

Experiment 25: ICP-MS Determination of Pb

I. Introduction:

In this lab we are to use the digested baby wipes from the students of a local area school. From these wipes we tried to determine the lead in the wipes using the ICP-MS. The lead cations are sent to a mass spectrometer at low pressure (via pressure "locks") to a quadrupole mass spectrometer analyzer. The lead is then gated (selected) from arriving at the detector by application of a combined AC/DC voltage across four electrodes. The detector is a single ion counter and is capable of absolute (no blank, instrumental noise) detection limits of pptillion.

II. Materials and Methods:

Our materials consisted of our digested baby wipes, 0.1% nitric acid, and lead standards. The standards were 10, 50, 100, and 200 ppb for lead and internal standards included 50 ppb of Th, V, and Sb.

The instrument was the ICP-MS, but due to time constraints and instrumental problems we also used the GFAA. The lead standards for lead remained the same, however, we did not need the internal standards of Th, V, and Sb.

III. Results:

David and I were the first group to run the ICP-MS. We were not sure of the calibration on detection of the instrument. We diluted our lead by 2000 times, we did this because we did not want to blow out the detector of the ICP-MS by having a too high concentration of lead. Our initial results were basically useless because our sample were too diluted. David and I were also unable to perform the experiment again due to time constraints and conflicting schedules. When we did have time to run the experiment over again, we were not able to use the ICP-MS because the instrument was not available for use. Therefore, we used the GFAA to determine our lead concentration.

Table 1 displays our lead standards and the absorbance that resulted from the GFAA. We used the data from this table to plot Chart 1. Chart 1 shows our calibration, however, the line was not completely straight so we added a trend line to fit it. The regression of the line was 0.9753.

Table 2 shows the results of the baby wipe digestion. The absorbance and the ppbillion are both displayed. We got the ppbillion by putting the absorbance in for y in the equation of the trend line: $y=0.0005 + 0.0031x$

From the data in Table 2 we can see that the concentration of lead goes down with a simple washing of counters. In Stewart 4, the lead reduced from 143.8 ppbillion to 55.8 ppbillion. And likewise, in Stewart 6, the lead reduced from 97.8 ppbillion to 69.8 ppbillion. Stewart 4 had a 61.2% decrease in lead while Stewart 6 had a 28.6% decrease. The baby wipe had an initial lead count itself. In addition, the two unknowns had a consistent concentration of lead: 43.8 and 45.8 for Stewart 4 and Stewart 6, respectively. This finding should be of concern to manufacturers of baby wipes. Overall, it is very important to demonstrate the importance of wiping the counters. The kids should be informed that a simple task such as washing the counters can decrease the amount of lead significantly.

Nonetheless, the levels of lead even before washing down were in the safe zone. The HUD limit dictates that lead should be under 5000 ppbillion, our sample were well below that limit.

David and I found the use of GFAA much easier than the ICP-MS. For a determination such as this one, we believe that the GFAA is a better instrument to use. The only problem with the GFAA is that there is more room for error. For example, if the drops placed in the GFAA graphite tube are not uniform or not the right size, our results may be off. Therefore, we did at least three trail of each sample to ensure that our results are accurate. Even though we did more trials in the GFAA, the relative amount of time to complete the experiment was the same as to the ICP-MS.

Table 1

Concentration (ppb)	Absorbance
10	0.009
50	0.031
100	0.041
200	0.1

Calibration Curve for GFAA

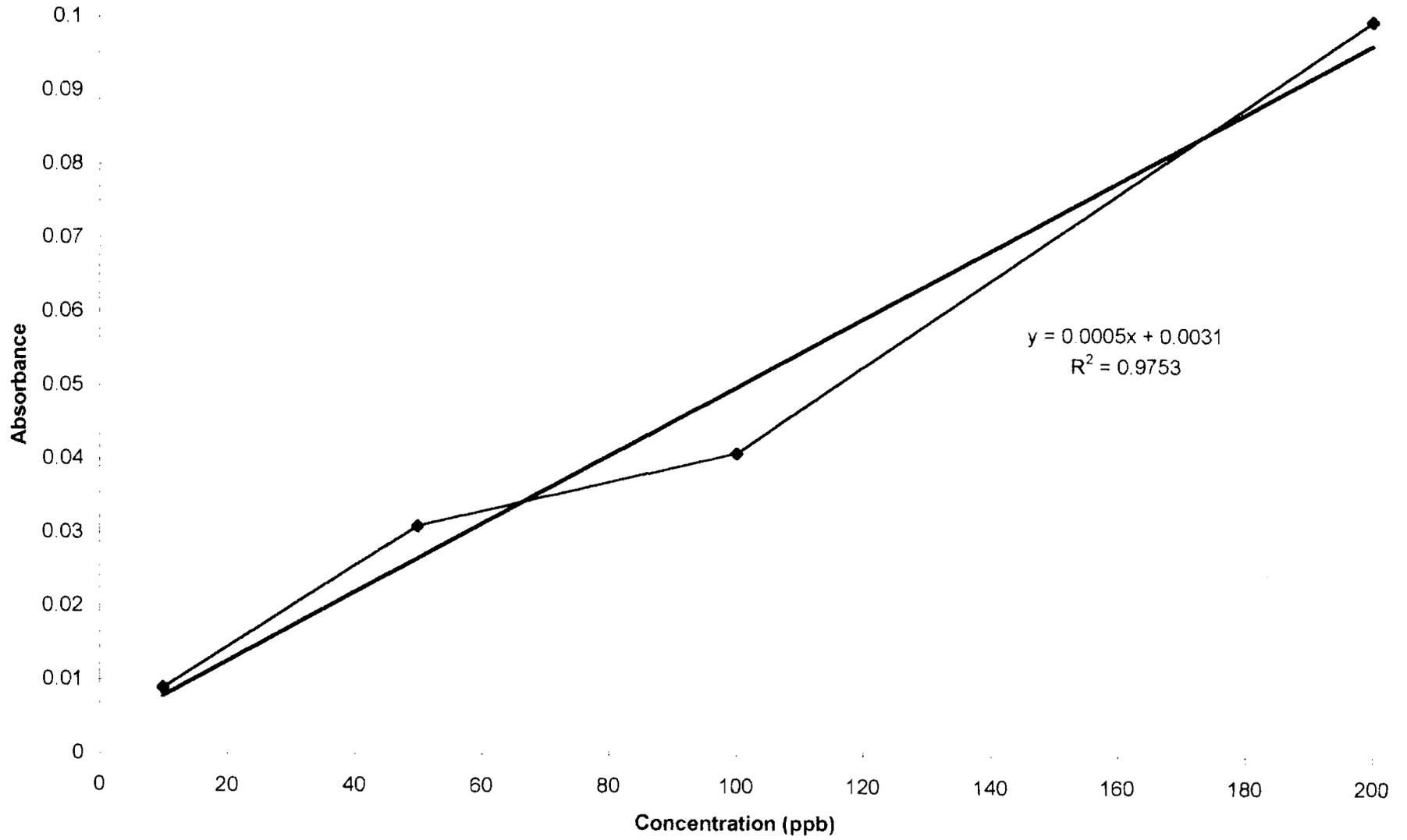


Table 2

	Steward 4 Absorbance	Steward 6 Absorbance	Steward 4 Conc. (ppb)	Steward 6 Conc. (ppb)
Unwashed Surface	0.075	0.052	143.8	97.8
Washed Surface	0.031	0.038	55.8	68.8
Baby Wipe alone	0.025	0.026	43.8	45.8
Blank run	1.919	1.1265	3132	2241



Stewart 4 & Stewart 6 ~~at~~ Lead determ. through CFAA.

Empty, then 0.009

Standards.

10 ppb → ~~0.008~~, 0.014, 2.009, 0.012 ↗
 50 ppb → 0.035, 0.031
 100 ppb → 0.038, 0.041, 0.041
 200 ppb → ~~0.208~~, 0.129, 0.100

Stewart 4

Unwashed Surface → 0.075, ~~0.060~~ 0.060
 Washed Surface → 0.031
 Baby Wipe → 0.025

Blank Standard → 1.919, 2.113

Stewart 6

Unwashed → 0.044, 0.052
 Washed → ~~0.042~~, ~~0.052~~ 0.038
 Baby Wipe → 0.026

Blank Standard → 0.831, 1.265

Blank

0.122
 0.178

