Experimental Procedure

Microfluidic devices provide a very simple, low cost format for producing an analytical sensor for copper that could measure water contamination. In this lab, copper solutions of different concentrations will be used to create a concentration cell. The concentration of one side of the cell will be held constant while the other will serve as the variable cell. Both electrodes will be metallic copper.

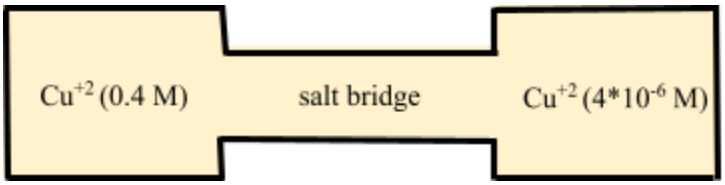

Figure 1. Depiction of microfluidic concentration cell (top view)

The salt bridge allows movement of ions in order to maintain electrical neutrality. In this experiment, you will use cornstarch as the medium to hold the ions and table salt (NaCl, sodium chloride) as the source of the ions.

Electrochemical cells ideally operate according to the Nernst.

$$E_{cell} = E^{\circ}_{cell} - \left(\frac{0.0592\,V}{n} * log\, Q \right)$$

Equation 1. Nernst Equation - E_{cell} is the electrical work done by the system, n is the number of electrons present in the system, and Q is the reaction quotient of the system.

Helpful videos

1. Setting up the multimeter
2. Setting up the electrodes
3. Making the salt bridge
4. Making electrochemical measurements

Materials

- Multimeter
- Dogbone wax printed chips

- Weighboats
- Pipette (and tips)
- Copper wires
- Tape
- Scissors
- Water
- Copper loaded chips
- Something to boil water (stove and saucepan, tea kettle, microwave)

Prelab

1. What are the two half-reactions taking place in the concentration cell?
2. What is E^o for this cell?
3. Given a 0.0001 M Cu^{+2} solution and a 1M Cu^{+2} solution in the concentration cell, what solution will be reduced? What solution will be oxidized? Explain your reasoning.
4. Calculate the theoretical E_{cell} value for the concentration cell.
5. Which side of the cell should each electrode connect to? What would a negative voltage mean?
6. Based on the Nernst equation, what should be plotted on the x and y axis to produce a linear calibration curve, what should be plotted on each axis?

Procedure

Corn Starch Salt Bridge

1. Boil water in small either in a saucepan on stove, in a teakettle, or in the microwave
2. Take ¼ cup of boiling water and place in a small dish
3. Add 1 tablespoon of salt to the hot water
4. Add 2 ½ tablespoons of cornstarch to the mixture
5. Stir until mixture thickens
6. Cut one dogbone chip away from the rest
7. Use a pipette to add the cornstarch solution to the skinny section in the dog bone chip, making a salt bridge

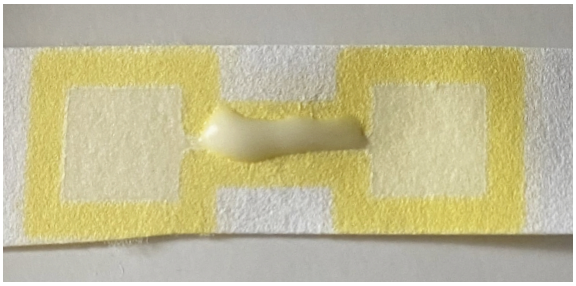

Figure 2. Corn starch salt bridge

Preparing Solutions

1. You will need to make 2 solutions to start with using one disk of the two concentrations you were provided with.
2. Begin by putting one of the disks you have been given into a 1.5mL eppendorf tube and adding 500 µL of water. What is the theoretical concentration of that solution?
3. Repeat this procedure with the second concentration you were provided with.

Making Measurements

4. Secure the copper wires to the electrodes. Wrapping the wires around the metal several times ensures a strong connection. Test the connection by switching the multimeter to measure resistance and touching the wrapped wires together. It should read 0 Ohms.
5. Develop a way to stabilize the copper electrodes so that they will be in contact vertically with the solution and you do not have to hold them. There are several ways to do this, just ensure the wires are not obstructed by anything.

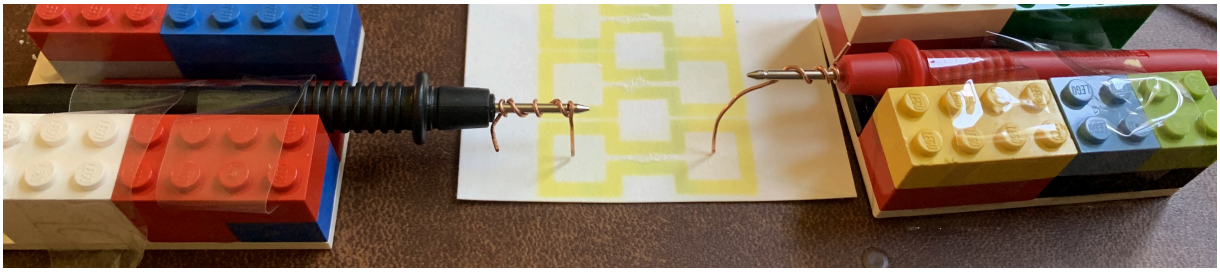

Figure 3. Example of electrode setup on dog bone chip

6. Turn on the voltmeter to the 2 V setting.
7. Put 80 µL of $1x10^{-4}$M in the cell that has one electrode. In the other cell, put 80 µL of the lowest diluted concentration made from the 1 M solution. These solutions should be placed in the cells very quickly with not much time in between deposition of solutions. Remember: these are the concentrations of the copper that was deposited on the copper chips, not the final concentration after rehydration and dilution
8. Monitor the multimeter. When the reading is relatively stable (no longer quickly changing) record the voltage. Pick a specific amount of time at which to measure, such as two or three minutes.
9. Pay careful attention to what makes a difference here in your measured potential. Ideally the potential should stabilize quickly and not change substantially.

Making the calibration curve

1. Make a new solution using one of the 1 M disks to make a stock solution. Dilute the stock solution in order to create 6-8 different concentration solutions over a concentration range of 10^6 range in concentrations to measure values and construct a calibration curve. This may best done using serial dilution
2. Prepare a fresh solution using one of the $1x10^{-4}$M chips. This is the solution that will be fixed as you vary the other side of the chip.
3. To measure different concentrations, start with a fresh chip. Begin by pipetting 80 µL of the concentration that won't change in the red side. Starting with the lowest concentration in the side of the chip you will be changing, pipet 80 µL and place the black electrode into that side. Measure the voltage.
4. To measure the next highest concentration suck off the solution using a pipette, dispose of the solution back in the weighboat, and put on the second lowest concentration solution on the red side. The solutions should be measured from lowest to highest concentration to avoid leaving excess copper on the chip.
5. Remove copper electrodes from the solution and dry them off.
6. Plot your calibration curve in light of the Nernst equation.

Questions you need to investigate in lab:

1. What side should the different solutions be added to (black or red)? What does changing the electrode sides do?
2. What are the best dilution factors for this experiment? What is the linear range?
3. Is it best to insulate the wires? Do different kinds of tape work better than others?

Post-Lab Questions

1. Compare the experimental E_{cell} value to the theoretical value you calculated for the prelab. What is the percent error? What are some things that might have contributed to this difference?
2. In preparing your solutions for your calibration curve, you likely used serial dilution as the technique. Practically, what is the advantage of using this method? Analytically, why do we generally avoid using this technique and prefer to use dilutions of a single stock solution?
3. Plot your calibration curve keeping the Nernst equation in mind. What is the significance of the slope? Does it agree with the theoretical value? Why or why not?
4. What is the significance of the y intercept? What should it be theoretically? Please explain your answer.

5. What happened to the sign of your potential as you changed the concentration? How is the sign of the potential related to electron flow?
6. What is the linear range of your calibration curve? Please compare these to the values for a typical absorbance/colorimetric measurement.
7. Remove one of your standards from somewhere near the middle of your calibration curve and replot your calibration curve. Treating the standard you removed as your sample, plug the potential you measured into your calibration curve and solve for your unknown concentration. As a reminder, the rule for propagating uncertainty with an exponential relationship is: $x=10^{ba}$ $\sigma_x=|x|\left|(bln(10)\sigma_a)\right|$. What is the % RSD and how does that compare to absorbance/colorimetric measurements?
8. What did you discover were important design considerations in conducting the experiment? What are three possible sources of error of this experiment? Human error is not an acceptable answer.
9. How would you theoretically change the experiment to ensure optimal results?