The Wondrous Graphite Furnace

I. Purpose.

We shall analyze the lead in our liquid samples by drying and atomizing our samples, and then probing the atom vapor with a monochromatic beam of light to excite valence electrons in Pb. We shall also perform a background correction (depending on the instrument used).

II. Materials/Methods.

For a full explanation of the procedure please look at lab manual (p410 to p413)

Solutions used: Stock solution of: 0.2% HNO_3, 0.2 mg $NH_4H_2PO_4$, and .01mg $Mg(NO_3)_2$ per injection. Also make a 1L solution by adding 10g of $NH_4H_2PO_4$, 0.5g of $Mg(NO_3)_2$, and 2.86mL of HNO_3 and diluting to 1L with de-ionized water.

III. Data/Results.
Calibration curve.
See appendix 1 for the calibration curve.
Calibration curve data:

Sample	Abs.	Abs.	Abs.	Abs.	Abs.	Abs.
Trial #	1	2	3	4	5	6
Blank*	.058	.072	.059	.042	.037	.042
0.025ppm	.154	.160	.161	.156	-----	-----
0.050ppm	.308	.298	.332	.313	-----	-----
0.075ppm	.403	.384	.421	.369	-----	-----
0.100ppm	.521	.527	.488	.539	.485	.458

*Note: we used our own blank average for our unknown samples.

LOD & LOQ
Using the formula: $\bar{x} + 3\sigma$ for LOD, and after using the standard error bar (*appendix 1*),

LOD=**.32025.** And with x̄ + 9σ for LOQ, and again using the standard error bars, LOQ=**.86025.**

Results:

Sample*	Avg. Absorbance	ppm Pb in sample	ppm Pb in soil
Alex 1	0.5848	0.1145	572.5
Alex 2	0.5948	0.1167	583.5
Alex 3	0.5661	0.1105	552.5
Alex 4	0.552	0.1075	537.5
Alex 5	0.5618	0.1096	548
Jeff 1	0.1072	0.01159	57.95
Jeff 2	0.1131	0.01287	64.33
Jeff 3	0.0936	0.00866	43.3
Jeff 4	0.0892	0.00772	38.6
Jeff 5	0.0894	0.00776	38.8
Faiz 1	0.1794	0.02716	135.8
Faiz 2	0.1848	0.02832	141.6
Faiz 3	0.1398	0.01862	93.1
Faiz 4	0.151	0.02103	105.15
Faiz 5	0.15	0.02082	104.1

*Samples 1 and 2 are spiked; samples 3, 4, and 5 are unspiked.

x̄

Stdev. for blank runs : .05207 +- .009945

Sample calculations.
ppm Pb in sample is obtained from the calibration curve.
ppm Pb in soil = ppm Pb in sample * dilution coefficient * digestion coefficient
= ppm Pb in sample * (50 g sample/1 g digest) * (100 g digest / 1 g soil) = 5000 * ppm Pb in sample

IV. Discussion.

With our particular data and circumstances, we absolutely love the graphite furnace technique. The techniques and methods involved in this procedure are very simple to understand, when one is given direction (after all, three idiots like Faiz, Alex and Jeff figured it out!). And performing a paper trail is not even that bad either, compared to some other methods. This technique is so good, because our data shows us that we can measure our quality control samples.

When performing a lead analysis with a graphite furnace, the calibration curve obtained should have an R^2 value pretty close to unity. This lets us know that the concentration data that we obtain is reliable and also that the graphite furnace technique on the whole also a good method for lead detection. An R^2 value such as 0.9928, which happens to be the number that we received, lets us know that our concentrations for lead are indeed reliable. Also important when running such an analysis, is that our internal error (i.e. the stdev between our blank runs) is low. A low standard deviation, shows us that our operator error is not contributing greatly to the total error incurred in the analysis, because operator error along with scattering are two of the main sources of instrumental error in this analysis. As expected, variation can creep in from all sources; so when we consider variation in our measurements we have to take into account errors in the digestion process, and that there may be different types of Pb present in the soil that we collected. While still on the subject of variance and error, background absorbance can also cause a problem in our measurements. We can account for what this background absorbance is, by subtracting off the blank absorbance taken prior to the particular run in question.

Using a flame to excite lead to cause emission would be a bad idea for basically two reasons. Firstly, we feel that for the wavelength line that we wish to monitor for lead, cannot be successfully excited in regular flame temperatures. Secondly, if the flame were to get that high in temperature, the flame does not heat in a constant way at the same high speeds as a graphite furnace. The flame would also have hot and cold pockets that would cause some problems in the decomposition of the sample (some sample would decompose, whereas other sample may remain intact).

The choice of HNO_3 is an extremely crucial variable in this analysis. Moreover, the nitrate can be decomposed to lead oxide and nitrous gas , followed shortly by the production of the lead atom phase. Nitrates also decompose at low temperatures, which allows for the removal of constituents and transformations, as opposed to formation of gas phase species. This has certain ramifications for the choice of our matrix, in that we must choose a matrix containing HNO_3 so that the matrix does not volatilize causing an underestimation of the sample present.

The turn around time for the graphite furnace technique is quite efficient, and we know this because after injecting the sample into the furnace, it took only a matter of seconds to obtain an absorbance measurement.

As with any experiment involving toxic reagents, one must take extreme care when disposing the reagents. That is why after this experiment, our group disposed of the lead waste and all other hazardous chemicals in an appropriate disposal bottle. Alluding to this last part on hazardous chemicals and pollutants, lead often contaminates tap water and

must be removed by suitable means. One possible way of removing the polluted tap water, would be to add barley to remove the lead contaminant. The barley would extract the lead, by complexing the lead, and the subsequent barley-lead complex molecules would be large enough to be filtered out of the tap water.

Individual Responsibilities

Injector: Alex _____

Sample preparation: Jeff _____

Data Collection & Writing/Typing report: Faiz _____

Absorbance vs. Concentration of Pb

$y = 4.64x + 0.0534$

$R^2 = 0.9928$

Absorbance

Concentration (ppm)

◆ Absorbance

── Linear (Absorbance)