

## Guidelines for Writing-Up Experiment 1

The three separate parts of Experiment 1 make up a fairly comprehensive study of  $\text{Fe}^{+2}/\text{Fe}^{+3}$  redox chemistry in solution. You have studied what happens when there is no current flowing (equilibrium), when a little current flows (linearized electrode kinetics), the effects of mixing (rotating disk electrode) and no mixing (Cottrell behavior), and how reaction products can be collected at a concentric ring (ring-disk studies). Try to merge these separate experiments into a cohesive story about the behavior of the iron redox system.

Each team should turn in a single report. I will look critically at these write-ups since you have been given ample time to digest the material and are being allowed to team write the report.

### Elements to include in your report

Please include the following items in your report. Label each section clearly with a heading.

#### Introduction.

Provide some general motivation, and include a nice cyclic voltammogram (CV) from EXP 1C. This CV can be used to describe, in general introductory terms, all the features of the  $\text{Fe(II)}/\text{Fe(III)}$  system that were explored experimentally in this series of labs.

#### Experiment.

Include a general description of the equipment and procedures. You may describe a potentiostat in generic terms, but don't need to go into detail about all three of the different potentiostats you used. You may refer the reader to the appropriate Lab Descriptions I gave you for specific details.

#### Results and Discussion

- (1) Equilibrium behavior. Describe the relevant theory and assumptions. Compare the equilibrium behavior of the system to that predicted by the Nernst equation. Make a plot of Equilibrium Potential vs.  $\log(\text{Fe}^{+2}/\text{Fe}^{+3})$  and compare data with Nernst prediction. Discuss the implications of the results.
- (2) Charge transfer kinetics near equilibrium. Describe the relevant theory and assumptions. Compare your data with the predicted behavior and determine the exchange current density. Is the  $i_0$  you found reasonable? How far from equilibrium could you go before the theory you were using did not hold anymore? What caused the theory to "break down" beyond those potentials? In short, discuss the implications of your data.
- (3) Mass Transfer Limitations far from equilibrium in mixed and unmixed systems. Describe the relevant theory and assumptions for the two cases studied: unmixed electrolyte subjected to a potential step, and steady-state mass transfer at the rotating disk electrode (RDE). Each of these two methods allow you to determine the diffusion coefficient for  $\text{Fe}^{+2}$  and  $\text{Fe}^{+3}$ . Make plots of  $i$  vs.  $t^{-1/2}$  and fit to the Cottrell Equation to find the diffusivity. For the RDE experiments, plot  $i$  vs.  $(\text{angular rotation rate})^{1/2}$  and determine the diffusivity that way. Compare the diffusion coefficient for  $\text{Fe}^{+2}$  and  $\text{Fe}^{+3}$  you determined from the two different methods. Are they physically reasonable? Discuss any limitations in either dataset or the accompanying theory, and describe which method you believe is better suited for diffusion coefficient measurements.
- (4) Analysis of products using a rotating ring-disk electrode (RRDE). Calculate the theoretically predicted collection efficiency for the electrode you used. How close are the experimental values to the theory? Does the collection efficiency depend on disk rotation rate? Discuss.

#### Implications and Concluding Remarks

- (1) Summarize the implications of the laboratory. Might any of the experiments we did have relevance to technology?
- (2) Describe any errors or improvements you would suggest for making the experiments or their write-ups better.

**ChemE 461**  
**Grade sheet for Exp. 1**

**TEAM:** \_\_\_\_\_  
\_\_\_\_\_

**COMMUNICATION**

Clarity, brevity, and organization \_\_\_\_\_ (7)

**TECHNICAL CONTENT**

Theory \_\_\_\_\_ (10)  
(Is it correct? Are the limits of the theory well laid out?)

Experimental \_\_\_\_\_ (8)  
(Are diagrams clear and description sufficient to replicate results—remember that details can be given in an appendix)

Results \_\_\_\_\_ (15)  
(Measured what was asked? Calculates relevant physical parameters?  
Plots results in a manner that should yield straight lines? Gives clear sample calculations in appendix A?)

Discussion \_\_\_\_\_ (15)  
(Relates observations to one another and gives physicochemical *reasons* for observed behavior? Compares quantitative and qualitative results from one experiment to next? Compares to theory?)

Conclusions and Recommendations \_\_\_\_\_ (5)  
(Evidence in report leads reader to conclusions?  
Are thoughtful recommendations given?)

Total \_\_\_\_\_  
(60 max)

**COMMENTS:**