Chem 122 **Instrumental Analysis** **Fall 2010**

Laboratory #4
Estimation of Metal Concentration in Water Samples by Differential Pulse Anodic Stripping Voltammetry (DPASV)

Your fourth laboratory assignment is to quantitatively determine the concentrations of lead, cadmium and copper in the water samples you have already analyzed by two other methods. The technique to be used is differential pulse anodic stripping voltammetry. This equipment is one of the most sensitive and widely-used techniques for measuring metal concentrations in water. While a hanging mercury drop electrode (HMDE) is most commonly used, you will instead create a film mercury electrode. ***This topic is complex and at a minimum you must read through Section 25H in Skoog on stripping voltammetry.*** Additional reading is also listed below.

Due to time constraints you should use the standard addition method and limit yourself to three additions for each sample. The upper concentration limit for a mercury film electrode is 10^{-7} M, so you will need to calculate the corresponding maximum ppb for Cd, Pb and Cu. Use only millipore water for this experiment. You will need to purge the solution for 5 minutes the first time the cell is loaded (accurately measure 10 mL for all samples) and 30 seconds after each spike. The values of deposition time, potential and scan will be provided.

The total time on the instrument for each pair of students is limited to two hours. A two hour slot must be reserved on the sign-up sheet. As always, you will need to design your experiment such that you can determine all figures of merit for the method: sensitivity, uncertainty, detection limits, etc. Before beginning the lab, you will be required to answer basic questions and show your revised pre-lab (which must include calculations and overall procedure) for preparation of the stock solutions.

Stock solutions to be prepared:

- 50 mL volumetric flask of 2.5 ppm $HgCl_2$
- 10 mL volumetric flask of Cd from 1,000 ppm standard
- 10 mL volumetric flask of Pb from 1,000 ppm standard
- 10 mL volumetric flask of Cu from 1,000 ppm standard

Required reading from Skoog, 6th edition:

- All of Section 25H, p. 748-750: Stripping Voltammetry

Recommended reading from Skoog, 6th edition:

- Chapter 22: Intro to Electroanalytical Chem
- Ch. 23 p. 659-662: Potentiometry
- Ch. 25 p 716-728, 742-748: Voltammetry