INS Chemistry Winter 2008

Lab 1: Emission spectra of elements and unknown identification.

For new students: Remember to bring safety goggles, wear closed toe shoes, and bring a laboratory notebook to lab. See the instructions for maintaining a lab notebook at http://academic.evergreen.edu/curricular/ins/0708/fall/handouts/Lab%20Notebook%20Guideline.pdf.

Useful reading: See the text page 283. There is a nice historic excerpt on the development of this method (1860) from Kirchhoff and Bunsen at http://dbhs.wvusd.k12.ca.us/webdocs/Chem-History/Kirchhoff-Bunsen-1860.html. When you are done, you may wish to consult the CRC Handbook; most editions have a table titled something like "Persistent spectrum lines of the elements." How do your observed lines match those of the reference?

Safety: As usual, remember your safety glasses and dress appropriately for laboratory. Because of open flames, this is a good opportunity to emphasize that long hair should be tied back in lab and loose clothing should be secured. If you haven't had one of Greg's lessons in acid spill cleanup, you might want to try one today.

Introduction:

A fundamental tool in astronomy, mineralogy, and analytical chemistry is the use of emission and absorption spectra to identify and quantify the elements present in a sample. In this lab we will use a propane flame to excite the electrons in elements to higher energy levels. Since the pattern of allowed energy levels is different for each element, the emitted light photons will have different energies, and consequently, different wavelengths.

Procedure

1. First familiarize yourself with the hand-held spectrometers. Look at a light source and be sure that you can find the wavelengths of the emitted light. Try this with the room ceiling lights. Do they emit light evenly across the visible spectrum or are there more intense emitted lines? Also note that the spectrum is labeled both in wavelength (depending on your model, in nm or Angstroms) and in electron-volts (eV). This is a measure of how much energy is contained in a photon of light at that wavelength. What is the range of eV values for visible light? What is the relationship between wavelength and eV value?
2. Now observe one of the gas emission lamps. Describe its appearance to your eye and then observe with the spectrometer. What are the strongest emission lines? Are colors visible with the spectrometer that were not obvious looking at the light with your eyes? (The emission tubes, particularly with elements that are not noble gases, tend to deteriorate and get very hot with prolonged use. For this reason, the power should be turned off whenever the tube is not being observed, and they should be given short cooling periods during long observations.)
3. Now look at the emission spectra of several metallic elements. Here we will work much as Bunsen did, introducing salts into the flame and then observing the results with a spectrometer. There are two commonly used ways to introduce the

salt to the flame-either by putting the salt on an inert surface and the heating the surface, or by spraying a mist of the solution into the flame, usually in a volatile solvent such as ethanol. This solvent method can also be used with small alcohol lamps. The challenge with this method is that many of the salts used have very poor solubility in ethanol. We will use a nichrome wire to add the elements to the flame. (We may also have some of the alcohol lamps to try.)

A. Clean glassware is vital for this method to produce reliable results. Thoroughly clean several small test tubes, being sure to rinse several times with DI water. You should then rinse each test tube with a small amount of 6 M HCl. In labeled tubes collect a small amount of the standard solutions provided: NaCl, LiCl, $CaCl_2$, KCl, $BaCl_2$, $SrCl_2$, and CuCl2. (Why is it useful to have all of these as the chloride salts? Be sure to note the concentration of the salts.)
B. In a hood set up a burner with gas and after ignition adjust the gas and air to produce an inner blue cone and no yellow color.
C. Clean your nichrome wire with dilute HCl and then hold it in the pale blue part of the flame. Repeat this process until the wire gives no color to the flame.
D. Dip your wire into one of your known solutions and place the tip of the wire into the same portion of the flame. Observe the emitted light with the spectrometer and also provide your description of the flame. It is very difficult for one person to both hold the sample and use the spectrometer, so do this with two people.
E. Repeat the cleaning procedure with your wire and the observation process with all of the elements provided.
F. The sodium emission lines are very intense. If possible we will get some pieces of cobalt glass that act as filters of these wavelengths. If you can, also observe the sodium and potassium lines through this glass.

4. After gathering data on all of the known samples, try your hand with some unknown samples. Take an unknown, note its ID label, and repeat your observation as in part C. Some of the unknowns are single compounds, and some are mixtures. In identifying your unknowns in your notes, be as specific as possible in explaining the basis of your identification. What specific lines were most useful in confirming your identification? Also, in justifying your decision, negative evidence is also useful-were there lines missing that allowed you to rule out some elements?