Chemistry 324 - Instrumental Analysis
Experiment 5 (revision 1)
Effect of Instrumental Variables on Measurement - Atomic Absorption Spectrometer

Purpose: To introduce you to the factors that will influence an atomic absorption analysis.

Instrumentation to be used: Varian SpectrAA640 flame atomic absorption spectrometer

Summary Procedure: In this experiment you will change various instrumental parameters and note the effect on instrument response.

Detailed Procedure:
Using the Perkin-Elmer cookbook (3 copies are in the quant lab in the blue binders), choose the most sensitive wavelength for Cu and K. Based on the maximum concentration within the linear range, prepare 2 standards (total volume of 100 mL using distilled water) at one-half that concentration and another at one-fourth that concentration (i.e., if the maximum concentration within the linear range is 20 ppm, prepare standards of 10 and 5 ppm) using volumetric flasks. Stock solutions of Cu and K are in the quant/pchem prep room.

Using the atomic absorption spectrometer, set up separate methods for both elements. Do this by defining a new method (make sure manual sampling is chosen). Choose a slit width of 0.2 nm. The instructor will help you with the choices. For each method, determine using 16 five-second measurements. (Note: Check to see if methods have already been set up by the previous group.) The precision will be measured as the standard deviation (not the relative standard deviation) of the 16 measurements. You will have to calculate it, since the instrument only gives you a mean and a relative standard deviation.

- The flame-off precision (measured as standard deviation in absorbance units) - this measures only the noise from the hollow cathode lamp and/or the detector.
- The flame-off precision when the source is attenuated by a screen (determine the effective absorbance of the screen) - this tells you whether the noise is flicker (lamp instability) or white (detector photon noise). White noise is proportional to the square root of the change in intensity. Flicker noise is directly proportional to the intensity.
- The flame-on precision while water is being aspirated - note the absorbance of the flame. If the noise increases significantly and the flame is absorbing a significant amount of the source radiation, then the flame instability is the source of this noise.
- The absorbance and precision while running each of the two standards - If the noise increases significantly when the standards are measured, then analyte absorption flicker noise, due to variations in the sample introduction system, are a significant source of noise.

For Cu only, change the method so that the slit width (spectral bandpass) is 0.7 nm and repeat the above experiment. Repeat for a slit width of 2.0 nm. By increasing the slit width, you are increasing the intensity of hollow cathode lamp that reaches the detector, but you could also be increasing the stray light as well as the emission intensity from the flame that reaches the detector.

For Cu only, change the method to use the shortest wavelength available and repeat the experiment using a slit width (spectral bandpass) of 0.7 nm. Flame absorbance becomes more significant at shorter wavelengths.

For Cu only, at the shortest wavelength, adjust the flame to be fuel-rich and repeat the experiment using a slit width (spectral bandpass) of 0.7 nm. Do this in the optimize mode and raise the acetylene flow rate until the flame becomes a bright yellow. This will increase the emission from the flame.

Questions:

1. For each method, determine a detection limit based on the absorbance of the standard and the noise on the water blank when the flame is on. The detection limit is 3 x the standard deviation of the water blank.
2. Compare the sensitivity (i.e., absorbance per unit concentration) and noise levels as a function of slit width. Explain any differences.
3. Compare the sensitivity and noise levels as a function of Cu wavelength used. Explain any differences.
4. Does the noise level change when the source is attenuated by the screeen? Explain.
5. Is the noise greater when the flame is on in all cases? Explain your results.
6. How does the change in flame stoichiometry (i.e., using a fuel-rich flame) affect the sensitivity and noise level for copper measurements?
7. For each of the above cases, suggest the limiting noise sources at the detection limit and at high concentrations.
8. Based on your data, what would be the best conditions (slit width, flame stoichiometry, wavelength) to use for the determination of copper?