RC TIME CONSTANT, IR DROP, AND MASS TRANSPORT LIMITS

General Schedule and Comments (this text is the same text that was present in Lab 2)

In general, the format for this class will be the same every week and is as follows:

1. Present brief outline of experiments, goals, and updates
2. Break into groups of two or three, set-up work space, and connect to the Bio-Logic potentiostat using the EC-Lab software on a PC laptop
3. Perform experimental procedure(s), while being assisted by the TA and the Professor
4. As a class, discuss the activity, provide feedback, and recommend other activities
5. Clean-up work space, return items, and store electrodes for subsequent weeks. (You are not dismissed from the discussion section until this is complete.)

Since your discussion section is short (110 minutes) and we want to cover as much as possible, please use your time wisely. NOTE: You will need to save or print procedures and reference publications/files before you attend the hands-on discussion sections. This will help you to become acquainted with the experiments and more importantly, will allow you to stay connected to the potentiostat as you run the experiments, because there are sometimes issues with using the Internet and the potentiostat simultaneously.

Introduction/Overview

Two weeks ago you fabricated most of the electrodes that you will use throughout the course and in the hands-on discussion sections and tried them out using cyclic voltammetry. This week, you will use these electrodes, and varying concentrations of supporting electrolyte, to determine the RC time constant for charging an electrode, the iR drop between the WE and RE, and the mass-transport-limited current by performing cyclic voltammetry experiments and chronoamperometry experiments.

Purpose

The purpose of this hands-on discussion activity is to become more familiar with the processes of diffusion, migration, and convection in electrochemical measurements. First, you will determine the RC time constant for charging your carbon button electrode as a function of the concentration of supporting electrolyte. Then, using your platinum microelectrode, you will determine the iR drop in solution between your WE and RE and the mass-transport-limited current for a Faradaic reaction as a function of the concentration of supporting electrolyte. This activity will provide you with a better understanding of the Nernst–Planck equation and will introduce you to common effects that mass transport has on rather standard electrochemical experiments.
Safety

You must bring personal protective equipment to the lab (i.e. lab coat and safety glasses/goggles). You must wear closed-toe shoes, pants, and a tee-shirt that covers your entire torso to be allowed to work in the hands-on discussion sections. These sections are mandatory so please do not get a zero due to improper lab attire. While in lab you will need to wear gloves, which we will supply (nitrile gloves). To reduce the possibility of electric shock to you and lab mates be sure you are working on the correct channel of the potentiostat and that all persons are away from the experimental apparatus before starting an electrochemical experiment. Moreover, do not touch the electrodes while a potential bias is being applied between them.

Procedures

Part A: Determination of the RC time constant

Tools/materials needed: 50 mL beaker, stir bar, rubber bands, carbon button electrode, Ag/AgCl (KCl sat’d) reference electrode, carbon cloth counter electrode, solutions of K$_2$SO$_4$ (0.1 mM, 1 mM, 10 mM, 100 mM)

1. Set-up a three-electrode electrochemical cell in a clean beaker and then fill the beaker with ~25 mL of the most dilute aqueous electrolyte solution (0.1 mM K$_2$SO$_4$). Perform the following electrochemical measurements, without stirring.
   a. OCV: for 30 seconds, recording the potential every second
   b. CA: apply 0.2 V vs RE for ~60 sec, recording average current every 0.1 sec
   c. Repeat the CA measurement, with stirring

2. Replace the solution with the next most concentrated solution (i.e. 1 mM K$_2$SO$_4$) and perform the same measurements, but for CA only run for ~30 sec, recording every 10 ms.

3. Replace the solution with the next most concentrated solution (i.e. 10 mM K$_2$SO$_4$) and perform the same measurements, but for CA only run for ~10 sec, recording every 1 ms.

4. Replace the solution with the most concentrated solution (i.e. 100 mM K$_2$SO$_4$) and perform the same measurements, but for CA only run for ~2 sec, recording every 200 µs.
Part B: Determination of the iR drop in solution and mass-transport-limited current

Tools/materials needed: 50 mL beaker, stir bar, rubber bands, platinum microelectrode, Ag/AgCl (KCl sat’d) reference electrode, carbon cloth counter electrode, solutions of K₂SO₄ each with 0.5 mM ferricyanide/ferrocyanide (0 mM, 1 mM, 10 mM, 100 mM)

(1) Set-up a three-electrode electrochemical cell in a clean beaker and then fill the beaker with ~25 mL of deionized water containing 0.5 mM Fe³⁺(CN)₆³⁻ and 0.5 mM Fe²⁺(CN)₆⁴⁻. Perform the following electrochemical measurements, with vigorous stirring.
   a. OCV: for 30 seconds, recording the potential every second
   b. CV: sweep between -100 mV and 600 mV vs RE at a scan rate of 10 mV/s for several reproducible sweeps

(2) Repeat the procedure for each of the other three electrolyte solutions, in increasing order of concentration of K₂SO₄.

Assignment (assigned with next week’s activity; due Monday, February 27, 2017 at 3pm PST)

For the next assignment you will be required to interpret your data. Therefore, it is advantageous to think about your results and discuss them with others in the context of the Nernst–Planck equation and related information that we discussed during the lecture classes.