Experiment 23: Hot Plate Digestions

SYNOPSIS: Samples to be used the rest of the semester will be prepared and stored. The general procedure is that given as a standard by the EPA. The digested sample will be measured on all instrumental methods in the rest of the semester.

PROCEDURE:

1. Obtain 11 50 mL beakers.
3. Mix the sample thoroughly to achieve homogeneity. Pick out debris and bugs. Grind soil to small particle size. Weigh five 1.00 g samples into separate erlenmeyer flasks. Weight a sixth sample into a weighing dish, label and set aside to air dry. Reweigh during the next lab section. This will be your control for the dry weight of the soil.
4. Place a weighing dish on the balance. Note the dish weight and/or tare to zero. Successively inject aliquots of de-ionized water each time noting the new successive weight. Determine the relative standard deviation associated with the pipette volume.
5. To two of the soil samples and one empty (three total) flasks, add 100 uL of 1000 ppm Pb standard. The two soil spikes and the one soil-less sample will constitute controls, as will the one soil-less lead-less sample.

   These steps will take about 1-1.5 hours.
6. In hood, to each flask, add 10 mL of 1:1 HNO_3:H_2O, mix and cover with a watch glass and heat to 95C, without boiling, for 15 minutes. A Dial level of about 3 on the hot plate should be approximately right. Cool. (You may speed up cooling by placing the erlenmeyer flask in a larger beaker filled with cold tap water). Add 5 mL of concentrated HNO_3, heat without boiling for 30 minutes to complete the **oxidation**. Elevate the watch glass on a bent piece of glass tubing and evaporate to 5 mL without boiling.

   The entire step 6 will take about 1-1.5 hours.
7. Cool, add 2 mL water and 3 mL of 30% H_2O_2. Warm on hot plate covered. "Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence." Heat until end of bubbling. Elevate the watch glass on a bent piece of glass tubing and evaporate to 5 mL without boiling. **Step 7 will take about 2 hours.**
8. Cool and dilute in a volumetric with de-ionized water to 100 mL.
9. Let sediment by sitting overnight and remove supernatant to acid-washed basic EDTA rinsed storage bottle. Label.

REPORT In addition to materials, methods, and results your report should include the following information:

1. How will you make use of the controls?
2. Why is HNO_3 used and not HCl (or H_2SO_4) to digest your sample? At what temperature does NO_3^- decompose? (See Chapter 7: equation 7.5, incinerators).

3. Why can't you boil your samples?
4. How long do you need to digest the sample to get all the lead out?
5. In what chemical forms will lead most likely be in your soil sample? Which will be hardest to solubilize?
6. Would you expect soils near a galena ore body (vol. i) to have the same rate of digestion as soils in an urban playground?
7. What implications might this have for the bioavailability of lead when ingested?
8. What materials in this lab would you consider hazardous?
9. Give a chemical reason for acid rinsing and EDTA rinsing your storage bottles.