rise to the possibility to different valance states of lead. Each valance carries a different ionization/ atomization energy. Therefore if the temperature of the machine is not great enough not all of the higher energy lead will atomize. Since it doesn't atomize it can't absorb, giving rise to the differences in the lead concentrations. However, the $Pb^{+} \rightarrow Pb^{+2} + e$ reaction doesn't effect our analysis.

From the data given in table 10- 3 of our text book, we look at Barium's ionization potential at 2000K and notice that only 0. 0006 of the barium present would be ionized at this temperature. We mention barium's data, it is the closest, in terms of ionization potential, to Pb given on table 10- 3. Since the graphite furnace only reaches 2300° C, we believe that not enough Pb would have been ionized to alter the accuracy of the results. As well, lead could not be detected by a flame test.

Upon calculations , we discovered that a flame temperature of 5083.33 K would be needed to stimulate lead to the point of light emission. Normal flame temperature only reaches 1900° C (or 2173 K), a flame test clearly fails to detect lead.

Background absorbance interferes with the best possible detection of lead absorbance in the samples. However, there is a means to determine the background absorbance. It can be done working within the configurations of instrument as is, in the lab.

To determine the background absorbance, we would have to look at our sample data and determine the amount of lead " supposedly" detected. Taking these calculated amounts of lead from out samples we could run the machine with these specific quantities of pure lead and compare these new results with the results from our samples. Theoretically they should match. However, any difference we calculate can be attributed to background absorbance.

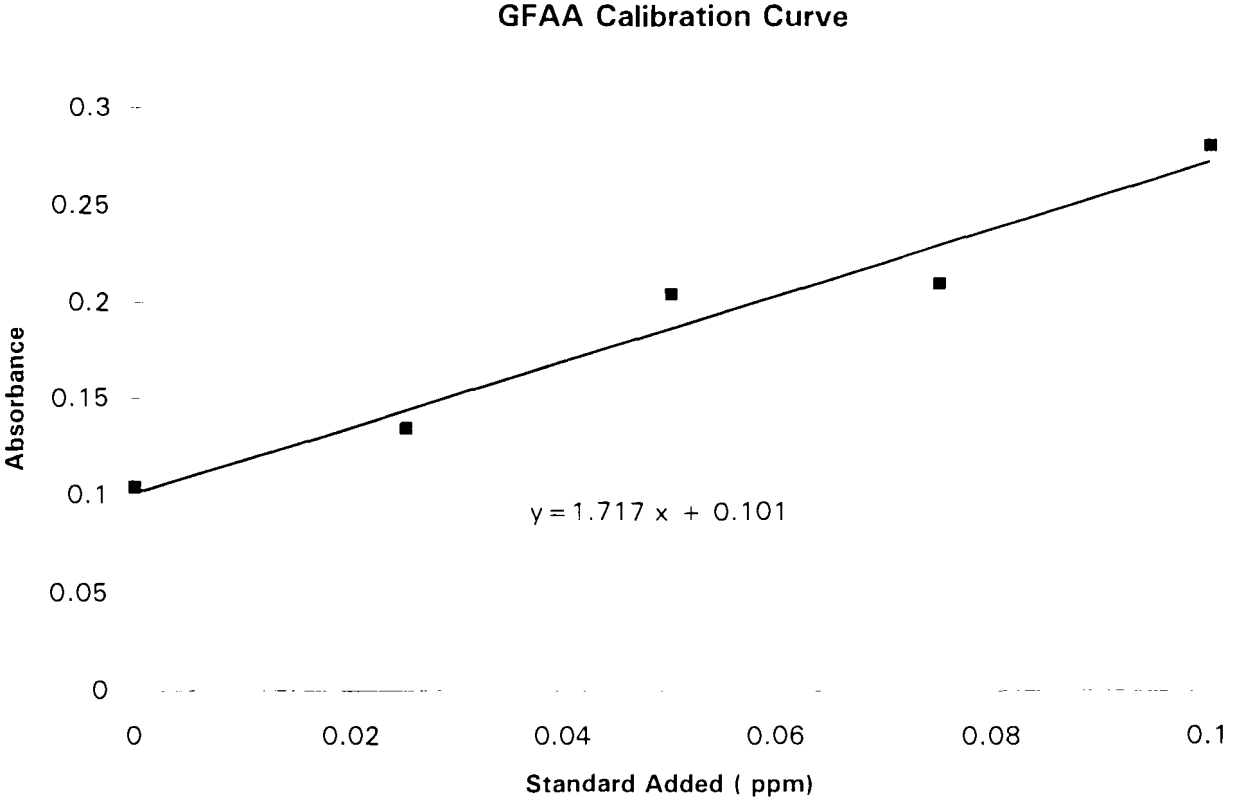
We added Nitric Acid to our samples for one specific reason, because it electrostatically interacts with the lead in the beginning phases of the FAA process. Most of the lead in our samples comes in a valence state of Pb^{+2}. The

HNO_3 therefore dissociates in the aa and the NO_3^- bonds electrostatically with the lead to keep it stabilized through the drying and into the ashing processes. This is the reason the aa matrix is used for this experiment. The aa, with the nitric and ammonium phosphite, keeps the lead stabilized until it reaches the point at which it can be atomized for absorption spectroscopy.

Assuming that there is lead in all of our samples , we do follow certain hazardous disposal procedures. These procedures involve the placing of waste into waste containers and not into the sink. Afterwards, we must rinse our sample glassware with EDTA, followed with a deionized H_2O rinse. Lastly, the contents of the waste containers must be prepared for disposal by trained experts.

This method of lead detection is fairly easy to instruct others with. Configuration of the instrumental variables can be as easy as following a list of simple written instructions that involve primarily pushing buttons. As for putting the sample into the graphite furnace , some difficulty may arise. However, if one remembers a few points, one should have little trouble. For example, make sure to actually deposit the sample into the small hole in the tube; however, do not jam the tip of the ependorf all the way into the tube. Obviously, these simple require low skills and don't require an extremely skilled technician.

The idea of a paper trail being adapted to the graphite furnace appears to be an unpractical application. In our experiment we had only one wavelength of light exposed to our sample and subsequently only one absorbance. Were talking about a one point graph. We believe looking at the absorbance data collectively on a table would be the only useful way to manipulate data. Therefore, we wouldn' t bother to create a paper trail.


GFAA Calibration Curve
0.3
0.25
0.2
0.15
0.1
0.05
0
Absorbance
y = 1.717 x + 0.101
0
0.02
0.04
0.06
0.08
0.1
Standard Added (ppm)

Absorbance vs. Concentration (Calibration Curve for GFAA)