

ES 203
Lab Four
Measuring lead in the environment

Lead poisoning is a significant problem among children in the United States. The relatively low levels of lead that have been shown to affect children, coupled with the apparent irreversibility of some of the effects and the widespread distribution of lead in our environments has caused lead poisoning to be one of the more intractable environmental health problems. In class we have discussed the multitude of potential exposure routes. Lead paint represents a significant route of exposure, accessible either through paint chips or dust created by abrasion (e.g. windows) or deterioration.

How would you determine if you were living in a house with leaded paint? How would you determine the risk to your child in that house?

Lead-based paints were outlawed in 1978 so the age of the house is a decent guide to the risk posed by living in a particular home. Recently built houses are almost certain not to contain leaded paints; older houses, unless renovated are almost certain to contain leaded paints.

The best method involve dissolving a chip of paint in strong acid (called “digestion”) and analyzing it on an expensive piece of equipment such as an inductively coupled plasma atomic emission spectrometer (ICP-AES). These analyses require careful sampling at the site and transportation to the laboratory – this must all occur without contamination. The instruments require trained personnel to run them and careful and routine calibration to meet federal standards. It can take several weeks or longer for results to be returned to the client.

Another faster method uses portable X-ray fluorescence (XRF) instruments. XRF instruments present a potential radiation hazard to the user because they direct high energy gamma and X-rays onto a surface. Thus they too require trained operators. The instruments, while portable, are still expensive, costing \$7,000-\$45,000 per unit. XRF instruments generally provide an answer within a minute and do not damage the paint. Because the measurements are so quick and non-invasive, it is easy to make multiple measurements of a single residence in a relatively short period.

Yet another method for testing lead paint relies on the use of lead detection kits that are commercially available and can be used by the resident without the need to resort to experts. These kits contain a chemical that changes color in the presence of lead. They do not quantitate the amount of lead. Rather a color change indicates “yes, lead is presence” and no change in color indicates that it is not. The kits cost between \$1 and \$15 and are available at many local hardware stores.

The federal standard classifies paint as hazardous if it contains 1.0 mg lead/cm² of paint or if it contains 0.5% lead by weight.

Two common criteria for all public health screening measures are the rate of false positives and the rate of false negatives. A false positive is a test result that comes back positive when in fact

it isn't and a false negative is a test result that comes back negative when in fact it isn't. Given the situation, you can probably imagine circumstances in which you'd rather that a test err on the side of false positives (e.g. you'd rather go for a second test and be certain you don't have a disease) or on the side of false negatives (e.g. you'd rather not be bothered with more testing since the outcome isn't terribly important). In general, many tests strive to have false positive and false negative results occur less than 5% of the time.

When estimating risk, you can probably trust the federal government and assume that any paint that is leaded isn't good for your child. Remediation strategies are available and there are professional contractors who can guide your actions. When cost is a real factor, as it often is in rental units, there are less expensive steps you can take to mitigate the danger including regular dusting with a solution of trisodium phosphate (TSP) and vacuuming with a vacuum that contains a heppa filter.

Lab overview: In this lab you will be given a piece of paint from an old house in Lewiston and asked to determine the amount of lead in the paint using both the "best" method (the digestion process plus ICP analysis) and the "worst" method – the commercial lead paint kits. You will be given a couple of different commercial lead paint kits. Use one of each. Pool your results with others in the class to determine the following:

1. the amount of lead in the paint chip
2. the standard error in that measurement
3. the rate of false positives in each paint test kit
4. the rate of false negatives in each paint test kit

Given the small size of the class this year, we may not have enough data to answer questions 2-4. If that is the case, simply state whether the paint test kit results agreed or disagreed with the results obtained from the ICP measurement.

In addition, go to the EPA web site and download their IEUBKwin32 model.

<http://www.epa.gov/superfund/programs/lead/products.htm>

As described by EPA, the Integrated Exposure Uptake Biokinetic Model for Lead in Children (IEUBK):

... attempts to predict blood-lead concentrations (PbBs) for children exposed to lead in their environment. The IEUBK model allows the user to input relevant absorption parameters (e.g., the fraction of lead absorbed from water) as well as intake and exposure rates. Using these inputs, the model rapidly calculates and recalculates a complex set of equations to estimate the potential concentration of lead in the blood for a hypothetical child or population of children (6 months to 7 years of age). Measured lead concentration is not only an indication of exposure, but also a widely-used index for discerning future health problems.

The EPA and the Centers for Disease Control and Prevention (CDC) have determined that childhood PbB concentrations at or above 10 micrograms of Pb per deciliter of

blood ($\mu\text{g Pb/dL}$) present risks to children's health. Accordingly, EPA management actions seek to limit the risk that children will have lead concentrations above $10 \mu\text{g Pb/dL}$ and the Agency conducts risk assessments that reduce the likelihood that such exposures would occur. The IEUBK model calculates the probability that children's PbB concentrations will exceed $10 \mu\text{g Pb/dL}$ (or other user-entered value). By varying the data entered into the model, the user can evaluate how changes in environmental conditions may affect PbB levels in exposed children.

Using this model, enter the concentration of lead in dust that a child living in your house would experience and run the model (you can allow the default values to remain unchanged).

5. What would the predicted blood lead levels be for a child living in this environment?

Note: you will need to make some assumption about the situation to complete this part of the assignment. For example, you will need to decide how much of the dust in the house comes from the leaded paint. There isn't a right or wrong way with how to proceed here. You just need to be explicit about the choices you make.

Plan of action: Each lab section will divide up into groups of two (groups of three ONLY in the case of an odd number of students in lab). Each group of two will be given a paint chip and instructed on how to prepare this sample for analysis by the inductively coupled plasma atomic emission spectrometer (ICP-AES). The first week you will prepare the sample for analysis and begin constructing the environmental risk model you want to use. You will need to do some research to obtain all the values you will need to input into the model. The second week you will dilute your sample for analysis and we will run the samples on the ICP-AES. You will then take the results from the ICP and enter them into your model to arrive at an estimate of the health risk.

Experimental Details:

Below is a standard recipe for digesting paint using the microwave digester:

Microwave Digestion of Paint Chips for ICP-AES Analysis

The conventional method for preparing samples for ICP analysis entails heating the samples in concentrated acids on a hot plate. This method needs to be closely monitored, and generates large quantities of acid fumes. Microwave sample preparation circumvents the use of a hot plate by using microwave energy to heat the sample and elevated pressures to digest/dissolve the sample within a short amount of time. Smaller quantities of acid are used, and up to 12 samples can be digested at a time.

NOTE: Make sure to wear a full set of clothing and covered shoes to lab this week. We will be working with concentrated HNO_3 , and you will be sent away if safety precautions are not taken.

Safety Considerations (Beyond the Basics of Good Laboratory Practice)

1. All vessel components must be dry and free of particulate matter. (This eliminates localized hot spots that can superheat the vessels and cause them to fail.)
2. Never heat liquids in a sealed vessel that is not equipped with a pressure release device!
3. Never attempt to digest samples larger than 0.5 grams if the organic content and composition of the samples is unknown.
4. Microwave heating of alkaline or salt solutions in open or closed vessels will concentrate these solutions, causing precipitation of salts and formation of crystal deposits on vessel walls. These deposits will cause localized heating and may lead to failure of the vessels.
5. Do not heat high boiling point acids (phosphoric or sulfuric acids) inside microwave digestion vessels. These acids will heat to temperatures beyond the melting temperature of the vessel containers.
6. See instrument manual for list of chemicals NOT compatible with the microwave.

Procedure for Microwave Acid Digestion of Paint Chips

1. Wear gloves, safety glasses, and long sleeves, and long pants throughout entire lab period.
2. Each group should choose one of the following:
 - Vessel body
 - Vessel cover
 - Cap (top part)
 - Cap (bottom part)
 - Vent fitting (Gray fitting)
 - Sleeve
 - Safety glasses
 - Gloves
 - 10 mL pipette
 - volumetric flasks
 - 50 ml disposable centrifuge tubes
3. Install new rupture membrane into gray vent fitting, as instructed.
4. Tare the vessel body and add NO MORE THAN 0.5 grams of paint chip to the vessel.
5. In the hood, use the cap tops as a stand to steady the vessel body, and add 10.0 ml HNO_3 to each vessel, being VERY careful not to spill or splash the acid. Also, make sure height of sash results in reasonable airflow through the hood. If a reaction occurs, wait 15 minutes, or until reaction appears to be complete.

6. Assemble the vessels as instructed, putting the cover on the vessel and threading the cap top and cap bottom on hand tight. Thread the gray vent fitting on the cover until the threads are completely engaged. Take note of the vessel #.
7. Place vessels into a sleeve and in the carousel making sure that the pressure control vessel is in the appropriate position, and that the load is balanced. Thread the white pressure release fitting onto the gray vent fitting until threads are completely engaged. Take note of the position # on the carousel.
8. Turn the microwave on (switch in back).
9. On outside of microwave, turn the handle of the two-way valve counterclockwise to the horizontal (open) position.
10. Place pressure sensing line inside microwave in a beaker, and push plunger of syringe (syringe located on top of the microwave) until water begins to come out of the pressure tubing into the beaker. Remove beaker.
11. Place carousel into the microwave.
12. For the pressure control vessel, secure the pressure sensing line through the vertical standoff of the vessel carousel and attach to the pressure port on the pressure control cover until threads are completely engaged.
13. Secure loose pressure release tubes so they do not touch the roof of the microwave.
14. With the microwave door open, press "F4" to rotate the turntable. Make sure that the pressure sensing tube does not become entangled.
15. Turn the handle of the two-wave valve to the vertical (neutral) position.
16. Run the microwave program for PAINT.
17. After program has been run, let cool for 5 minutes.
18. In the microwave, spin carousel (F4) until pressure control vessel is towards the back. Release pressure control vessel at the pressure sensing line. Wait for ~1 minutes.
19. Transfer entire carousel to the hood in Dana 306.
20. One person at a time AND IN THE HOOD AND WITH THE HOOD SASH AT THE PROPER HEIGHT, release white fitting on gray vent fitting, and unscrew cap. Remove cover GENTLY. What do you see? Let samples degas for 5 minutes.
21. Gently swirl vessel and pour all contents into 50 ml volumetric flask. SLOWLY add Ultra-Pure DI water to graduated cylinder making sure you first add 5-10 ml water to vessel, swirl

and pour into flask. REMEMBER- this is an exothermic reaction!! Make sure you add 5-10 ml of water at a time, and make sure you swirl the contents of the graduated cylinder between water additions. Dilute the sample up to a final volume of 50.0 ml.

22. Transfer sample from graduated cylinder to labeled 50 ml disposable centrifuge tube. Take note of your dilution. (If you started out with 0.5 g paint chip and brought the solution up to 50.0 ml, this would be a 1:100 dilution.)
23. If an additional dilution is called for (e.g., 1:10), with a 1.0 ml disposable syringe, transfer and filter (at 0.45 um mesh) 1.0 ml of solution into a 15 ml centrifuge tube. Dilute sample up to a final volume of 10.0 ml. Cap, shake, label, and make note of your dilutions.
24. If no additional dilution is called for, with a 10.0 ml disposable syringe, transfer and filter (at 0.45 um mesh) 15.0 ml of solution into a 15 ml centrifuge tube. Cap, and label.
26. Analyse by ICP-AES for Pb

Sample Data Table

Samp. ID	Mass paint chip (g)	Conc HNO ₃ (ml)	Vessel # / Position #	Dil. #1 Final Vol (ml): paint chip (g)	Dil. #2 Vol Dil. #1 (ml)	Dil. #2 Final Vol (ml)	Run #	Pb (ppm)	

Lab Write up: Your lab write up should answer questions 1-5 above and provide adequate documentation to support the answers you arrived at.