Chem 404 - Experiment 3 – Atomic Absorption Spectrometry

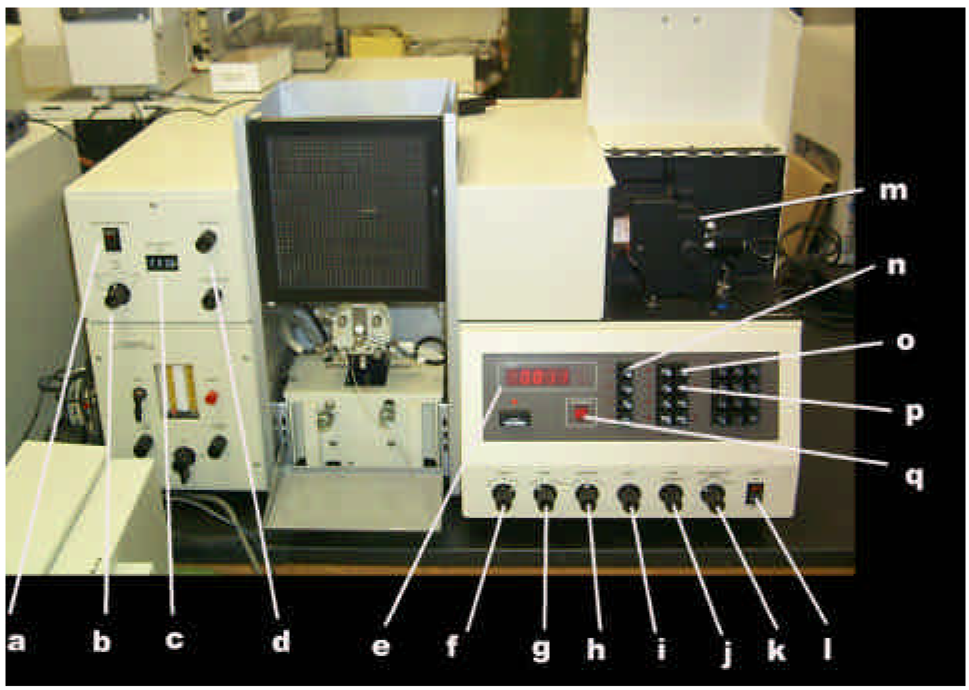
Summary: In this experiment you will:

- Prepare a calibration curve and determine the limit of detection for the determination of lead by atomic absorption spectrometry.
- Study the effect of spectral bandpass and lamp current on measurement linearity and sensitivity.
- Test the effectiveness of several water purification systems for the removal of lead.
- Use the calibration curve and the method of standard addition to determine the concentration of lead in the test solutions.

Location: Meet in the Instrumentation Lab (310).

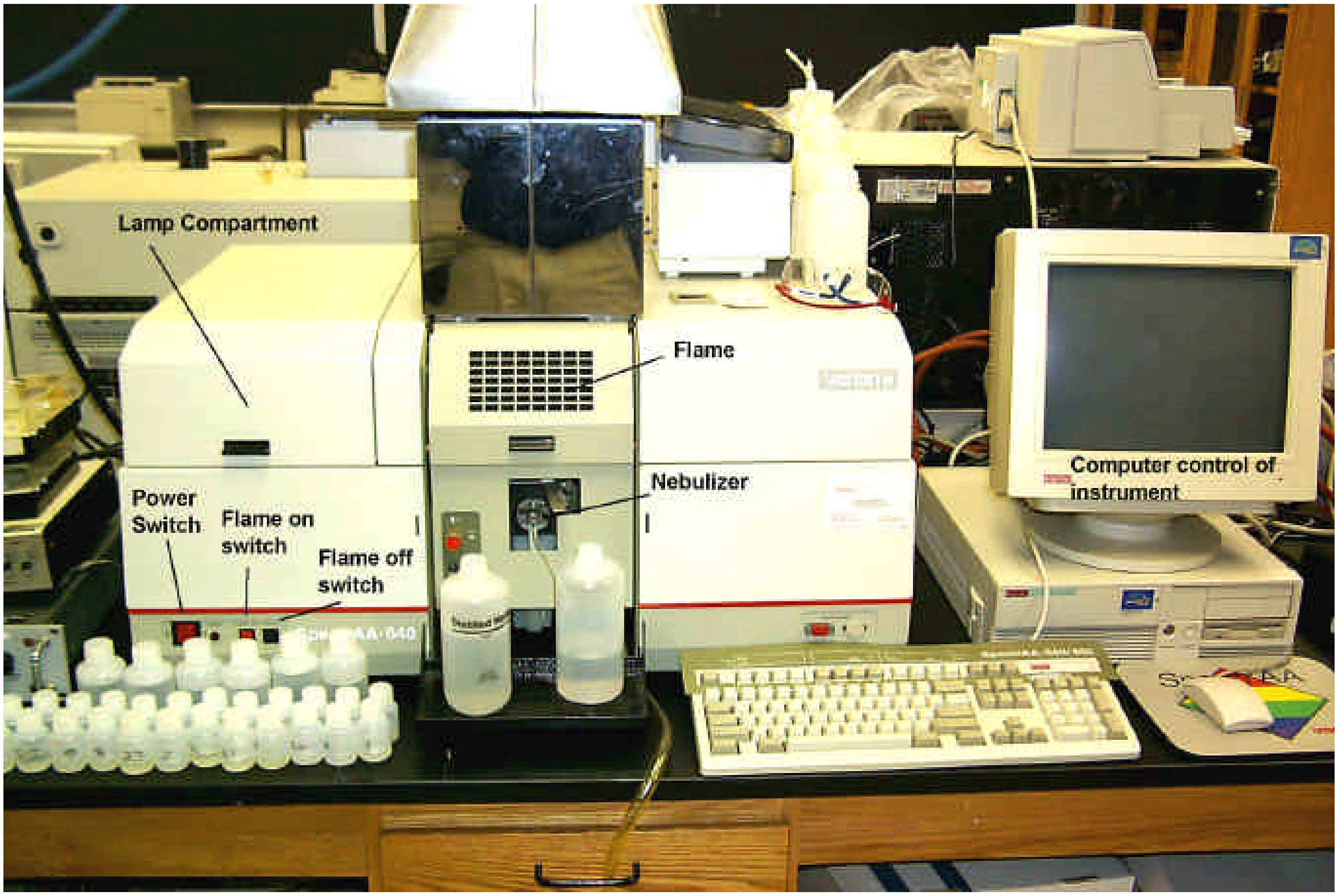
Prelab: Be familiar with chapters in your textbook covering atomic absorption spectrometry and methods of calibration (calibration curve, standard addition, and internal standardization).

Procedure: You will most likely be using the Perkin-Elmer AA spectrometer, unless I can get the Varian AA spectrometer working by class time. Operating information for the Perkin-Elmer instrument is shown below.



a) power switch for the wavelength drive (be sure to turn it off after each scan)
b) spectral bandpass setting. This is the range of wavelengths passed by the monochromator
c) the central wavelength being passed by the monochromator
d) manual setting knobs for the monochromator
e) the intensity observed by the spectrometer
f) the instrument signal switch
g) the instrument mode switch (set to continuous reading)
h) the instrument time constant (set to TC1 for fast response)
i) the instrument gain switch (set so that the intensity reading in (q) is approximately 90)
j) the lamp current (as specified for the lamp)
k) the signal type (set to AA)
l) the instrument power switch
m) the hollow cathode lamp (position must be optimized for maximum intensity. Don't touch the front of the lamp, which is quartz.)
n) the autozero switch (should be pressed before each scan to zero the instrument)
o) the measurement time setting
p) the expansion setting (for scale expansion)
q) the lamp intensity readout

The Varian AA instrument is computer-controlled and is shown below. If we use this instrument, the instructor will explain how to use the software.



(a) Prepare a calibration curve and determine the limit of detection for the determination of lead by atomic absorption spectrometry.

I have prepared a series of solutions in plastic bottles containing lead at various concentrations in about 1% nitric acid. Using the 283.3 nm Pb wavelength, the recommended lamp current and a spectral bandwidth of 0.2 nm, optimize the instrument using the highest standard concentration that is on the linear portion of the calibration curve (see the Perkin-Elmer cookbook for details).

- Measure each standard twice, starting from the blank and increasing the concentration to prepare a calibration curve.
- Take 15 replicate 3 second measurements to determine a limit of detection

Repeat the above measurements for spectral bandwidths of 0.7 nm and 2.0 nm.
List and explain any differences that you observe in the limit of detection, the sensitivity, and the linear range for these sets of measurements.

(b) Study the effect of spectral bandpass and lamp current on measurement linearity and sensitivity.

Using the maximum lamp current and a spectral bandwidth of 0.7nm, repeat the measurements that were performed in part (a).

List and explain any differences that you observe in the limit of detection, the sensitivity, and the linear range for these sets of measurements.

(c) Test the effectiveness of several water purification systems for the removal of lead.

Using the Pur and Culligan water filters, design an experiment to determine the percent of lead removed from water. Design your experiment considering the following:

- the detection limit for lead by flame AAS
- the linear range of the AAS measurement
- the capacity of the filter (it can filter 151 liters of water containing 0.15 ppm lead)
- the need for a slightly acid solution to stabilize the lead

(d) Use the calibration curve and the method of standard addition to determine the concentration of lead in the test solutions.

Use a calibration curve and optimum instrument conditions to determine lead in the effluent samples. Check recovery of a lead spike in the sample using the method of standard addition.

Answer the following:

- Report the % removal of Pb in each of the two filters. Based on the reproducibility of AA measurements and how well your duplicates for the Pur filter agreed, estimate the relative error on your value of % removal of Pb. How do your results compare with the manufacturer's specifications?
- Report the recovery of your standard addition. Why is standard addition used?
- Why is it best to only make measurements in the linear range of the AA instrument?
- If a solution to be tested has a concentration that is higher than the linear range of the AA instrument, there are several ways to make the measurement without diluting the solution to lower the concentration. Suggest at least 3 and list the advantages and disadvantages of each.
- The "uncertainty" or "noise" in an atomic absorption measurement is a result of random instability or drift in various components of the instrument. Suggest some possible sources of noise that might have affected your measurement of Pb.
- Why is it useful to modulate (chop or pulse) the light source for an absorption spectrometer? Is source modulation more important in a UV/Visible absorption spectrophotometer or in an atomic absorption spectrometer? Why?